2,4-D

During the Plant's annual maintenance shutdown, installation of the secondary acidification tank and the other changes (mainly instrumentation) necessary for the conversion of the unit to pressure operation for 2,4,5-T were completed. The Operating Instructions for the unit were revised to reflect the changes made, and were re-issued.

Engineering coverage was provided for the first week of operation in the revised unit. The settling tank was left in the system, and all operations were carried out using it. Minor problems, principally leaks, resulted in some start-up delays. Mechanical operation of the secondary acidification tank was satisfactory. The ultimate effect of the two-stage acidification on raw material usage has yet to be determined.

Late in August, a large-scale test of the spray drying of Na 2,4-D was made at Custom Processing. The run proceeded very well, with a drying rate of 700 lbs./hr. being easily attained. Thus, drying charges should be very close to the estimated 6-7¢/lb. The moisture content of the Na 2,4-D was about 8%, a little higher than we expected to be able to go to, but the product remained free-flowing. The approximately 6,200 pounds of product, including 1,200 pounds which must be repackaged, were returned to Newark to await shipping instructions.

2,4,5-T

No 2,4,5-T was produced during August. To prepare for the impending start-up of the 2,4,5-T melt process, some changes were made in the unit. The most significant of these was to change the location of the control valves to the settling tank so that it can be used on "T" production for the time being. Some additional work, including installation of a new pump and a pressure-reducing valve, and the changing of the drop size at the TCP drop tank remains to be completed prior to starting up "T". This work is underway or will be done during the clean-out period.

Operating Instructions for "T" production have been issued. The section of the system to be pressurized was pressure-tested while running "D", a number of leaks were found and corrected. However, no major problems were found with the unit.

MCA/DCP/HCl

The analysis of MCA for DCA content recently has been troublesome, since we have not been able to accurately determine the DCA content in our MCA. Analytical results using a new column packing for the chromatographic analysis, however, seem to be more promising. The most recent data indicate that our DCA content may be running about 5%, which is much higher than was thought to be the case. Work continues on improving the techniques of MCA analysis. Based on the results obtained to date, a more extensive study of the MCA chlorination will be made as soon as manpower becomes available.

DS

An evaluation of the resistance of the rubber linings in our HCl tanks to a sample of Bennel Products acid was completed. Some sign of attack was detected, and when the age of the linings (up to 10 years in 2 tanks) is considered, introduction of this acid into our system seems inadvisable.

No work was done on the improvement of HCl quality during August, due to the absence of the Engineer working in this area. Because of the start-ups of the new unit, work in this area will be further delayed.

TCP

Analysis of the last runs made in the lab column indicated that satisfactory removal of the p-dioxin was continuing. No signs of carbon saturation were detected. A test in which the effluent resulting from eluting the column was extracted with benzene, indicated that the benzene could remove 75% of the p-dioxin from 10 times its volume of effluent. Allowing for less efficient operation in the Plant, a 5:1 ratio would be more likely in actual operation. This ratio doesn't seem to be acceptable for use. No further work in this area is planned at present.

A pilot still column was constructed to provide additional operating data and also for use in cleaning up the TCP on inventory. As of this writing, over 10,000 gallons of solution have been treated. Operation seems to be quite good; the pressure drop is low, and removal of the p-dioxin has been essentially complete for most of the run.

DACAMINES

Major work in this area again centered on the evaluation and resolution of the problem of bulging cans. Tests completed during the month indicated that all Dacamines, whether made from HCHO or trioxane acid, will react with steel, liberating H_2 if sufficient water (about 1%) is present in the formulation. Thus, the presence of water in some of the Dacamine-4T made at Newark this year seems to be the main cause of our problem.

Water can be formed during the reaction between amine and acid. Regardless of the rules of mixing the components of the Dacamine-4T used to make Newark's difference in the final moisture content of a number of lots is apparently the main source of water was a "wet batch" resulting from the failure of a tank heater.

Evaluation of new samples of Hopco's RJ7 emulsifier was completed. As before, this emulsifier was rated to be acceptable, but not quite as good as the Igepal CO-970 we are using. Secondine samples prepared from samples of Hopco Dacamines were also made, and these are now undergoing storage tests.

A special Dacamine-4T Dacogin formulation was tested at the request of the Sales Department. Performance appeared OK.

EXPANSION (APPROPRIATION NO. 6739)

During the first part of August, construction activity was at a peak, coincident with our annual shutdown. Emphasis was given to completion of the work in the existing 2,4-D Unit, which was available for service on the 15th.

Following completion of this work, piping in the TCP area picked up after several weeks in which very little was done in this area. As of this writing, piping in the TCP area is about finished, but about two weeks' work remains to wrap up the instrumentation in the Unit. Installation of all major equipment in the 2,4-D area was completed at month's end, and piping is underway.

MISCELLANEOUS

The process work on the production of Silvex acid and ester at Newark was completed. Production in the "T" reactor would be possible, but downstream handling of the Silvex would have to be different. Due to the solubility of the sodium Silvex removal of the substantial quantities of TCP remaining, following completion of the condensation, would have to be by steam distillation or by means of a caustic wash following esterification. Also, because of the higher melting point and resulting higher pressure, processing through the melt system would not be possible.

Therefore, if production of Silvex were started in existing equipment, the reaction could be carried out in the "T" reactor with all further processing (acidification, purification, and esterification), best being done in one of the esterifiers.

Work continued on formulating Dacthal, but to no avail.

The Trainees assigned to the Plant have now all completed their assignments; Messrs. Borzelli and Kohn leaving late in August, and Mr. Sacks the first week in September. Performance of all three while at Newark was very good. Absences due to vacations and illness totalled eight weeks among the Technical personnel during August.

The following appropriations were closed in August:

No. 6741-6	- Fans	- $ 342	Expended
6741-15	- Teflon-lined Pump	- 638	"
6741-17	- Pump with Mechanical Seal	- 520	"
6741-20	- DCP Separator	- 580	"
6741-21	- Wash Column Feed Pump	- 500	"
6741-24	- Process Filter Leaves	- 1,166	"
6741-25	- DCP Mist Eliminator	- 980	"
6748	- Gas Chromatograph	- 3,275	"
6749	- Spare 1000-Gallon Reactor	- 9,119	"

F. Gordon Steward

FGS/nc

F. GORDON STEWARD

9/13/67

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2,4-D

Average DCP Conversion, %	95.3
Average Cycle Time, Hours	8.1
Average Cooking Time, Hours	2.6
Average Cooking Temperature, °C	101.3
Usage #/# Product, DCP/MCA	.871/.605
Average Product Assay, %	98.5

MCA/DCP

	MCA	DCP	
Number of Batches	25	33	
Average Batch Size, Lbs.	8,302	10,905	
Average Reaction Time, Hours	8.1	12.1	
Average/Maximum Reaction Temp. °C	109/122	79/86	
Average Exit Gas Temperature, °C	-18	19	
Usage #/# Product, Chlorine	.457	.496	
Usage #/# Product, Acetic or Phenol	.597	.578	
Product Assay, % MCA -	93.1	2,4-DCP)	
DCA -	4.0	2,6-DCP)	None
Acetic -	2.8	o-Cl-p)	Assayed
Anhydride -	0	2,4,6-TCP)	

HCl

Average Phenol Content, PPM	130
Average Sulfate Content, PPM	60

2,4,5-T

Average TCP Conversion, %	NO
Average Cycle Time, Hours	
Average Cooking Time, Hours	
Average Cooking Temperature, °C	PRODUCTION
Usage #/# TCP/MCA	
Average Product Assay, %	

TCP

Number of Batches	
Average Batch Size, Lbs.	
Average Reaction/Digestion Time, Hrs.	NO
Average/Maximum Autoclave Temp. °C	
Maximum Temp. in Anisole Still, °C	PRODUCTION
Usage #/# Product, T_4CB	
Methanol	
Caustic(Liq./Solid)	

ESTERS

	Butyl-D	Butyl-T	2-EH-D	2-EH-T
Number Batches	53			
Average Batch Size, Lbs.	7,659			
Average Cycle Time, Hours	26.6		NO PRODUCTION	
Average Reaction Temperature, °C	110			
Average Free Acid, %	0.8			
Average Color	-			

DS 00001110

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