2,4-D

The study of the heat transfer in the 2,4-D reactor was completed. Results showed that the heat transfer coefficient of 48-57 BTU/hr., sq. ft., °F, that we are experiencing is in the range one would expect when cooling a thick slurry with water. The easiest means of increasing the heat transfer of the reactor is believed to be by increasing effective transfer area through use of cooling baffles. Major increases in water flow to the present jacket, or in agitation in the vessel, appear more difficult.

An evaluation of controlling the flow of H_2SO_4 to the acidification tank at a set ratio of the slurry flow to the tank was completed. Such a control system did not seem feasible at this time, due to the wide variations in slurry concentrations encountered.

MCA/DCP/HCl

The plugging problem at the DCP separator was eliminated by a piping revision to direct the returned material to only one chlorinator. The rate of collection of this separator is quite low, so that it is no problem to occasionally manually drop the returned liquid to the chlorinator.

Throughout the month, Muriatic Acid quality was poor with regard to contained chlorophenols and sulfates. Several things can be contributing to this problem, but as yet, we have not identified which, if any, is the major source. Problem areas noted include:

1. The demister in the DCP scrubber was found to be partially plugged with phenols, which most likely is the principle cause of the higher-than-normal sulfate content detected. The demister was cleaned and will henceforth be inspected for plugging whenever the sulfate content gets out of line.

2. The continued high-pressure drop across the new separator may have sufficiently changed gas velocities in the other collectors, so that their collection efficiency has been reduced. To evaluate this point, a bypass has just been installed around the separator to enable overall system performance to be checked at various pressure drops across the separator.

3. The chlorophenol content detected in the Muriatic Acid was found to vary from well in the hundreds of ppm (in one case, over a 1000 ppm) early in the chlorinations, to lows of about 30 ppm in the middle, with a subsequent rise at the end of the run.

   Correlation of the available data to run conditions is difficult; however, some trends appear when correlating average conditions by months. Though we know of no major change in DCP operating conditions, we will attempt to control variables such as exit gas and reactor temperatures more closely, to try to reduce the excessive phenol discharge early in the runs.

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D.P.

Tests to evaluate the effectiveness of both absorption on activated carbon and filtration are currently underway. Filtration was hampered by rapid plugging of the filter cartridges and though final analysis of the samples has not yet been completed, it appears that the p-dioxin removal of the properties used was not very good. The results of the carbon test appear much more encouraging, since complete removal of the 2,3,7,8-p-dioxin isomer can be effected. This test is continuing to determine the saturation point for the carbon bed.

Rapid plugging of the collector installed on the T.E.T. tank has become a problem. Efforts are underway to correct the situation with the main approach being to heat the tank top and nozzle in the vicinity of the collector sufficiently that crystals don't form and bridge across the opening to the collector.

Drawings of the new condenser for the methanol still were reviewed and approved. Delivery is scheduled for mid-July, so that the unit will be available for installation during the annual shutdown.

MECHANICAL

Evaluations continued of emulsifiers for use with the 2# Decamine-D at the lower amine-to-acid ratio. One (Chemsol CS-100-3) was superior to the others evaluated last month, and is recommended for use.

Two samples of alkyl diamines prepared by Hooker were evaluated. The results were interesting, since both the 2# and 4# Decamine-D's looked good (clear, fluid, and cold temperature stable), and because of an ether oxygen in the side chain, these materials might not be covered by Armour's patent. Additional testing of these diamines will continue when more material is received.

EXPANSION (APPROPRIATION NO. 2739)

Construction work on the process changes mainly centered around the installation of the new DCP tank which was put in service on May 2nd. The "D" centrifuge was removed and excavation started for a foundation to be installed where it had been located. The caustic tank was also set during the month.

Completion of the Warehouse was delayed at least two weeks due to the sprinkler contractor's failure to get started on the new sprinkler system. He has now finished, except for a test to be witnessed by the F.I.A. Thus, the Warehouse is essentially complete (a few external lights remain to be connected). Progress in completing the tank farm piping and lighting was almost non-existent, due to the diversion of available manpower to priority work such as the DCP system, and wiring within the building.

Total progress on the job in the immediate future is uncertain, since the Pipefitters struck on Monday, May 8th, over a jurisdictional dispute with the Ironworkers. Treadwell hopes to have the dispute settled shortly, and a meeting is scheduled to be held Thursday, May 11th, between the parties concerned. How long the Fitters can stay out without seriously affecting the job schedule is not known at this time.

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[illegible]

[illegible] for [illegible] and the U. S. [illegible], and [illegible] submitted to [illegible]. Also, [illegible] on the draft copy of [illegible] spec.

No appropriations were closed during the month.

[illegible]

[illegible]

5/[illegible]

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MAXUS1223279

2,4-D	"D" SIDE	"T" SIDE	TOTALS
Average TCP Conversion, %	94.4	No	94.4
Average Cycle Time, Hours	7.4	Production	7.4
Average Cooking Time, Hours	2.2	This	2.2
Average Cooking Temperature, °C	103.0	Side	103.0
Usage #/# Product, DCP/MCA	.869/.599		.869/.599
Average Product Assay, %	99.5		99.5

MCA/DCP	MCA	DCP
Number of Batches	62	51
Average Batch Size, Lbs.	8,303	10,904
Average Reaction Time, Hours	6.6	13.6
Average/Maximum Reaction Temp. °C	108/118	79/91
Average Exit Gas Temperature, °C	-15	20
Usage #/# Product, Chlorine	.457	.496
Usage #/# Product, Acetic or Phenol	.598	.581
Product Assay, % MCA-	99.2	2,4-DCP - 92.9
DCP-	- 0 - *	2,6-DCP - 6.7
Acetic-	0.7	o-Cl p - 0.4
Anhydride-	- 0 -	2,4,6-TCP - 0

* None detected, but some believed present.

ECl	
Average Phenol Content, ppm	175
Average Sulfate Content, ppm	78

2,4,5-T	
Average TCP Conversion, %	80.2
Average Cycle Time, Hours	6.7
Average Cooking Time, Hours	4.2
Average Cooking Temperature, °C	105
Usage #/# TCP/MCA	.880/.573
Average Product Assay, %	None Assayed

TCP		TCP Assay
Number of Batches	64	2,4,5-TCP - 91.6%
Average Batch Size, Lbs.	2,426	DCP - 3.7%
Average Reaction/Digestion Time, Hrs.	1.9/5.0	Anisole* - 0.7%
Average/Maximum Autoclave Temp. °C	170/174	p-Dioxin* 115 ppm
Maximum Temp. in Anisole Still, °C	105	* and related compounds.
Usage #/# Product, TLCE	1.083	
Methanol	.511	
Caustic (Liq./Solid)	.413/.306	

ESTERS	BUTYL-D	BUTYL-T	2-EH-D	2-EH-T
Number Batches	21	40	28	No
Average Batch Size, Lbs.	7,591	5,364	7,555	Production
Average Cycle Time, Hours	26.2	20.4	20.4	
Average Reaction Temperature, °C	140	141	152	
Average Free Acid, %	0.8	0.0(ASTM)	1.8	
Average Color	2.2	-		

FGS/nc
5/10/67