OPERATING COMMENTS
PLANT TECHNICAL
SEPTEMBER, 1967

## 2,4-D

Production of 2,4-D was continued for the first two weeks of September. During the second week, Na 2,4-D slurry was prepared for the first production drying run at Custom Processing. Preparation of the slurry did not proceed as well as the single batch prepared in August. A major problem was our inability to produce uniform slurry of the desired concentration --- most batches were too thick and apparently varied within the batch. This problem accentuated pumping problems that we were experiencing, and led to frequent plugging of the transfer lines. Thus, the filtration time already lengthened to allow for double filtration was further extended.

Start-up of the drying operation was also hampered by the high slurry concentration, but once conditions were again established, drying proceeded fairly well. Some variation in moisture content in the Na 2,4-D was noted, with product containing more than about 8% water, exhibiting some "stickiness" and tendency to agglomerate. A few drums had moisture contents above the 11.9% maximum and were noticeably wet.

Based on the experience gained in this run, it is evident that separate facilities for the second filtration and slurry preparation should be provided so that 2,4-D capacity is not reduced when processing Na 2,4-D. Such facilities along with a spray dryer should be installed if we desire to continue marketing the 95% Na 2,4-D.

## 2,4,5-T

Production of 2,4,5-T started on September 18th, with Engineering assistance being given during the start-up. Many problems came up which were resolved by the collective efforts of Production and Technical personnel. Some problems which demanded particular attention were:

1. Maintaining an even pressure on the various sections of the Unit --- solved by air-padding the Unit.

2. Pumping problems which primarily seemed to be due to trash remaining from construction plugging the pumps.

3. Delays in heating up caused by the poor state of the slurry tank which is soon to be replaced.

4. Repeated plugging of lines and valves which was attacked by changing to diaphragm valves, rerouting lines, improving steam tracing, and installation of more steam-out points. This was our biggest problem and still causes trouble.

Notwithstanding the problems by month's end, production could be maintained if careful attention were paid to the operating conditions of the Unit. Changes are currently being made to alleviate the plugging problems with the most important being the [illegible] of the settling tank.

MCA/DCP/HCl

Some additional work was done on developing an accurate chromatographic method for determining DCA content in our MCA. A number of different substances were tried as internal standards, but we still would like to improve the accuracy of our results. Work continues in this area in conjunction with some chlorination studies now underway.

A new sampler was installed for use on the DCP Unit, but it had not been tried at month's end, since the Unit had been down. Again, no work was done on the HCl quality study.

TCP

As previously indicated, a 12" diameter pilot carbon tower was built to treat TCP in inventory when we shut down TCP production in July. Something over 25,000 gallons of TCP were treated in this column, with the effluent running about 1 ppm p-dioxin. Pressure drop in the column remained low throughout the run. The slight amount of solid carry-over noticed was easily removed by a secondary filter even at superficial velocities greater than 1000 lbs./hr., square foot, in the column.

The process piping in the TCP purification area was completed on September 15th. Processing of some dilute leach water was attempted on September 16th, but a gasket leak on the acidification tank delayed operations. First purification of substantial quantities of TCP was carried out September 22nd. Due to the fact that instrumentation at that time had not been finished (it was finished at the end of September), the TCP was diluted batch-wise in the intermediate storage tank and run through the carbon tower to storage. Using this operating procedure, performance of the carbon tower has been very good to date, with all p-dioxin levels reported running <1 ppm. A check of "T" acid produced also indicated a low p-dioxin content.

DACAMINES

Evaluation of several alternate materials for Dacamine production was the major work area during September. A sample of a "high" IV diamine received from Armour was tested. Our results on the iodine value test were no higher than for standard Duomeen-O and performance of the material is only slightly better than standard Duomeen-O. A sample of the Kemamine D-999 shipment was also checked and again, we found an IV at 85.3 much lower than the 99+ they indicated. Performance in cold temperature stability of this material, though better than Duomeen-O, did not come up to the earlier sample of Kemamine D-999 evaluated. We have also requested a copy of the analytical procedure used by Humko, to try to resolve the discrepancy in IV noted.

The evaluation of Nopco diamines indicates that their RH diamine could be used in the Dacamines. Its use, besides the obvious advantage of buying within Diamond, might also allow the elimination of cyclohexanone from the Dacamine-4D formulation. More work should be done in this area.

Additional work was done on the special Dacamine-4T, Dacagin formulation.

EXPANSION (APPROPRIATION NO. 6739)

As already indicated, the piping work in TCP was completed September 15th, and the instrumentation at the end of the month. Installation of the permanent carbon tower and completion of the insulation remain to be finished. September saw the big push in piping work on the 2,4-D Unit. Much progress was made during the month, but work still continues in this area, particularly instrumentation piping. Because of the low efficiency exhibited by the Pipefitters, arrangements are now being made to remove them from the job and finish up with our men, augumented by outside help under our direction.

MISCELLANEOUS

Some work was done on the Singleshot formulation to better clarify the nature of this material so that the patent position can be fully evaluated. Several complaint investigations were also completed during the month.

Absences among the Technical personnel due to vacations and illness totalled five weeks during September.

No appropriations were closed in September.

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## 2,4-D

| | |
|---|---|
| Average DCP Conversion, % | 96.1 |
| Average Cycle Time, Hours | 10.8 |
| Average Cooking Time, Hours | 1.7 |
| Average Cooking Temperature, °C | 102.2 |
| Usage #/# Product, DCP/MCA | .829/.615 |
| Average Product Assay, % | None Flaked |

## MCA/DCP

| | MCA | DCP |
|---|---|---|
| Number of Batches | 28 | 14 |
| Average Batch Size, Lbs. | 8,303 | 10,714 |
| Average Reaction Time, Hours | 6.6 | 14.7 |
| Average/Maximum Reaction Temp. °C | 112/121 | 83/94 (Inst. out of cali- |
| Average Exit Gas Temperature, °C | -16 | 18 bration) |
| Usage #/# Product, Chlorine | .395 | .496 |
| Usage #/# Product, Acetic or Phenol | .603 | .580 |
| Product Assay, % MCA) | | 2,4-DCP-) |
| DCA) | None | 2,6-DCP-) None |
| Acetic) | Assayed | o-Cl-p-) Assayed |
| Anhydride) | | 2,4,6-TCP-) |

## HCl

| | |
|---|---|
| Average Phenol Content, ppm | 116 |
| Average Sulfate Content, ppm | 63 |

## 2,4,5-T

| | |
|---|---|
| Average TCP Conversion, % | 80.4 |
| Average Cycle Time, Hours | 13.4 |
| Average Cooking Time, Hours | 6.5 |
| Average Cooking Temperature, °C | 97.0 |
| Usage #/# TCP/MCA | .930/.583 |
| Average Product Assay, % | None Assayed |

## TCP

| | |
|---|---|
| Number of Batches | 25 |
| Average Batch Size, Lbs. | 2,311 |
| Average Reaction/Digestion Time, Hrs. | 2/5.2 |
| Average/Maximum Autoclave Temp. °C | 164/173 |
| Maximum Temp. in Anisole Still, °C | 105 |
| Usage #/# Product, TetCB | 1.100 |
| Methanol | .568 |
| Caustic (Liq./Solid) | .441/.319 |

## ESTERS

| | BUTYL-D | BUTYL-T | 2-EH-D | 2-EH-T |
|---|---|---|---|---|
| Number of Batches | 80 | 4 | | |
| Average Batch Size, Lbs. | 7,554 | 4,707 | NO PRODUCTION | |
| Average Cycle Time, Hours | 24.2 | 27.5 | | |
| Average Reaction Temperature, °C | 147 | 145 | | |
| Average Free Acid, % | 1.0 | 0 (ASTM) | | |
| Average Color | - | - | | |

DS



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