

MEMORANDUM FOR THE DIRECTOR  
PLANT OPERATIONS  
MAY, 1967

2.4-D

Major emphasis during the month was directed toward finding a means of spray drying Ra-2,4-D. Initial consideration was given to the possibility of drying the material at Kearny. However, a visit to Kearny indicated that use of either of their dryers was impractical, since the units are committed to current production, and clean-out problems would be severe.

Following elimination of Kearny from consideration, efforts were directed toward finding a custom dryer who could dry the Ra-2,4-D. Of several concerns contacted, the only one still under active consideration is Custom Processing of Trenton.

At the suggestion of Purchasing, Regoo was contacted. Though they can't dry the material in equipment now operating, they said that they still have most of the components of a dryer dismantled last year. Use of this equipment as the nucleus of a spray drying facility at Newark might enable us to enter the Ra-2,4-D market on favorable terms, with a minimum capital investment. This possibility will be explored further as soon as we are informed of the procedure to be followed in making additional contact with Regoo.

We have again examined the problem of poor ventilation in the Flaker room. Changes will be made shortly to slope and flush the vent duct from the flaker in the hope that this will eliminate the plugging of this duct. Turbine performance of the bag packer is also being studied. It is felt that the main cause of our problem is the formation of lumps in the bin, which restrict flow to the packer. Efforts will be continued in this area.

MEA/BSP/MSL

The quality of Mariotte acid continued generally unsatisfactory during May, with both phenols and sulfates running higher than desired. The mist eliminator was found to be plugging rapidly with chlorophenols, and it was cleaned twice during the month. Sulfate level, however, still continued high.

Continued examination of the performance of the cyclone separator indicated that the pressure drop across this unit was not as great as first was thought. Tests of the performance of this unit showed that the quantity of chlorophenols removed were not great enough to bring the Mariotte acid into spec. This cyclone can remove the equivalent of about 15 to 25 ppm of phenols in our acid, so at best, it can only be effective as the final clean-up of a generally clear stream.

Because of the failure of our efforts to date to consistently produce specification acid, efforts will be directed toward:

1. Modification of operating procedures to minimize carry-over of chlorophenols from the BSP chlorinators.
2. Evaluation of <sup>DS</sup> previously untried methods of stripping the phenol contaminants from the gas stream such as solvent extraction or freezing them out in a refrigerated condenser or crystallizer.

STEWARD -15 ID

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The work-up of samples remaining from Filtration Work was in April and indicated that in no case was removal of the  $2.5, 7.8$  isomer complete. An additional test using a process on the filter element was made in May, but results have not yet been analyzed. However, in this last run, as with in all earlier runs, rapid plugging of the filter element was observed. Thus, use of relatively small diaphragm filters as the primary stage of TSP purification does not appear feasible, and work directed in this area has been discontinued.

Analysis directed toward the development of a large column for the removal of  $2.5, 7.8$  isomer by absorption on activated carbon has not been completed. The test work with a small diameter column of  $100 \mu$  diameter and a length of 100 cm. indicated that a bed of about 75 grams of activated carbon could remove 500 mg of  $2.5, 7.8$  isomer from a feed which would be produced in the plant. The only operating problem in that the column gradually plugs with solid largely thought to be ferrous hydroxide. Ferriatic acid has been used to remove these solids and "regenerate" the bed. This test is now continuing after the second regeneration.

Efforts taken to position additional heat in the vicinity of the TSP collector did not eliminate the rapid plugging of this unit. Analysis of some of the caked TSP removed from the collector showed  $2.5, 7.8$  isomer. Since the presence of water is known to cause plugging in the collector, a search is now underway to see whether there is a leak in some of the insulated pipe or the steam coil in the tank.

#### DIAMINES

Test work on the Diamines was at a low ebb pending the arrival of additional diamine and oxidant samples from Hopes. A meeting was held with representatives of Hunk concerning our requirements as to boiling value of the diamine and what they can reasonably expect to make. Following this meeting, they proposed that the minimum IV spec on their Diamine D-999 be lowered from 95 to 94. It is our feeling that D-999 at a minimum IV of 94 should be acceptable, but that we should hold strictly to this value, since further lowering of the IV might be detrimental to the Diamine properties.

#### EXPANSION (APPROXIMATE NO. 6739)

Construction work on the warehouse and tank area were finally completed during May. Main emphasis on the process changes was directed toward installation of the new vacuum pump and hot water system to enable early removal of existing units that must be relocated. Installation of power wiring within the building is now well underway as is piping work near the new acoustic tank. Skidways for the TSP purification unit has also been installed.

Engineering work is essentially complete. Some further checking of the construction schedule with regard to alternate manning levels was finished during the month.

DS 000070 94

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DS 00001074

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MEMORANDUM

Chlorination of a larger sample of mixed chloroacetic acids was completed and this material has been given to the Sales Department for delivery to the Toms River Chemical Company. Work directed toward the modification of the proposed formulation of a 2% emulsifiable concentrate of 2,4-D is under way, and a revised formulation should be available shortly.

Two complaints were evaluated during the month. One, a problem with Dacazine-D plugging spray equipment which was attributed to residual flocc acid which has not been fully dissolved at the time of manufacture. The second was a request by Harshah to examine a formulated 2-D-D sample for possible Tordion contamination. No Tordion was found, but the 2-D-D level was significantly higher than in our retained sample.

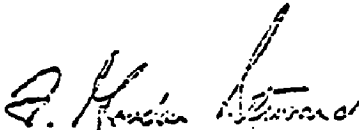
Increased use of the new chromatograph which was installed in April widened our chromatographic capabilities considerably. New power wiring was installed to the Instrument Room in May to minimize the possibility of overloading any one circuit.

An Engineer attended a Chemical Industry Council pollution workshop to keep abreast of developments in this area. New legislation presently awaiting the Governor's signature is being reviewed to assess its possible impact on the Plant.

No appropriations were closed in May.

EGS/xc

6/7/67

  
F. GORDON STEWARD

DS 00001075

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2.4-D

* Average DCP Conversion, %	93.9
Average Cycle Time, Hours	7.3
Average Cooking Time, Hours	1.8
Average Cooking Temperature, °C	102.5
Usage #/# Product, DCP/MCA	.852/.585
Average Product Assay, %	99.3

MCA/DCP

MCA

DCP

Number of Batches	66	65
Average Batch Size, Lbs.	8,368	10,862
Average Reaction Time, Hours	6.7	14.6**
Average/Maximum Reaction Temp. °C	110/116	80/89
Average Exit Gas Temperature, °C	-17	19
Usage #/# Product, Chlorine	.457	.456
Usage #/# Product, Acetic or Phenol	.595	.520
MCA-)	99.5	2,4-DCP - 92.6
DCM-)	Trace†	2,6-DCP - 6.5
Acetic-)	1.5	o-Cl <sub>2</sub> - 0.5
Anhydride-)	-0-	2,4,6-TCP - 0.4

\* DCP assay believed to be low.  
 \*\* Time somewhat long due to running only on recovered Cl<sub>2</sub> for 28% of the time.

ECL

Average Phenol Content, ppm	149
Average Sulfate Content, ppm	102

2,4,5-T

Average TCP Conversion, %	79.3
Average Cycle Time, Hours	6.5
Average Cooking Time, Hours	4.0
Average Cooking Temperature, °C	104
Usage #/# TCP/MCA	.832/.549
Average Product Assay, %	None Assayed.

TCP

Number of batches	74	<u>TCP Assay</u>	
Average Batch Size, Lbs.	2,351	2,4,5-TCP	- 87.0
Average Reaction/Digestion Time, Hrs.	1.9/5.1	DCP	- 1.4
Average/Maximum Autoclave Temp. °C	169/174	Anisole†	- 0.5
Maximum Temp. in Anisole Still, °C	105	p-dioxin†	- 71 ppm
Usage #/# Product, Et <sub>2</sub> O	1.093	* and related compounds.	
Methanol	.557		
Caustic (liq./Solid)	.434/.316		

ESMERS

Number Batches	DS	<u>ESMERS-D</u>	<u>ESMERS-T</u>	<u>2-EE-D</u>	<u>2-EE-T</u>
Average Batch Size, Lbs.	10001076	55	41	22	20
Average Cycle Time, Hours	19.7	7,574	5,568	7,568	Production
Average Reaction Temperature, °C	142	19.7	21.7	19.2	
Average Free Acid, %	0.8	146	146	157	
Average Color	-	0.8	0.0 (ASEN)	1.8	

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FCS/cc  
6/7/67