OPERATING COMMENTS
PLANT TECHNICAL
FEBRUARY 1967

2,4-D

Operation of the 2,4-D Unit was again closely followed during the month. Recycle of the wash liquor to the reactor was continued, with beneficial results. Additional product samples are now being assayed to again check for possible changes in product quality. Previous assays have shown no significant change due to the recycle and if, as expected, the current samples again indicate this, the quantity of wash liquor being recycled will be increased.

As indicated last month, we plan to record the flow of sulfate to the 6x6 sprays to enable it to be more closely controlled. This installation is essentially complete and will be operating shortly.

Further examination of the data on the centrifuging tests indicate that centrifuging would not be a satisfactory replacement for rotary vacuum filtration, even when using counter-current washing. In view of this, the centrifuging tests have been terminated.

A statistical evaluation of the assays of the Monsanto 2,4-D acid received both at Newark and Des Moines was made to check the validity of their claim of 99.6% assay, based on a statistical evaluation. This evaluation showed their claim to be invalid. Based on the assays of acid received by Diamond, a statistical assay of 99% 2,4-D acid was obtained.

MCA/DCP/HCl

The major change during February in our continuing program to improve HCl quality was to install the new DCP vapor-liquid separator. It appears to be capable of removing somewhere in the order of 20 to 30 pounds per day of additional chlorophenols; however, its operation has been hampered by a higher than anticipated pressure drop. Piping changes to alleviate this problem are now being planned.

TCP

Work in this area was limited to more tests on the efficiency of activated carbon as a means of purifying the TCP. These tests showed that the absorption can be accomplished with very short residence times. Since carbon can only be used if the quantity to be used is within manageable limits, additional tests directed toward determining the saturation point are being run (the first saturation tests were inconclusive).

DACAMINES

One additional Plant-scale test batch of Dacamine produced from Kommamine-D-999 was made during February. However, major emphasis was again given to additional laboratory work on the Dacamines.

The evaluation of Tennoco 400 provided some interesting results, particularly the fact that the Dacamines made with it were only about half as viscous (300 cps vs. 650 cps) as samples prepared with HAN. The low temperature

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stability using Tenneco 400 is also somewhat improved. Negative effects of using the Tenneco 400 include: lower flash point, lower gravity which would necessitate reformulation and probably relabeling, and the fact that the emulsification when using the Tenneco solvent is below standard performance. Some emulsifiers have already been tested in an attempt to improve the emulsification, but as yet, the problem has not been finally resolved; though, Igepal CO-990 appears to be a satisfactory replacement for the CO-970 now being used in the 4# Decamines.

Additional tests evaluating the properties of batches made from the Kemamine D-999 received in the 10-drum shipment indicate that this material did not perform as well as the original D-999 sample received. Thus, the specification on iodine value appears to be critical. The evaluation of Duomeen-O RD-3797 indicates that this material is much superior to standard Duomeen-O, performing about the same as the latest D-999 received. This is surprising, since the iodine value of the sample is very low.

Pilot samples of Nopco's emulsifier RDY were tested. Results were unsatisfactory since the performance of neither sample was equivalent to Igepal CO-970 as had been the initial RDY sample.

EXTENSION (APPROPRIATION NO. 6739)

Construction work increased during February, both on the tank farm and warehouse. The concrete work is now nearly complete, with the tank farm pad and warehouse periphery being the main areas of unfinished work. Following the initiation of overtime work on the piping, a much larger crew was obtained and this work started to move. At this writing, the tank farm piping is about 85-90% complete.

Work was started in other areas including the TCP area where the tank pad and building foundation were poured, the Fischer Building extension for which the excavation was underway at month's-end (the foundation is now in and the steel on the job site), and the DCP tank which was set in place on February 27th.

Engineering is proceeding on the details of piping, etc. Some segments of the proposed layouts have been reviewed and approved. Additional drawings presently are awaiting our review. Major activity was given to resolving the interference between the proposed layout and the existing conduits in the building. The best course of action was felt to be to relocate three pieces of equipment and associated piping and necessary changes to accomplish this are underway.

MISCELLANEOUS

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Some work was done on a few miscellaneous formulations including:

1. A 1.4# Decamine-D was prepared.
2. Checking the cold temperature stability of the 2#-1# Butyl-D/Iso-T for Unico.
3. Testing means of working off the mixed Decamines at Des Moines.

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No appropriations were closed during February, 1967.

F. Gordon Steward (c).

F. GORDON STEWARD

FGS/nc
3/8/67

OPERATING DATA - FEBRUARY, 1967

2,4-D	"D" Side	"T" Side	Totals
Average DCP Conversion, %	93.4	No	93.4
Average Cycle Time, Hours	8.9	Production	8.9
Average Cooking Time, Hours	2.0	This	2.0
Average Cooking Temperature, °C	103.6	Side	103.6
Usage #/# Product, DCP/MCA	.893/.620		.893/.620
Average Product Assay, %	99.4		99.4

MCA/DCP	MCA		DCP
Number of Batches	42		46
Average Batch Size, Lbs.	8,303		10,918
Average Reaction Time, Hours	7.2		12
Average/Maximum Reaction Temp. °C	116/132		77/91
Average Exit Gas Temperature, °C	-15		17
Usage #/# Product, Chlorine	.481		.514
Usage #/# Product, Acetic or Phenol	.658		.585
Product Assay, % MCA -	98.1	2,4-DCP -	93.4
DCA -	-0-	2,6-DCP -	6.2
Acetic -	0.9	o-Cl P -	0.4
Anhydride -	1.0	2,4,6-TCP -	-0-

HCl

Average Phenol Content, ppm	75
Average Sulfate Content, ppm	73

2,4,5-T

Average TCP Conversion, %	Not Determined
Average Cycle Time, Hours	
Average Cooking Time, Hours	
Average Cooking Temperature, °C	
Usage #/# TCP/MCA	.931/.573
Average Product Assay, %	None Assayed

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TCP

Number of Batches	37
Average Batch Size, Lbs.	2,417
Average Reaction/Digestion Time, Hrs.	1.9/5
Average/Maximum Autoclave Temp. °C	169/174
Maximum Temp. in Anisole Still, °C	105
Usage #/# Product, T CB	1.120
Methanol	.501
Caustic(Liq./Solid)	.427/.384

ESTERS	BUTYL-D	BUTYL-T	2-EH-D	2-EH-T
Number Batches	51	17	8	3
Average Batch Size, Lbs.	6,973	5,486	8,009	7,112
Average Cycle Time, Hours	23.6	24.6	21.0	20.0
Average Reaction Temperature, °C	143	137	152	159
Average Free Acid, %	0.8	1.5	1.7	
Average Color	2.2	-	-	-

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