

Interoffice Communication

To P. L. Fetzner
 From H. L. Hackett
 Date September 12, 1973
 Subject Progress Report, August 1973

Summary

Chloroprene Removal from Recycle EDC -- The chloroprene in recycle EDC was reduced from 2950 to 30 parts per million by addition of anhydrous hydrogen chloride on passage through an alumina bed at 100 degrees Centigrade. Residence time was six minutes based on total bed volume. The mole ratio of hydrogen chloride to charged chloroprene was eleven. Runs are planned at 165°C (bottoms temperature of vinyl column) to try to bring the excess hydrogen chloride required down to more practical levels.

HCl Column Test Run -- An additional set of data around the column was obtained when the plant was running at reduced rate (70 percent) just before turnaround. The data was forwarded to Process Engineering, Ponca City.

Turnaround Samples -- Samples taken from several units during the turnaround were sent to Research and Development for characterization. Included are deposits from the light ends, HCl, and quench columns.

Miscellaneous -- Some time was spent tabulating data from the light ends column fouling test run and assisting in the work here on the recent VCM quality (gel) problem.

Homer L. Hackett

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bs

CC: LNV-RDG-RHG-GGD-JEM-DCS-JCL

Chloroprene Removal from Recycle EDC

An unsuccessful (R-18) and a partially successful run (R-19) was made with an alumina bed at room temperature. The partially successful run gave 80.9 percent removal of the contained chloroprene. In this run the reaction train consisted of a one liter charge vessel filled with 500 ml of recycle EDC and pressured to 120 psig with nitrogen. The valved bottom outlet lead into the top of another 1 liter vessel pressured to 100 psig with plant HCl. The bed just below, consisted of a 75 cc sample bomb containing 37.62 g of partially crushed Houdry spheres. The valve on the bottom of the alumina bed leads into a receiver with provisions for nitrogen sweeping the vapor space through a Fisher-Mulligan gas washer. The bed was first presaturated with HCl by opening the valve from the HCl reservoir. The pressure equalized at 30 psig and the alumina bed became almost too hot to touch. The valve was closed. After the bed partially cooled, recycle EDC was pressured into the HCl vessel with nitrogen allowing the pressure to come to 120 psig. The valve into the bed was opened slowly, then EDC was slowly bled into the receiver at an average flow rate of 4.55 ml/min. The collected EDC was water washed and the water combined with that from the gas scrubber. Titration showed 4.26 g of recovered HCl. Chloroprene removal by gas chromatography was 80.9 percent.

The best alumina run to date (R-20) employed an atmospheric steam jacketed tubular reactor. The packed section of the bed was 1.05 cm in diameter by 70 cm high. The total volume of the bed was 60.6 cubic centimeters and contained 33.10 g of partially crushed Houdry spheres with the fines screened out. VC Bottoms (500 ml, 8/27/73, 1350 hrs., 2950 ppm chloroprene) was charged to a nitrogen flushed 1 liter sample cylinder. The sample cylinder was pressured to 30 psig with HCl from the C-202 O.H. sample point. The HCl was introduced through the bottom valve. The sample cylinder was then connected to the top of the alumina bed and the valve between was opened. Steam was passed through the jacket. Nitrogen was introduced into the top of the sample cylinder to bring the pressure to 120 psig. Additional nitrogen was charged periodically to maintain at least 100 psig during the run. Ten minutes were allowed for heating up the bed before opening the exit valve to start the run. After the first 100 ml, the collected EDC was transferred by gravity into the second receiver. The remainder was collected in the first receiver. The entire 500 ml charge (except for ca. 5 ml hold-up) was passed through the bed in 50 minutes time for an average flow rate of 10 ml per minute. This would represent a residence time of 6 minutes based on total bed volume. At the end of the run the charge vessel and the bed were pressured back up to 60 psig with nitrogen, then bled down through the system to flush out most of the remaining HCl. The EDC in the second receiver was washed with an equal volume of water and the EDC layer discarded. Water from the gas washer was added to the water from above and used to wash the product EDC in the first receiver. The EDC layer was again washed with additional water which was then combined with the other water. By titration 8.23 g (.226 moles) of HCl was recovered from the run. The 395 ml of EDC product in the first receiver contained 30 ppm chloroprene. This represents 98.98% chloroprene removal. The amount of chloroprene in the charge was 1.844 grams or 0.0208 moles. Assuming only one mole of hydrogen chloride reacted per mole of chloroprene charged (and disregarding reaction with any other unsaturated compounds), the amount of HCl charged is calculated to have been approximately $0.226 + 0.0208 = 0.2468$ moles. This is roughly an 11 fold excess of HCl.

HCl Column Test Run Data

Selected streams were resampled just before turnaround when the plant was operating at about seventy percent of capacity. The analytical data is shown in Table I.

TABLE I

HCl COLUMN TEST RUN DATA

Reduced Rate (70%)

Component (Wt. %)	Quench Reflux 8/3/73 1215 Hrs.	Gas Feed to C-202 from H-211 8/3/73, 1500 Hrs.	C-202 Column Bottoms 8/3/73 1400 Hrs.
Acetylene	-	.0618	-
VCM	31.536	33.1028	43.1333
EDC	61.477	2.3458	56.8605
HCl	6.987	64.49	.0062

PPM (Mole)	C-202 O.H. Gas 8/3/73 0900 Hrs.	C-202 O.H. Liquid 8/3/73 1115 Hrs.
Methane	40	-
Ethane	9	-
Ethylene	92	40
Acetylene	2087	4120
VCM	6	6
Ethyl Chloride	Tr	Tr
EDC	11	Tr
Benzene	29	53
Unknown #9 (1)	-	Tr
HCl	Remainder	Remainder

(1)

This peak corresponds to Unknown #9 as designated in previous data.

NAME

John D. Minott

MONTH

August 1973

FROM

TO 23x8 = 184

CCR 000034058

PROJECT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
Staff Meetings	2			X	X						X	X						X	X	1				X	X	1				
Mail Reading	5	1	1																				1			1		1		
Stack Air Plan Filing	10	4	5			2	1	3	4	4			7	6	6	2	6			4	7	5	4	4			5	6	6	6
Stack Air Sampling	5	3	1					1																						
Cooling tower Studies	1	1																												
Secondary system	10	1						2	2	1			1	1	1	1					1	1					1	1	1	1
Carbon absorption testing	9		2			2	1	2	1						1															
Monthly Polln. Report	X																													
Plant outage work	9					3	5		1																					
Roy Weston Visit	8					1	1	1					1		1	1				1							1			
Weather	10																										8	8	8	8
Weekend Coverage	1																1													
WRC Cap Study													8																	
Budget Meetings	14															4				2		2	4	2						
Safety Director Assistance	3									2														1						
Polln = 181																														
Safety - 3																														
	8	8	8	X	X	8	8	8	8	8	X	X	8	8	8	8	8	X	X	8	8	8	8	8	X	X	8	8	8	8