

OPERATING CONCEPTS
PLANT TESTS
NOVEMBER, 1966

OPERATING DATA

Operating and analytical data summarizing some aspects of Plant operations during November are attached as an addendum to this report.

2,4-D

Work directed toward the elimination of DCP from our flake acid is currently underway. Major emphasis at this time is being given to obtaining an accurate material balance around the 6x6 filter to determine the DCP removal during the filtration, and the quantity of acid lost from this part of our system. The balances obtained on the runs made to date have not been very good, primarily due to the difficulty of obtaining representative samples from the system. The sampling procedures were modified, and additional runs are being made. Hopefully, a satisfactory balance will be obtained shortly and it will be the basis for an evaluation of the use of double filtration of the 2,4-D.

A filter cloth recommended as being less prone to blinding than the cloth presently being used was tested in the Plant. Though Lab tests had indicated that it gave equivalent performance to the filter cloth now being used, it was found to be unsatisfactory for Plant use. The major problem was that cake retention was poor due to random flaws inherent in the weave of the cloth.

The corrosion tests evaluating a number of alloys for possible use in the 2,4-D system have been completed. Based on the results obtained, cost, and availability of the alloys, it was recommended that any alloy pumps for the desalter service be made of Durimet-20. However, it is hoped that the Gaulds Teflon-lined pump which has now been received, will perform satisfactorily. If so, it will be used in our more corrosive environments.

An economic study evaluating the use of HCl in lieu of H₂SO₄ in the acidification of 2,4-D has been started (it should be finished in December). The spare agitator for the acidification tank ordered on Appropriation Number 6741-13 was also received during November.

MHA/ICP/EC1

The quality of the Muriatic Acid produced during November again presented a problem. It is felt that two causes primarily account for the higher-than-desired phenol and sulfate content in the acid, namely:

1. Inadequate mist elimination in the present 10" mist eliminator, allowing passage of sulfuric acid contaminated with phenols into the Muriatic Acid.
2. Excessive carry-over of phenols from the ICP Unit to the sulfuric stripper.

DS 00000998

To alleviate the preceding problems, a new 24" condenser was ordered (it is being installed today) to provide more effective removal of malformic acid from the gas stream. It is being installed upstream from the 10" condenser which will be left in service. Also, quotations are presently being obtained on the cost of fabricating a liquid-vapor separator of improved design, which will be installed to minimize carry-over of phenols from the TGP Unit.

TGP

An appropriation request for a new condenser for the methanol still was submitted on November 22, 1956, and we are awaiting approval before proceeding further with this.

Tests on the removal of p-dioxin from TGP were continued in both the Lab and in the Plant. Because plugging had presented problems on previous runs, a test was run in the Lab to determine whether the residence time between dilution and filtration affected the filtration efficiency. Results were encouraging, since it was found that p-dioxin removal was independent of time, while the removal of amide (which we don't want to remove), increased with time. Thus, the proposed use of an in-line mixer with immediate filtration would appear beneficial. This test will be reported to confirm these findings.

The Plant tests indicate that considerable p-dioxin (and related isomers) can be removed by filtration, though passage of one isomer is more than desired. However, fairly rapid plugging of the filters, plus an unexplained reduction in filtration efficiency after about 2 hours' operation, still present problems. The latter problem is particularly puzzling, since the pressure drop across the filters was not indicative of failure of the element. To investigate and try to solve these problems, tests will be continued in the following areas:

1. Filtration with filters of two different sizes and parasites in series to effect maximum removal of solids without rapid plugging.
2. Lab tests will be started to determine whether the addition of activated carbon to a filter could aid in removal of the p-dioxin. Filtration will be carried out not only at the very dilute strengths we have been using, but at concentrations up to the normal operating strength of 20%.

DECAMINES

The sample of Armour's specially-prepared Duocren-0-3522 was evaluated and found to be unacceptable for the Decamines, since it produced a product only slightly better than standard Duocren-0. Following the completion of this test, the results of all the Decamine tests were summarized in memo by M. Rosenfeld and F. G. Steward, recommending that:

1. We switch from Duocren-0 to Huzko's Kenamine D-999.
2. We change the amine/acid ratio in 2# Decamines from .425[#]/_# to .333[#]/_#.

- 3. The elimination of the cyclohexanone coupling solvent from the 4[#] Examiners be seriously considered, along with the possibility of switching emulsifiers in the 2[#] Examiners from Emcol AD6-45 to Igepal CO-970.

Late in November, Amour's Sales Representative called. He had no explanation as to why the sample they submitted was so far from what we need. He said they would get on the preparation of another sample to meet our specific needs. Though any additional samples will, of course, be evaluated, this does not alter the recommendation to switch dioximes in view of Amour's previous poor performance in meeting our requirements.

EXPANSION (APPROPRIATION NO. 6739)

Construction during November was delayed about two weeks, awaiting the selection of a concrete contractor. Work was finally started on November 21st, but progress has been slow due to the small work crew employed by the contractor. IACO and Treadwell supervision is working to have him get a more adequate crew on the job. The tanks are scheduled for delivery the week of December 19th, and the Warehouse has been delayed by the vendor to the week of January 8, 1967. Design work on the tank farm and Warehouse, though lagging somewhat, should meet these schedules.

Selection of major equipment continues with a number of items either on order or about ready to order. We are nearing completion of our review of the flow sheets. Layout and arrangement design is now underway.

MISCELLANEOUS

As a hold-over from our work on 3,4-DBA, a corrosion test of 316 stainless steel in the molten product is being run. Results will be forwarded to the Semi-Works when available.

Appropriations closed during November:

No. 6755-91	Fluid Meter	- \$522	expanded
No. 6755-9	Dial Scale	- 680	"
No. 6741-10	Teflon Spargers & Dip Pipes	- 590	"
No. 6741-12	Motor and Impeller-Mixer	- 490	"
No. 6741-14	Anisole Condenser	- 910	"

F. Gordon Steward
F. GORDON STEWARD

FGS/nc

12/7/66

DS 00001000

OPERATING DATA - NOVEMBER

<u>2,4-D</u>	<u>"D" Side</u>	<u>"T" Side</u>	<u>Totals</u>
Average DCP Conversion, %	93.5	90.3	92.6
Average Cycle Time, Hours	6.4	8.0	-
Average Cooking Time, Hours	1.8	2.3	-
Average Cooking Temperature, °C	103	105	-
Usage #/# Product, DCP/MCA	-	-	.930/.648
Average Product Assay, %	-	-	99.3

<u>MCA/DCP</u>	<u>MCA</u>	<u>DCP</u>
Number of Batches	71	77
Average Batch Size, Lbs.	8,490	10,812
Average Reaction Time, Hours	7.1	-
Average/Maximum Reaction Temp. °C	107/124	82/92
Average Exit Gas Temperature, °C	-15	22
Usage #/# Product, Chlorine	.395	.462
Usage #/# Product, Acetic or Phenol	.593	.580
Product Assay, % (2 samples)	MCA - 99.2	2,4-DCP - 89.2
	DCA - Nil	2,6-DCP - 6.9
	Acetic - 0.8	c-Cl P - 1.3
		2,4,6-TCP - 2.6

HCl

Average Phenol Content, ppm	130
Average Sulfate Content, ppm	71

2,4,5-T

Average TCP Conversion, %	74.0
Average Cycle Time, Hours	8.9
Average Cooking Time, Hours	4.9
Average Cooking Temperature, °C	103
Usage #/# TCP/MCA	.929/.590
Average Product Assay, %	None Assayed

TCP

Number of Batches	18
Average Batch Size, Lbs.	2,660
Average Reaction/Digestion Time, Hour	2/5
Average/Maximum Autoclave Temp. °C	166/180
Maximum Temp. in Anisole Still, °C	105
Usage #/# Product, T ₄ OB	1.120
	Methanol .611
	Caustic (Liq./Solid) .412/.320

ESTERS

<u>ESTERS</u>	<u>BUTYL-D</u>	<u>2-EH-D</u>
Number Batches	34	22
Average Batch Size, Lbs.	DS 07 213 1001	7,776
Average Cycle Time, Hours	24	22
Average Reaction Temperature, °C	141	154

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12/7/66