

OPERATING COMMENTS  
PLANT TECHNICAL  
JULY 1967

2.4-D

Work progressed on the study relating to spray drying Ec 2,4-D. The second test run at Custom Processing, which was made in an attempt to increase the water content to the desired level, indicated that this would be difficult. Product appearance and dryer operation both were marginal when operating with 1.7% free water. Based on these results, it was recommended that the moisture content be held at 6.5% to 7.5% (0 to 0.6% free water). Thus the Ma 2,4-D produced will have a moisture content similar to the material recently produced at the Semi-Works and the 2,4-D assay will be about 5% higher than stated on our label.

Following Cleveland's decision to proceed with drying a 5000-pound test batch of Ec 2,4-D, work orders were issued to make the piping changes to permit double filtration, and also the loading of slurry into drums. The necessary containers have also been ordered, and Operating Instructions for the double filtration drafted.

MEA/DCP/EOL

Work continued in the EOL area. Studies were started to more accurately determine the mode of operation and the efficiency of the  $H_2SO_4$  scrubber in the DCP system; however, data obtained before the shutdown of the Unit was inconclusive. This work will be continued to provide a basis for further improvement in the removal of chlorophenols.

Additional information from the Water Department indicated that we have been subject to random and radical changes in the sulfate content of our water supply, depending on the distribution of water purchased from Elizabeth. Since the purchase of this water was terminated late in June, the sulfate level in our city water should be more consistent and generally lower in the future. We will continue to sample the water to check this out.

A new sampling system for the DCP Unit was designed. The system should reduce the fumes now released when taking DCP samples, and it is also planned to install a small ventilating system to further reduce this problem.

DCP

Work-up of samples previously taken on the absorption tower has been delayed by problems with the flame ionization chromatograph. Efforts are being taken to expedite this repair so that this work can resume.

The tests run on a small pilot decanter indicate that the losses from the DCP recovery unit can be kept to the 0.2% stated on the flowchart, or slightly below, provided the operating temperature is kept around 60°C. The loss was largely due to solubility of DCP in the acid-water, with mechanical carry-over not being a major problem. Cooling the acid-water effluent from the decanter to 30°C could cut the loss by about 50%.

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Losses in samples taken from the present TCP recovery unit averaged slightly higher due to two significantly higher losses which probably indicate physical carry-over from the system. Presently, the introduction of "T" Mother Liquor into the TCP purification unit for recovery, as well as the advisability of installing a cooling tank to act as a secondary collector, are being evaluated.

Work orders were issued for the installation of the new methanol condenser. When the T<sub>1</sub>CB collector was removed for inspection, it was found to need new bags, which were requisitioned.

#### DACAMINES

The major effort with regard to the Dacamines was directed toward the solution of the problem of bulging cans first noted about the middle of July. The immediate cause of the bulging cans has been found to be due to hydrogen generation due to reaction with the steel cans. Thus, the problem appears limited to the 5-gallon cans, and the various drums, since the 1-gallon cans have a tin wash and seem to be immune to the problem.

The propensity to generate hydrogen and bulge the cans has been noted on a number of Newark batches produced this year. The H<sub>2</sub> content increases with increasing water content, but it is thought that water alone is not responsible for the problem. Also, the quantity of water found in some of the batches cannot be readily explained, since it is much higher than what should have been introduced with the reactants, or possibly formed during the reaction.

Efforts are continuing with both gas chromatograph and infra-red analysis of raw materials and product and preparation of lab batches under varying conditions to determine the cause of the hydrogen formation. Some differences have been noted, and major emphasis is being given to variation of raw materials and effect of reaction temperature.

Earlier in the month, evaluation of another emulsifier was completed. It, (Alcolac's Siponic E-15) can be added to the growing list which might be used both in 2 $\frac{1}{2}$  and 4 $\frac{1}{2}$  Dacamines, and which can result in satisfactory emulsification of the Dacamines in Uran 32.

#### EXPANSION

With the shutdown of the "T" Unit on July 4th, demolition proceeded quite rapidly. The new "T" slurry hold tank was placed in service on July 13th which enabled improved access to the building. Installation of major equipment for the "D" line generally is proceeding close to or ahead of schedule. Piping is another matter --- progress on the conversion from "D" to "T" has been slow, and little work has been done on the TCP purification system, leaving it some 6 weeks behind the schedule drawn up in June. Emphasis at this writing is being given to completion of the converted unit for start-up on "D" next week.

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MISCELLANEOUS

Process studies on the production of Silvex acid have essentially been completed. The attempt to eliminate the recovery of TCP by adjusting TCP/WCA mole ratios was unsuccessful. It appears that steam distillation of the acidified silvex will be the only practical means of removing the excess TCP from the Silvex. A report detailing the results of the study will be issued shortly.

Work was completed on three complaint samples during the month, all of which involved the Dacamines.

Of the Trainees assigned to the Plant, two are working directly in the Technical area, and the third is working on larger maintenance projects with Mr. E. A. Smith. Vacations totalling four weeks were taken by Technical personnel during July.

The following appropriations were closed in July:

No. 6741-18	- Plant pH Meter	- \$250	Expended
No. 6741-22	- Filter for Tank Farm	- 427	"
No. 6741-23	- Fume Scrubber Catwalk	- 960	"

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OPERATING DATA - JULY 1967

2.4-D

Average DCP Conversion, %	93.7
Average Cycle Time, Hours	7.8
Average Cooking Time, Hours	2.0
Average Cooking Temperature, °C	103.7
Usage #/# Product, DCP/MCA	.822/.592
Average Product Assay, %	None Assayed

MCA/DCP

Number of Batches	30	26
Average Batch Size, Lbs.	8,300	10,904
Average Reaction Time, Hours	6.6	13.5
Average/Maximum Reaction Temp. °C	109/121	76/82
Average Exit Gas Temperature, °C	-17	19
Usage #/# Product, Chlorine	.457	.496
Usage #/# Product, Acetic or Phenol	.619	.580
MCA	2,4-DCP -)	
DCA	2,6-DCP -)	None
Acetic	o-Cl-p -)	Assayed
Anhydride	2,4,6-TCP -)	

HCl

Average Phenol Content, ppm	122*	* Exclusive of acid-catalyzed.
Average Sulfate Content, ppm	79*	

2.4,5-T

Average TCP Conversion, %	Too few
Average Cycle Time, Hours	batches
Average Cooking Time, Hours	
Average Cooking Temperature, °C	
Usage #/# TCP/MCA	to
Average Product Assay, %	Calculate

TCP

Number of Batches	20	2,4,5-TCP -
Average Batch Size, Lbs.	2,561	LCP - None
Average Reaction/Digestion Time, Hrs.	1.9/5	Anisole* - Assayed
Average/Maximum Autoclave Temp. °C	168/173	p-Dioxin* -
Maximum Temp. in Anisole Still, °C	105	
Usage #/# Product, T <sub>2</sub> O <sub>8</sub>	.927	* and related impurities.
Methanol	.384	
Caustic (Liq./Solid)	.420/.334	

ESPES

	<u>EUTYL-D</u>	<u>EUTYL-T</u>	<u>2-EE-D</u>	<u>2-EE-T</u>
Number Batches	40	20	28	No
Average Batch Size, Lbs.	7,283	3,621	7,359	Production
Average Cycle Time, Hours	21.8	26.3	18.8	
Average Reaction Temperature, °C	143	142	157	
Average Free Acid, %	0.9	0.0 (ASTM)	2.0	
Average Color	-	-	-	

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