

OPERATING COMMENTS
PLANT TECHNICAL
NOVEMBER, 1967

2,4-D

Start-up of the 2,4-D Unit continued during the month. At month's end, all units except the flaker were operating. Production rates have risen greatly since the beginning of the month, but capacity operation has not yet been consistently maintained. Engineering shift coverage was terminated on November 10th.

About the middle of the month, the dryer was started up. Operations to date have been very good; the unit capacity is sufficient to meet our needs even at lower-than-design steam pressure, and the moisture content of the dried acid has been satisfactory (generally .1% or less). A detailed study of the unit's performance will be made this month to determine that the performance guarantees have been met.

Operation of the new flaker was attempted, but rapid breaking of the doctor blades caused a shutdown after a short time. Investigation of this problem led to the belief (later confirmed by Goulin-Birmingham) that the doctor assembly is improperly positioned in the flaker. They have provided necessary spacers to correctly position the doctor --- these have just been received and have not yet been installed. In the interim, the idle 2,4,5-T flaker was tied into the "D" system to maintain flake production. The vendor will be back-charged for labor expended by the Plant to make the necessary changes.

The new parts to correct the agitator overload have been received and installed. On the 29th, an unexplained failure of a pinion gear in the reactor agitator drive resulted in about 20 hours downtime. Performance of the agitators will be watched closely for the time being to see that everything is OK.

Plugging of several transfer lines caused considerable problems, but corrective tracing and relocation of some lines and the temporary removal of some of the magnetic flowmeters helped. The flowmeters were a cold spot in the line, since they initially were not traced. Additional data from the vendor indicated that the tube ends could be traced, and this has allowed their use though the problem has not been entirely eliminated.

Pumps also were a problem due to rapid failure of some of the mechanical seals and the cracking of the castings on the bearing housings of two pumps. One of the Engineers is now investigating our pump problems not only in the "D" Unit, but elsewhere in the Plant, to try to improve their performance.

Plans were drawn up for the production of sodium 2,4-D from Monsanto acid during 1968. It is proposed that we will convert the acid in the amine makeup tank where the operation should be quite simple. Changes necessary to make the Na 2,4-D slurry will not be very extensive.

2,4,5-T

Operation of the "T" Unit was much improved over October. About mid-month, the settling tank was again found to be leaking at the bottom nozzle only about 3 weeks after being patched. Because of the extremely vital role of this vessel in the "T" Unit, due to its role in relieving the system pressure, it was

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decided to purchase a new vessel for this service. Appropriation No. 6760 was submitted and has been approved for this vessel.

MEA/DCP/HCl

Because of the increased interest in outside sale of MCA, work was resumed on our studies of MEA production. The analytical methods have finally been established, and we find that they are fairly close to procedures being used by DuPont to evaluate our MCA. Analysis of samples show an average assay of 94.2% MEA, 5.1% DCA, and 0.7% acetic. Product consistency, however, is not good, since the MEA content varies from 92.5% to 95.5% and DCA from 3.5% to 6.7%. Since DuPont indicates a need for 97%-98% MCA with 1.5% to 2.0% DCA, our current production will not meet their specs. We are now investigating methods of improving MEA assay in our present unit.

No work was done on DCP or HCl production during November.

TCP

Operation of the TCP purification unit continued satisfactorily during the month. The carbon tower was cleaned for the first time on the 16th. This cleaning was not as smooth as had been expected, since common brick at the bottom of the tower reacted with the HCl used to clean the carbon liberating CO₂ which caused the acid to "burp" out the vent. Notwithstanding this problem (which will not arise on the permanent tower), the carbon was cleaned and successful operation was resumed. The tower was given a second cleaning using a revised procedure early in December, with improved results. The p-dioxin level can be controlled to <1 ppm. Since it does rise when the tower needs cleaning or is mis-operated, the p-dioxin levels will have to be constantly checked.

DACALONES

The only activity in this area was the preparation of some samples for Chemform and Nopco. Chemform will use their samples as a base stock for evaluating new emulsifiers. Nopco also was sent a base mix which they will use as a control check on the RDY they are going to make for us.

EXPANSION (APPROPRIATION NO. 6739)

With start-up, the active construction phase is essentially complete. Some items remain yet to be finished, and these will be finished as soon as possible. The most important item in this category is the permanent TCP tower which is due to be shipped this week. Insulation within the buildings is now finished, but work continues on the DCP and TCP tank.

The only open area not yet resolved with Engineering is the installation of equipment to control dust and fumes from the flaker. We have received a proposal from one vendor which we feel would do the job. Engineering has not yet agreed with out contention that this is an integral part of the appropriation, so this matter is still under discussion.

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MISCELLANEOUS

Several possible alternate Singleshot formulations, using Armour amines, were tried. To date, none has been satisfactory. Work continues with Armour on this project --- they now are trying to duplicate the G-3760A. Several mixed Iso-Butyl-D formulations were prepared for Amoco and sent to them.

Mr. W. Irizarry left the Technical Staff at the end of November. To date, only one candidate (who was unacceptable) was interviewed for this opening. Personnel is working on this, and another candidate will be in tomorrow for interview. We still have two openings for Technicians and few possible candidates were referred by Personnel for interview during November --- they are being requested to expedite their search, since our need is pressing.

The following appropriation was closed in November:

No. 6747 - TCP Methanol Still Condenser - \$2,473 expended.

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F. GORDON STEWARD

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2,4-D

Average DCP Conversion, %	95.2
Average Cycle Time, Hours	16.0
Average Cooking Time, Hours	2.2
Average Cooking Temperature, °C	105.4
Usage #/# Product, DCP/MCA	.878/.621
Average Product Assay, %	99.5

Cycle times lengthened by delays during initial start-up of operations.

MCA/DCP

	<u>MCA</u>	<u>DCP</u>
Number of Batches	49	43
Average Batch Size, Lbs.	8,303	10,904
Average Reaction Time, Hours	6.8	13.0
Average/Maximum Reaction Temp. °C	109/117	84/93
Average Exit Gas Temperature, °C	-17	19
Usage #/# Product, Chlorine	.457	.496
Usage #/# Product, Acetic or Phenol	.592	.587
Product Assay, %		
MCA)	94.2	2,4-DCP-)
DCA)	5.1	2,6-DCP-)
Acetic)	0.7	o-Cl-p-)
Anhydride)	-	2,4,6-TCP-)

HC1H

Average Phenol Content, ppm	93
Average Sulfate Content, ppm	92

2,4,5-T

Average TCP Conversion, %	75.7
Average Cycle Time, Hours	8.7
Average Cooking Time, Hours	3.4
Average Cooking Temperature, °C	100.0
Usage #/# TCP/MCA	.912/.584
Average Product Assay, %	None Assayed

TCP

Number of Batches	64	2,4,5-TCP -)	None
Average Batch Size, Lbs.	2,345	DCP -)	
Average Reaction/Digestion Time, Hrs.	2.0/5.0	Anisole(*) -)	Assayed
Average/Maximum Autoclave Temp. °C	167/172	p-Dioxin(*) -)	1.7 ppm
Maximum Temp. in Anisole Still, °C	105		
Usage #/# Product, T ₄ CB	1.110		
Methanol	.492		
Caustic(Liq./Solid)	.427/.300		

(*) and related impurities.

ESTERS

	<u>BUTYL-D</u>	<u>BUTYL-T</u>	<u>2-EH-D</u>	<u>2-EH-T</u>
Number of Batches	56	35	7	No
Average Batch Size, Lbs.	6,493	5,902	6,973	
Average Cycle Time, Hours	30.4	31.6	22	
Average Reaction Temperature, °C	140	145	151	Pro-
Average Free Acid, %	0.9	0(ASTM)	1.9	duc-
Average Color	-	-	-	tion

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