2,4-D

With continued satisfactory operation of the 2,4-D Unit, technical activity in this area decreased. The quantity of wash liquor being recycled to the reactor was increased by 20% to 600 gallons per batch, with continued beneficial results. Upon completion of the analysis of some samples now in the Lab, results will be summarized and this study will be brought to a close.

Installation of the recorder, measuring sodium sulfate flow to the 6#6 was completed, and it is generally operating satisfactorily. Data on the quantity being used to wash the batches now being taken will be used as a basis for control of the sulfate flow.

An evaluation of the heat transfer rate in the present 2,4-D reactor indicates that our rate of heat transfer has increased over what was experienced in the dimpled jacket vessel. Some additional work is being done to determine the maximum heat transfer coefficient that might be expected, and how it can be achieved.

Two miscellaneous studies in the 2,4-D area were completed during the month:

1. A long-term evaluation of the use of plastic-lined valves to replace glass-lined valves, which showed the plastic-lined valves to be unsuitable at our service conditions. Plaskon handling molten 2,4-D at 150°C failed due to rupture of the lining, and Kynar at 105°C softened sufficiently that the lining at the weir was deformed, resulting in a leak.

2. An economic evaluation of the use of HCl to acidify 2,4-D in lieu of H_2SO_4 showed this to be a highly uneconomic procedure. At the present 2,4-D volume, the use of HCl would increase annual 2,4-D manufacturing costs by $36,000.

MCA/DCP/HCl

Operation of the DCP separator was hampered by an inability of the liquid to return continuously to the chlorinators, due to an inadequate liquid seal. Late in the month, the seal height was increased, but frequent plugging of the 1" line still resulted in unsatisfactory operation. Additional corrective piping changes are currently being made.

Muriatic acid produced during March was of good quality. In early April, however, for some as yet unexplained reason, the phenol content in the acid rose considerably above specs. A study currently underway indicates that the amount of chlorophenols discharged from the DCP Unit can vary markedly with chlorination conditions. Effort is now being given to determine how this variation and the unsatisfactory operation of the DCP separator relate to the current high phenol concentration in the Muriatic acid.

A study of the isomer distribution during a DCP chlorination was made. The isomer distribution when plotted against specific gravity gave a family of curves almost identical to curves developed in 1963 by Mr. R. F. Lindemann.

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One of the Teflon dip pipes installed in MCA service last Fall failed due to a defect in manufacture. It was replaced by the vendor at no cost, and the new unit has been installed in the chlorinator.

TCP

Tests to determine the effectiveness of p-dioxin removal indicated that the most active 2,3,7,8 isomer is the most easily absorbed of the impurities in the stream. A quantitative answer on the effectiveness of carbon is not yet available due to uncertainty over what part a filtration step in the test procedure was playing in the p-dioxin removal. Additional carbon tests will be run this month. Filtration tests will also be resumed, using a Fulflo cartridge-type filter.

A preliminary evaluation was made of piping changes necessary to convert Tank #131 for use as an anisole still. Further work in this area is awaiting review of the proposed changes.

DECAMINES

A number of emulsifiers were evaluated in an effort to obtain comparable emulsification in 2% Decamine at the lower amine ratio (.333#/# acid) to what we are now getting. Several acceptable emulsifiers were found. Major work on the Decamines was directed toward preparing a Decamine "wax" bar. A number of formulations were tried, and samples of several promising ones were sent to Cleveland for evaluation. We are awaiting word on how the samples appear before proceeding further on this project.

EXPANSION (APPROPRIATION NO. 6739)

Construction work on the main process changes was started in March, with the main areas of activity being relocation of the Cl_2 vaporizer, erection of the steel ~~for the TCP~~ and Fisher Building expansions, and work on the DCP transfer lines. ~~Progress~~ Progress in the Warehouse and tank farm areas slowed drastically toward the end of the month. Adverse weather accounted for part of the delay, but a more significant reason for the slow-down was the small number of men available for assignment to these areas of work.

The major area of work in the engineering phase of the project was directed toward modification of the project schedule to permit operation of the "T" Unit into July. Detailed engineering continued on piping, electrical, etc.

MISCELLANEOUS

The proposed formulation for Singlechot (Crop Rider 322) was checked and found to be OK. The first samples were prepared in the Lab and subsequently, about 180 gallons were prepared in the Plant and packaged in one-gallon cans for use in sample shipments.

Mixed mono, di, and trichloroacetic acids were prepared in the Lab, with no particular problem. The major change between this chlorination and a standard MCA run was that the pot temperature had to be [illegible] 160°-170°C in the late stages of the chlorination to keep the chlorine efficiency at an acceptable level. A manufacturing cost estimate for the preparation of the mixed chloroacetic acids was prepared based on the results of the lab chlorination.

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A visit was made to Cargill Labs to pick up some Texmil for shipment, and also make a cursory evaluation of how the production operation was going.

A pollution survey listing all plant effluents was completed, and it has now been forwarded to Central Engineering for inclusion in the Corporate report to the Manufacturing Chemists Association. Two representatives also attended an Air Pollution Conference held in Philadelphia.

One personnel change in the Technical Group occurred early in the month with the reassignment of J. J. Lusardi to the Semi-Works. He was replaced by Mr. D. R. Gehman who joined the Plant on March 5th.

No appropriations were closed during the month.

F. Gordon Steward

F. GORDON STEWARD

FGS/mc

4/12/47

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OPERATING DATA - MARCH, 1967

2,4-D

	"D" SIDE	"T" SIDE	TOTALS
Average DCP Conversion, %	95.4	No	95.4
Average Cycle Time, Hours	9.0	Production	9.0
Average Cooking Time, Hours	1.9	This	1.9
Average Cooking Temperature, °C	102.5	Side	102.5
Usage #/# Product, DCP/MCA	.853/.604		.853/.604
Average Product Assay, %	99.4		99.4

MCA/DCP

Number of Batches	54		49
Average Batch Size, Lbs.	8,303		10,904
Average Reaction Time, Hours	7.1		13.4
Average/Maximum Reaction Temp. °C	108/120		80/90
Average Exit Gas Temperature, °C	-18		20
Usage #/# Product, Chlorine	.457		.499
Usage #/# Product, Acetic or Phenol	.596		.575
Product Assay, % MCA -)		2,4-DCP -	94.4
DCA -)	None	2,6-DCP -	4.5
Acetic -)	Assayed	o-Cl p -	1.1
Anhydride -)		2,4,6-TCP-	0

HCl

Average Phenol Content, ppm 88)Levels were reduced by addition
Average Sulfate Content, ppm 33)of purchased acid.

2,4,5-T

Average TCP Conversion, %	73.5
Average Cycle Time, Hours	Not determined
Average Cooking Time, Hours	4.8
Average Cooking Temperature, °C	104
Usage #/# TCP/MCA	.902/.573
Average Product Assay, %	None Assayed

TCP

		TCP ASSAY	
Number of Batches	54	2,4,5-TCP -)	
Average Batch Size, Lbs.	2,458	2,4-DCP -)	None
Average Reaction/Digestion Time, Hrs.	1.9/5.0	2,6-DCP -)	Assayed
Average/Maximum Autoclave Temp. °C	167/175	Anisole -)	
Maximum Temp. in Anisole Still, °C	105		
Usage #/# Product, T_4CB	1.047		
Methanol	.502		
Caustic (Liq./Solid)	.415/.337		

ESTERS

	BUTYL-D	BUTYL-T	2-EH-D	2-EH-T
Number Batches	49	15	11	11
Average Batch Size, Lbs.	7,251	5,608	7,266	6,231
Average Cycle Time, Hours	25.3	22.5	20.7	24.7
Average Reaction Temperature, °C	144	139	156	154
Average Free Acid, %	0.7	0.0(ASTM)	1.3	2.0
Average Color	1.8	-	-	-

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