2,4-D

Operation of the 2,4-D Unit was closely followed during the month. Particular attention was given to eliminating the effects of the recycle of wash liquor from the End to the reactor which was continued throughout January. Data indicate that 2,4-DCP and 2,4-D totalling about 160 pounds are being recycled to the reactor, and that neither product quality or reactor conversions have been adversely affected by the recycle. The recycle of the wash liquor is being continued and additional data on its performance is being taken to substantiate these observations.

Since the problem in controlling the losses from the End has been lack of adequate control of the sulfate fed to the sprays, caution is being taken to provide a means of controlling the sulfate flow. An orifice, transmitter, and recorder available in the Plant will be installed in the sulfate line to give a basis for manual control of the sulfate flow.

We again had some problems with the filter cloths in use in 2,4-D. Several End cloths apparently shrank due to an undetermined cause. The cloths used in the guncotton filter also were failing more rapidly due to the inability of the Dacron cloths to withstand exposure to the acidic wash column discharge that was being filtered. This problem can be corrected by switching to polypropylene which can be used both in acidic and alkaline service.

The Teflon-lined pump installed in December failed after 5 weeks' operation. Apparently abrasion caused a failure of the Teflon lining on the impeller shaft, resulting in rapid corrosion of the underlying metal and failure of the pump when the impeller fell off. Since the Teflon was in good condition throughout the rest of the pump, it will be equipped with a Hastelloy impeller and tried again.

Tests evaluating the use of a centrifuge in lieu of a rotary vacuum filter run both in our Lab and by Acetek do not look encouraging. Though the decision to examine centrifuging was based on the hope that centrifuging, particularly when using counter-current wash techniques would result in lower DCP content, this has not been the case. Data from the Lab tests indicate that the DCP content found has been higher than typical filtered 2,4-D. Information obtained will be re-evaluated to determine whether it will be worthwhile to continue these tests.

An evaluation of using the Tote-Tin system to ship 2,4-D to Des Moines was completed. This study indicated that both due to high capital cost and higher operating costs, use of this system was uneconomical.

MCA/DCP/TCP

Work continued in our effort to improve the quality of the Muriatic Acid product. The height of the DCP stripper was increased by 3', and the packing depth increased by a like amount, which reduced the chloropheral content somewhat. The new vapor-liquid separator was received and it will be installed shortly in the DCP line leading from the DCP distillation to trap additional organic carry-over and return it to the chlorinator.

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TCP

Additional tests to determine the effectiveness of activated carbon in the removal of p-dioxin from TCP were run in the Lab. These tests were directed toward determining the saturation point for p-dioxin on the carbon, and also the residence time required for absorption to be complete. Sufficient data are not yet available to ascertain the points under investigation. Additional tests on the effectiveness of carbon treatment to remove p-dioxin as well as more filtration tests will be run in February.

DACAMINES

Two Plant-scale test batches of Dacamines made with Kemamine D-999 were prepared in January. Two additional batches using alternate formulations will be prepared shortly.

Lab tests of the sample of Nopco KDT were completed and it was found to be comparable to the CO-970 now in use. As has been previously reported, Nopco is going to prepare a larger sample in their pilot plant for further tests by us. Several other emulsifiers were tested in a continuing effort to obtain an emulsifier which will give good bloom as well as a stable emulsion. Two ethoxylated oleyl alcohols and Stepan Agent 365-17 were tried, but they did not give the desired results. In all three cases, the performance of these emulsifiers was essentially comparable to the CO-970 now in use.

An attempt was made to lower the amine-to-acid ratio in Dacamine 4D to .25#/#, using some of the newer emulsifiers. Though stable formulations could be prepared, none had satisfactory emulsification.

The proposed formulations based on Kemamine D-999 were all tried with Uran 32, but the results were no better than with standard material. The Dacamine on standing separates as a gelatinous layer which cannot be readily redispersed.

Tests of Tenneco 400 and Armour's Duomeen-O-HD-5797 were started. Both materials result in Dacamines with some interesting properties. Additional evaluation must await completion of the tests.

EXPANSION (APPROPRIATION NO. 6729)

Construction activity accelerated markedly during January. Once the concrete contractor adequately manned the job, good progress was made. As of this writing, the work is nearing completion. The tank farm only requires completion of the slab and rebuilding of the dike, while the warehouse will be essentially finished when the last section of wall (about 25') and the other half of the pad are poured.

The ester tanks were all received in January and installed. Two tanks were turned over to the Plant here in the month for dead storage of Butyl-D and 2-EH-D esters. Piping has been underway since January 12th and to date progress has been very slow. This appears to be the main problem area.

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OPERATING DATA - JANUARY, 1967

2,4-D	"D" Side	"T" Side	Totals
Average DCP Conversion, %	93.7	92.4	93.1
Average Cycle Time, Hours	7.9	11.5	-
Average Cooking Time, Hours	2.2	5.0	-
Average Cooking Temperature, °C	103.6	104.8	-
Usage #/# Product, DCP/MCA	-	-	.918/.609
Average Product Assay, %	-	-	99.4

MCA/DCP	MCA	DCP	
Number of Batches	62	69	
Average Batch Size, Lbs.	8,303	10,835	
Average Reaction Time, Hours	7.0	10.9	
Average/Maximum Reaction Temp. °C	110/121	81/110	
Average Exit Gas Temperature, °C	-17	23	
Usage #/# Product, Chlorine	.431	.514	
Usage #/# Product, Acetic or Phenol	.616	.580	
Product Assay, %	None Assayed	- 2,4-DCP	- 90.2
		- 2,6-DCP	- 6.2
		- o-Cl P	- 1.8
		- 2,4,6-TCP	- 1.8

HCl

Average Phenol Content, ppm	125
Average Sulfate Content, ppm	51

2,4,5-T — No Production.

- Average TCP ~~Conversion, %~~
- Average Cycle Time, Hours
- Average Co~~oking Time~~, Hours.
- Average Co~~oking Temperature~~, °C
- Usage #/# TCP/MCA
- Average Product Assay, %

TCP	
Number of Batches	17
Average Batch Size, Lbs.	2,290
Average Reaction/Digestion Time, Hr.	2.1/5
Average/Maximum Autoclave Temp. °C	165/174
Maximum Temp. in Anisole Still, °C	105
Usage #/# Product, NaOH	1.090
Methanol	.611
Caustic (Liq./Sol.)	.161/.353

ESTERS	BUTYL-D	2-EH-D	BUTYL-T
Number Batches	56	20	2
Average Batch Size, Lbs.	6,702	7,546	7,166
Average Cycle Time, Hours	25	21	18.5
Average Reaction Temp.	140	150	135

FGS/[illegible]
2/8/67