



Interoffice Communication

To P. L. Fetzer
From H. L. Hackett
Date July 10, 1973
Subject Progress Report, June 1973

Summary

Plant Tars Drying--Flake calcium chloride was successful in drying tars in a recirculating system. The water level of the tars was reduced from 320 parts per million to 11 parts. A second series with the same bed reduced the water from 276 down to 148 parts per million before the bed became plugged. The bed was calculated to have absorbed 20.75 percent of its weight of water. Plugging problems and pump failures have so far prevented the attainment of higher loadings theoretically possible.

Analysis of Intermediate Streams--Hydrogen chloride from three sources was studied in an effort to establish whether or not several small peaks from an alumina column with sodium carbonate prescrubber were artifacts. The results were inconclusive.

Unknown Component in Heavy Ends Reflux--Recently an unknown component has been observed in heavy ends feed and in trace amounts in heavy ends reflux. Laboratory work has shown that the component is not chloroprene, trans-1,2 dichloroethylene, or chloral. EDC samples from the purification train were sent to R & D for help in identifying this component.

Miscellaneous--Several spot checks of the acid concentration in the aqueous phase of S-307 were made for Operations. Hydrogen chloride concentrations ranged from 26.1 to 26.3 weight percent.

The undersigned was Acting Chief Chemist the week of June 25th and will continue through July 6th.

Homer L. Hackett

Homer L. Hackett
Manufacturing Chemist

bs

Distribution
LNV-RDG-RHG-GGD-JDM-DCS-JCL

CCR 000034051

Plant Tars Drying

Two tank cars with stored tars were found to be wet. Since water contamination during storage is always a possibility and can lead to corrosion or interfere in several disposal schemes, it is important to find an economical way to dry tars. Some previous work involving single pass dessicant beds gave very poor results. Current work with calcium chloride in a recirculating system was more successful. Flake calcium chloride, 33.2 grams, dried 2.7 gallons of tars from 320 parts per million down to 11 parts in 7 3/4 hours circulating at a rate of 1667 milliliters per minute.

The same calcium chloride bed lowered the water from 276 ppm to 148 ppm on another 4.7 gallon batch of tars in 2.5 hours. The experiment was terminated at this point because of plugging by components in the tars.

Water uptake was calculated to be 20.75 percent of the initial calcium chloride weight. Several attempts to get higher loadings were plagued by plugging problems and pump failures.

Analysis of Intermediate Streams

Current work on this project was directed toward improving the analysis of components in hydrogen chloride. Hydrogen chloride from three sources was analyzed on an alumina column with and without a sodium carbonate precolumn. The three sources were hydrogen chloride generated from sodium chloride and sulfuric acid, 99.99 percent hydrogen chloride from the Chemical Plant, and samples of C-202 overhead. The results were inconclusive in that the several small peaks observed may or may not be artifacts. It is planned to investigate a new SF-96 Fluorolube on Teflon column and also a new amine type stationary phase, CRS-101 in the analysis of hydrogen chloride.

Unknown Component in Heavy Ends Reflux

Recently an unknown component with the same elution time as chloroprene (FFAP column) is sometimes observed in H.E. feed and in trace amounts in H.E. reflux. Careful checks on an SE-30 column show that the component is not chloroprene, trans-1,2 dichloroethylene, or chloral but no new peak was observed. The trans-1,2 dichloroethylene is poorly resolved from chloroprene on FFAP columns. It is found in only trace amounts in direct and recycle EDC. Oxychlorination is the principal source of trans-1,2 dichloroethylene. Chloral and aldehydes in general are often irreversibly absorbed on FFAP and only partially eluded. Ethylene dichloride (EDC) samples from the purification train from vinyl column bottoms to heavy ends reflux have been sent to Ponca City R & D for help in identifying the unknown component.

P. L. Fetzer
Progress Report, June 1973
Page 3

Spot Checks of Hydrogen Chloride Concentration in S-307 Aqueous Phase

As a precaution against freezing, spot checks of the acid concentration in the water phase from S-307 were made for Operations during some minor plant adjustments, June 19 and 20. The hydrogen chloride concentrations found ranged from 26.1 to 26.3 weight percent.

CCR 000034053