

OPERATING CONCERNS
PLANT TECHNICAL
JULY, 1965

2,4-D

The 2,4-D area was quite active during the month, with a number of studies underway. Brief studies in the Laboratory indicated that filtration at about 50°-55°C generally should be preferable to filtration at higher temperatures. Plant tests on sulfate consumption showed that about 1,100 gallons of H_2SO_4 were used to wash each batch, and that the concentration varied from 4.3% to 16% H_2SO_4 . This information was passed on to the Production Department for their use.

Data on the performance of the Larra dryer is being taken for use in detailed scale-up calculations for the new "D" Unit. Two runs indicate that we are getting an overall heat transfer coefficient of about 260 BTU/hour ft.², °F. Additional data will be taken at a higher steam pressure and the unit size will be based on the average of the results.

Tests on storing molten 2,4-D are now underway in glass, stainless steel, and lead. Though the tests have not been completed, results appear unsatisfactory, since significant darkening of the product was observed in all cases, and the 2,4-D seems to react with the lead.

Tests to obtain the necessary information required to calculate the scale-up of the reactor agitator were completed, and the data forwarded to a vendor for evaluation. Based on his preliminary evaluation, we should have no significant problem with the scale-up.

Assistance was provided the Production Department in laying out the changes necessary to convert the #5 esterifier to a temporary acidification tank following failure of the acidification tank agitator. A study to determine the major cost centers contributing to the large negative variance in the 2,4-D maintenance accounts is also underway.

MCA/DCP

The Teflon spargers for use in the MCA chlorinators were received, however, we are still awaiting delivery of the Teflon sample dip pipes. Installation of the spargers is planned during shutdown.

An appropriation request (No. 6743) for a new DCP scrubber was submitted and approved. Necessary materials were procured, and arrangements made for the unit to be fabricated in a local shop, to lessen the work load on the Plant force. Installation of this unit, which is scheduled during the 2nd week of shutdown, should lower the phenol content of our acid, thus eliminating a serious barrier to wider marketing of our acid.

A preliminary review was made of proposed standard analytical procedures for Muriatic Acid submitted by the Industrial Chemicals Division. Our concern over the exclusion, from the proposed standard procedures, of the UV procedure for determining phenols now used at Newark, was related to them as well as several general questions concerning the application of the tests

(particularly who would determine what tests would be run at each location).

TCP

One Plant test of the removal of p-dioxin from TCP by filtration was completed with encouraging results. About 700 gallons of 11.6% TCP solution were filtered through a small 10-micron cartridge filter, reducing the p-dioxin level from 48 ppm to 8 ppm on a 100% TCP basis. The anisole and related impurities were not removed during the filtration, and no appreciable cake build-up was detected. A second run made early in August using a 5-micron filter was less successful, even though essentially all the dioxin was removed, since the filter plugged (presumably with anisole) after a short time. Analysis of data from this second run continues, and more tests are planned to clearly define the operating conditions required to remove the p-dioxin without plugging or other operational problems.

While working up TCP samples for analysis, two impurities, apparently closely related to anisole, were detected in about the same concentration as the anisole. An attempt is being made to isolate and identify these compounds.

ESTERS

Lab tests on the production of esters at higher temperatures and various catalyst levels were completed. Though the reaction rate increased with temperature, a catalyst will definitely be required even at high temperatures, to achieve rapid and complete reaction. Runs using catalyst were from 92% to 99% complete in one-half hour, compared to 48% to 57% completion for the runs without catalyst. The no-catalyst runs were only 82% to 89% complete after two hours' reaction. Though beneficial with regard to reaction rate, higher temperature and catalyst levels also promote darkening of the ester, so their use must be approached with caution. In addition to the work underway at Desark, Research has been provided copies of the current Operating Instructions and the data recently obtained, so that they can evaluate the state of the process and prepare recommendations on what future work is necessary.

Quotations from two manufacturers of hot oil units indicate that a 600,000 BTU/hr. unit (which would be sufficient to run two esterifiers) would cost about \$10,000 to \$13,000, depending on the design. Consideration of the installation of such a unit is continuing in light of the information listed above.

Analysis of three samples of Monsanto ester shipped to Des Moines this spring has been completed. Our assays, averaging the saponification value and total chlorine assays, following correction for DCP, were somewhat lower in all cases than the results reported by Monsanto. The Monsanto procedure requires assay by total chlorine only, so it is less stringent than our procedure, and would result in significant errors when assaying esters containing substantial quantities of DCP. Thus, the "over-standard material" sold by some of our competitors can be partly attributed to less stringent analytical techniques.

DACIMINES

Though we are not yet in a position to make recommendations on the revision of the Dacimine formulations, significant gains were made in our knowledge of the Dacimine system. Storage tests of product prepared from Formnyte 808 and Duocman-O Special indicate that although both are better than material made from Duocman-O, they don't approach the stability of product made with Kammine D-999. Since all the amines were found to have comparable amine values, but widely differing iodine values, the importance of the IV, which measures the degree of unsaturation is quite evident. D-999 with an IV of 97 produces a very stable product, while Dacimine produced from the diamines with lower IV's (Formnyte 808, 85; Duocman-O Special, 81; Duocman-O, 71) tends to fall out in the freezer.

Both Forestort and Anzur have been contacted and they both say they can produce a material with an IV comparable to Kammine D-999, and will send samples to us. It is hoped that their material will be cheaper than the relatively expensive D-999.

Two possible formulations to make a solid Dacimine for surface treatment were prepared, and they outwardly looked OK.

PROPOSED EXPANSION

With the approval of the appropriation, design work is getting started. Since priority has been given to the tank farm and warehouse, initial work has been done in these areas. Two additional warehouse quotations were received and vendor contact made to discuss our piling requirements.

MISCELLANEOUS

Four special 55-gallon samples of mixed D and T technical butyl esters and one 55-gallon sample of a 5% Dacimine-D/T were prepared.

Two Engineers were temporarily assigned to the Production and Maintenance Departments to give vacation coverage.

The following appropriation was closed during the month:

No. 6755-92 - Rotameters - \$686 expended

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FGS/nc

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