2,4-D

Work continued on spray drying Na-2,4-D. Following a test at Custom Processing in Trenton, they indicated that drying costs for the Na-2,4-D would be in the range of 6-7¢/lb. Analysis of the product obtained from the test indicated that, like the Semi-Works, they had over-dried the material, obtaining essentially 100% monohydrate, without the desired 5% free water. When this point was discussed with Custom Processing, they indicated that the desired moisture content should be attainable provided the material remains free flowing at this condition. An additional test will be run to investigate this point.

The recent Semi-Works' production in which the assay ran about 5% over specification, and specimen label, points up the need for clarification from Sales in this area. Specifically, we need information on what deviation from product specifications will be allowed.

Tests are also underway to determine the quantity of water wash required during the second filtration to reduce the residual Na_2SO_4 content to an acceptable level. Plans also are being drawn up for the minor piping changes necessary to be able to recycle for the second filtration, and also load the Na-2,4-D slurry for shipment.

The vent duct leading from the flaker was sloped and flushed in an attempt to minimize plugging of this duct. Plans also were drawn up for the installation of a system to divert the effluent from the pressure filter to the sewer when the sodium sulfate tank is full.

MCA/DCP/HCl

The quality of the Muriatic Acid transferred to storage improved during the month, but this result is somewhat deceptive, since much of our production (including the material with higher phenol content), had to be neutralized because of lack of sales.

Efforts to improve operating techniques have shown some results with reaction and exit gas temperatures being reduced somewhat. Major emphasis on corrective action is currently being directed toward further cooling of the HCl stream to condense and remove additional chlorophenols, and toward improving the efficiency of the H_2SO_4 scrubber in the system. We have some indication that additional cooling already installed is beneficial.

One thing noticed during the month was that for the first time since we started spot checking the sulfate content of the city water, a marked change in the concentration was observed. Both water samples taken during the first half of June contained higher-than-normal sulfate concentrations, with one sample running above 50 ppm. Later in the month, very low sulfate readings of 4 ppm were observed. This variation was of course also observed in our Muriatic Acid. In view of these results, we are going to more closely follow the sulfate content in our city water supply to see to what extent it is playing in our problems in attaining the 50 ppm sulfate specification in our acid.

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TCP

Operation of the experimental column in which p-dioxin is removed from the TCP solution by absorption on activated carbon continued. The column has been successfully cleaned with HCl several times and again placed in service and found to be removing essentially all of the most active 2,3,7,8 isomer. Operating cycles on a plant-sized column appear practical. Work is continuing to attempt to find the point at which the carbon becomes saturated with the p-dioxin.

Work is also underway to test the performance of the continuous decanter proposed for the new unit. A small pilot unit has been set up in the Lab and has been operating successfully during short duration runs. Additional tests are being made, directed toward minimizing the TCP loss from this unit.

The jacketed casing of the T_4CB transfer pump was found to be leaking, and has now been replaced. With the elimination of this source of water from the system, the performance of the T_4CB collector will be again evaluated to see if it is satisfactory. The fabrication of the new condenser for the methanol still was completed, and it has been received from the vendor. It will be installed during the annual shutdown.

A review of reports, memos, etc., reporting the results of earlier TCP process studies has been completed. A summary of pertinent data was made for use during future work on the TCP process.

DACAMINES

Two new emulsifiers from Stepan have been evaluated for possible use with the Dacamines. Agent 351-60 gave very good performance at the recommended lower amine-to-acid ratio (comparable to the Chemsol CS-100-3 previously recommended). Their Agent 351-70, though it did not give as good a bloom in the 2# Dacamine, could be used in both the 2# and 4# formulations.

The performance of 2# Dacamine formulated with Stepan's Agent 351-60 and Chemsol's CS-100-3 along with Singleshot was checked in Uran 32. All gave acceptable stable emulsions, much superior to the performance of the standard Dacamine. Thus, to meet the requirement for a product compatible with the liquid fertilizer, we can either change to another emulsifier and go with the 2# Dacamine, or offer Singleshot.

EXPANSION (APPROPRIATION NO. 6739)

Installation of the new vacuum system and hot water heater was completed about the middle of June, and these units were placed in service, allowing removal of the old units and the installation of several foundations. The amine makeup tank was taken out of service on June 13th, and a major effort since then has been directed toward the installation of the new "T" slurry hold tank. The "T" unit was turned over to construction on July 5th and demolition is now well underway.

MISCELLANEOUS

Process studies on the production of Silvex acid using Plant TCP and a sample of monochloropropionic acid obtained from France have been started. Initial

efforts are being directed toward eliminating the steam distillation to recover residual TCP included in previous processes. This work is continuing.

Work is underway on the development of an emulsifiable concentrate containing Dacthal. Of the formulations tested to date, none has been satisfactory.

An Engineer attended a Chamber of Commerce session at which the impact of some new anti-pollution legislation was discussed. The general impression was that though there will be no immediate effect on the Plant, the tightening of air pollution controls authorized by this legislation could eventually affect our operations.

Two Engineering Trainees, Messrs. R. D. Forzelli and F. Sacks, and one Summer Trainee, Mr. P. M. Kohn, joined the Plant in June. They were given a general orientation to our activities and then assigned to various technical projects.

No appropriations were closed in June.

F. Gordon Steward

FGS/nc

F. GORDON STEWARD

7/12/67

OPERATING DATA - JUNE 1967

2,4-D

Average DCP Conversion, %	92.2
Average Cycle Time, Hours	8.2
Average Cooking Time, Hours	1.9
Average Cooking Temperature, °C	102.4
Usage #/# Product, DCP/MCA	.874/.581
Average Product Assay, %	99.1

MCA/DCP

	MCA	DCP
Number of Batches	57	51
Average Batch Size, Lbs.	8,302	10,794
Average Reaction Time, Hours	6.6	16.1
Average/Maximum Reaction Temp. °C	112/118	78/90
Average Exit Gas Temperature, °C	-14	17
Usage #/# Product, Chlorine	.457	.496
Usage #/# Product, Acetic or Phenol	.579	.580
Product Assay, %	None Assayed	2,4-DCP - 90.7 2,6-DCP - 8.6 o-Cl-p - 0.7 2,4,6-TCP - 0.0
MCA		
DCA		
Acetic Anhydride		

HCl

Average Phenol Content, ppm	120*
Average Sulfate Content, ppm	91*

*Exclusive of acid neutralized.

2,4,5-T

Average TCP Conversion, %	76.5
Average Cycle Time, Hours	6.8
Average Cooking Time, Hours	4.6
Average Cooking Temperature, °C	104
Usage #/# TCP/MCA	.849/.560
Average Product Assay, %	None Assayed

TCP

Number of Batches	74
Average Batch Size, Lbs.	2,370
Average Reaction/Digestion Time, Hrs.	1.9/5
Average/Maximum Autoclave Temp., °C	167/171
Maximum Temp. in Anisole Still, °C	105
Usage #/# Product, T_4CB	1.078
Methanol	.511
Caustic (Liq./Solid)	.405/.336

TCP Assay

2,4,5-TCP	- 92.3
DCP	- 1.6
Anisole *	- 0.8
p-dioxin	- Not Assayed

* and related compounds.

ESTERS

	BUTYL-D	BUTYL-T	2-EH-D	2-EH-T
Number Batches	62	43	1	No Production
Average Batch Size, Lbs.	7,699	4,054	7,184	
Average Cycle Time, Hours	22.1	22.8	-	
Average Reaction Temperature, °C	146	143		
Average Free Acid, %	1.1	0.0(ASTM)	-	
Average Color	-	-	-	

DS

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