



Interoffice Communication

To P.L. Fetzner
From H.L. Hackett
Date November 16, 1971
Subject Progress Report, October 1971

Methyl Chloride Problem

Some additional caustic recycle studies were made on removal of 2-chloroethanol. T-102 caustic out (1.36% NaOH, adjusted to 1.95% with solid sodium hydroxide) was used to treat twice the volume of T-102 EDC out in each of four successive treatments. Each treatment consisted of 15 minutes agitation at 40°C. The following 2-chloroethanol levels were found.

	<u>2-Chloroethanol</u>	<u>Percent Removed</u>
T-102 EDC Out (9/30/71 - 1200 hrs)	1822 ppm	-
EDC layer after first treatment	342 ppm	81
EDC layer after second treatment	979 ppm	46
EDC layer after third treatment	1439 ppm	21
EDC layer after fourth treatment	1600 ppm	12

These results are much different from previous data for recycle of an artificial 3.5 percent caustic which gave better than 90 percent removal after the fourth recycle. (HLH Progress Report, September 1971)

A three-step experiment (done twice) was made to roughly simulate the initial behavior of one of the proposed caustic treatment schemes. In this proposal fresh make-up caustic would be added to a second caustic wash vessel. The spent caustic (except for an amount equal to the make-up) would be recycled back to T-102. The experimental steps are outlined below along with the results. All ppm values are for 2-chloroethanol unless labeled otherwise. Upper values are from the first run and lower values from the second. Each step was made at a treatment ratio of 2/1 EDC to caustic, 40°C for 15 minutes. In Step 2 an artificial feed to the caustic wash was prepared by mixing the three EDC streams in the ratio corresponding to the relative flows as measured on 10/7/71.

Step (1) corresponds to initial results expected in the proposed second reactor; Step (2) to those in the present caustic wash (T-102), with caustic recycled from the second reactor; and Step (3) to the EDC that would be produced by the combined treatment.

- 1) T-102 EDC Out (10/7/71 1400, 40 ml, 466 ppm) + 2.04% NaOH (20 ml)
 (10/12/71 0940, 40 ml, 665 ppm)

23 ppm	1.95% NaOH
43 ppm	1.83% NaOH
- 2) S-309, T-103, EDCTSOH Mix (40 ml, 1686 ppm, 400 ppm HCl) + NaOH
 from Step 1 (20 ml)
 (40 ml, 2153 ppm, 380 ppm HCl)

15 ppm	1.25% NaOH
42 ppm	1.21% NaOH
- 3) EDC from Step 2 (40 ml) + 2.04% NaOH (20 ml)

15 ppm	1.91% NaOH
20 ppm	1.91% NaOH

The results in Step (2) (97% removal of 2-chloroethanol) are much better than would be expected on comparison with the first experiment where only 81% removal was obtained on recycle of caustic from the caustic wash system.

Plant Vent Problems

An effort is underway to identify and determine the source of the components in the C-500 vent gas stack which on several occasions has lead to objectionable odors in the plant vicinity. Dr. C. Starks and Mr. E. Setzkorn, R&D, visited the plant to help in these efforts. They found that the strong oxidant was almost entirely chlorine, and that little or no chlorine oxides, HCl or HOCl were present. Their chlorine levels were almost an order of magnitude higher than those obtained by the Kitagawa Tube method. Detailed results were reported in a letter to D.O. Popovac dated 10/25/71.

During a period when the odor was occasionally present just north of the laboratory, air was pumped through KI solution (1272 liters in 23 hours) but no trace of oxidants were found.

Some preliminary checking was done on two different chlorine methods but neither appears to be any better than the KI method.

Cloudy Monomer Problem

Very cloudy monomer has occurred in several storage spheres during the month. Recently attention has been directed to ways in which oxygen could enter the process streams since it appears to be involved in cloud formation. The caustic going to the vinyl decanter was found to have 6.5 ppm dissolved oxygen. A measurement of 0.5 ppm in the caustic out may be incorrect because of the sparging effect of vaporizing vinyl from the sample.

An attempt was made at the same time to measure oxygen in the vinyl before and after the decanter by vaporization of liquid samples and use of the Teledyne Oxygen Analyzer. Both the vinyl in and the vinyl out showed 6 ppm, but these results could also be in error. Less than 1 ppm dissolved oxygen was found in the well water used to make up the caustic. The water became oxygen-saturated in the "Break" Tank, T-431. Blanketing the "Break" tank with nitrogen reduced the tank effluent to 2.5 ppm dissolved oxygen. However, Pump P-431B apparently brings in enough air through the packing to completely resaturate the water before it goes to the caustic blender.

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