

OPERATING COMMENTS
PLANT TECHNICAL
OCTOBER, 1967

2,4-D

The Technical Staff was heavily committed during the month to the completion of construction on the new 2,4-D Unit, its preparation for operation, and at month's end, the actual start-up.

The main portion of the construction crew left the job site on October 16th. Following that, a majority of the instrumentation work and a significant amount of piping, steam tracing, etc., were completed by Plant forces. Newark Engineers, along with the Central Engineering personnel on the site, laid out the details of the many jobs done and checked out the unit prior to operation. During this period, equipment data for the unit was gathered together, spare parts lists prepared, and Operating Instructions issued.

Two Engineers (J. D. Anderson from Greens Bayou, and J. A. Mueller from Research), arrived on October 24th to assist in the start-up. Following several days in which they became familiar with the unit, they joined the shift rotation and participated in the actual start-up.

The unit commenced operations on October 30th, so total production and operating experience by month's end was not significant. Several problems, however, have come to light during operations to date, including:

1. The filter hold tank agitator was overloaded by a factor of 4-5 times. The vendor (Philadelphia Gear) was contacted, and they recommended a change in gear ratio and impeller diameter to alleviate the problem. The gear ratio change has been made, and the unit is temporarily being operated with only two blades until the smaller impellers are received.
2. A similar, but much less serious overload was observed on the 2,4-D reactor. This will be corrected by shortening the upper impeller blades on the unit.
3. *which one?* One magnetic flowmeter was found to have been damaged in installation and had to be removed from the line prior to start-up.
4. Initially, pH control was erratic, but readjustment of the instruments largely eliminated this problem.
5. Plugging of certain transfer lines was also a significant problem which led to considerable downtime during the first days of operation. Since two of the magnetic flowmeters seemed to be causing or accentuating the plugging, one was temporarily removed and the other bypassed to allow production to proceed. This plugging problem must be investigated in more detail.

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As of this writing, operations through the wash column are proceeding fairly well, but drying and flaking remain to be started. Production rates are rising, but will probably remain below capacity for a while, as the other units are started and changes to ease the operation of the unit are made. An effort must be made to get instruments not in service or not working properly into operation, and the reaction cycle time must be analyzed so that steps can be taken to reduce it.

2,4,5-T

Operation of the "T" Acid Unit did not proceed as well during the early part of the month as had been hoped. Continued operational problems, particularly plugging of transfer lines, plus operational errors, resulted in excessive downtime during this period. Because of this, Engineering shift coverage, which had been terminated on the 6th, was re-instituted on the 17th, and is still continuing.

Late in the month, the old 6x6 realurry tank which had been leaking was replaced with a new tank which eliminated a major problem in the operation of the unit. Also at this time, a new agitator was installed in the settling tank; and some piping revisions to simplify the unit were made. During the last week or so, production has been rising, partly as a result of these changes, but primarily because of increased Operator familiarity with the unit.

MCA/DCP/ECL

Essentially no work was done in these areas due to low production and the commitment of personnel to the 2,4-D and 2,4,5-T start-ups.

TCP

Treatment of all TCP produced continued in a temporary carbon tower during October. Operation was by batchwise dilution of the TCP in the intermediate storage tank until October 24th when the specific gravity controller was put into service, and dilution was started, using the in-line mixer. Operation of this controller has been quite satisfactory to date. The only control problem in the TCP Unit has been a problem at times in maintaining a consistent flow of TCP to the mixer. This flow is controlled manually, and it is believed that a discharge of salt cake from the anisole still, resulting from the use of the new spargers, is causing plugging. This is not believed to be a serious problem. Checks of the p-dioxin level made during the month were all satisfactory.

DACAMINES

Rechecks of iodine values on the Armour "high" IV sample, and on the shipment of Kemamine D-999 confirmed earlier results --- the IV's are lower than expected.

Discussions were held with representatives of Nopco concerning the use of EDY in the Dacamine-4D. Samples of the mix, less emulsifier, along with the emulsion test procedures, have been sent to Nopco for use in testing their

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product. Emulsion tests on the Dacamines were also run jointly, to familiarize Nopco personnel with the emulsification of the Dacamines.

EXPANSION (APPROPRIATION NO. 6739)

As already indicated, much finishing-up work was done in the 2,4-D area with Plant personnel during October, following the departure of the bulk of the construction force. The only construction work now underway is the insulation work, which is nearing completion. Some additional items need to be finished off, but these will be completed with Plant forces in November, as time permits.

In addition, before the job can be considered finished, the problem of providing some form of dust control at the flaker, and the apparent need for insulation on the DCP and TCP tanks must be resolved. Discussions are currently underway with Central Engineering on the question of the tank insulation, and we are awaiting work from them regarding the dust collector.

MISCELLANEOUS

~~Additional work was done on the Singleshot formulation to see if one of the Armour Ethoxsens could be substituted for the G-3780A. This would appear possible, though the exact formulation was not determined.~~

Two additional Lab Technicians were authorized during the month. One Technician resigned, and one new Technician was hired (he reported to work on November 8th), so that we still have two vacancies in the Lab.

The 200-gallon spare glass-lined vessel authorized by Appropriation No. 6758 was ordered ~~during the month.~~

~~No appropriations were closed in October.~~

FGS/ac

11/8/67

F. Gordon Steward
F. GORDON STEWARD

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OPERATING DATA - OCTOBER, 1967

2,4-D

Average DCP Conversion, %	-	Too few batches
Average Cycle Time, Hours	-	
Average Cooking Time, Hours	-	to be
Average Cooking Temperature, °C	-	
Usage #/# Product, DCP/MCA	-	meaningful.
Average Product Assay, %	None Flaked	

MCA/DCP

	<u>MCA</u>	<u>DCP</u>
Number of Batches	10	5
Average Batch Size, Lbs.	8,303	Too few batches to be meaningful.
Average Reaction Time, Hours	7.2	
Average/Maximum Reaction Temp. °C	108/114	
Average Exit Gas Temperature, °C	-16	
Usage #/# Product, Chlorine	.457	.496
Usage #/# Product, Acetic or Phenol	.621	.579
Product Assay, % (*)		
	MCA)	2,4-DCP-)
	DCA)	2,6-DCP-)
	Acetic)	o-Cl-p-)
	Anhydride)	2,4,6-TCP-)
	90.3	None Assayed.
	7.2	
	2.5	
	-	

(*) Only 1 sample.

HCl

Average Phenol Content, ppm	Not enough production to be meaningful.
Average Sulfate Content, ppm	

2,4,5-T

Average TCP Conversion, %	77.9
Average Cycle Time, Hours	12.6
Average Cooking Time, Hours	8.0
Average Cooking Temperature, °C	99.0
Usage #/# TCP/MCA	.93/.573
Average Product Assay, %	None Assayed

TCP

Number of Batches	43
Average Batch Size, Lbs.	2,311
Average reaction/digestion time, Hrs.	1.9/5.1
Average/Maximum Autoclave Temp. °C	166/172
Maximum Temp. in Anisole Still, °C	105
Usage #/# Product, T ₄ CB	1.107
	Methanol
	.523
	Caustic (Liq./Solid)
	.423/.314

ESTERS

	<u>BUTYL-D</u>	<u>BUTYL-T</u>	<u>2-EH-D</u>	<u>2-EH-T</u>
Number Batches	81	22		
Average Batch Size, Lbs.	7,732	6,520		NO PRODUCTION
Average Cycle Time, Hours	21.2	26.2		
Average Reaction Temperature, °C	143	145		
Average Free Acid, %	0.9	0 (ASTM)		
Average Color	2.2	-		

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PGS/nc

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