INTER-OFFICE MEMO **TENNECO CHEMICALS, INC.**

TO J. W. Poarch AT Burlington DATE January 12, 1978

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SUBJECT DEI-508, COPOLYMER REACTOR RECOVERY (R-4 TESTS)

RECOMMENDATIONS

1. It is recommended that Engineering and Production proceed with the installation of a low pressure steam header (25 psig, 150-300 lbs/hr) to all Copolymer reactors for use in reactor EPA recoveries.

2. It is recommended that a high pressure water cleaning system (4000-6000 psig, 40 gpm) be installed in copolymer for reactor cleaning to minimize reactor entry and lost time due to increased reactor fouling that will be associated with the EPA recovery procedure.

3. It is also recommended that Engineering and Production proceed rapidly with the design and utilization of closed reactor operation in Copolymer.

CONCLUSIONS

1. It is necessary to use high jacket temperature (180°F) during recovery with all of the techniques studied to minimize the reactor recovery time for EPA compliance.

2. In all reactor recovery procedures evaluated, the residual VCM in the reactor vapor space is dependent on jacket temperature during recovery and the degree of wall fouling. The higher jacket temperatures during recovery accelerate the wall scale buildup.

3. There is no economic justification for installation of Fetterolf reactor flush valves in Copolymer Reactors unless a clean reactor technology is used. The total recovery time to meet EPA requirements with either the Flush Procedure or Steam Purge Procedure is approximately equivalent. This amounts to 65-70 minutes dump to vacuum break. The installation of low pressure steam for an EPA recovery procedure is the most economical.

CONCLUSIONS (continued)

4. The use of the Fetterolf flush valve between charges will not remove wall scale buildup and did little to prevent it. It was only effective in rinsing the loose resin out of the reactor. It would have excellent application in conjunction with a clean reactor technology.

5. Removal of wall buildup with the high pressure water system (4000-6000 psig) was evaluated. This method was effective provided it was done at the proper frequency. This frequency has to be before wall buildup gets too heavy, gives kinetics problems and peels off in large sheets during cleaning. These large sheets will plug the dump hole and require entry of the reactor (involves two men) for removal. The jet cleaning frequency for the steam purge recovery procedure recommended is estimated to be in the area of 3-5 batches.

6. A 12.4% productivity loss can be expected with the EPA steam purge recovery procedure recommended. This is due to extended recovery and extra cleaning time. Utilizing a high pressure cleaning system with this steam purge procedure will reduce the loss to near 6.5%. If closed reactor operation in copolymer is developed and utilized, this loss can be eliminated and a 4.5% gain in productivity realized.

DISCUSSION

Copolymer reactor recovery evaluations under DEI-508 were complete in July 1976 and a summary report was issued August 23, 1976 (P. Scheiner/L. Medeiros). This report summarizes tests run with a steam purge reactor recovery procedure (test procedure #3, attached) and a nitrogen purge reactor recovery procedure. Concluded in this report was that the steam purge recovery procedure would result in approximately a 15% reduction in productivity due to excessive wall fouling and extended recovery times. A portion of this would be regained if a high pressure water cleaning system were incorporated. The nitrogen purge recovery procedure has since been eliminated from consideration but was estimated to encompass near a 7% loss in productivity; this loss would have been due only to extended recovery time. The cost of nitrogen and the problems of dealing with large volumes of VCM contaminated nitrogen were the main reasons for rejection of this process.

DISCUSSION (continued)

Further copolymer reactor recovery work was initiated in an effort to minimize the accelerated wall fouling effect noted with a hot jacket and steam purge recovery procedure and also to evaluate an alternate copolymer recovery procedure involving a reactor flush. Table I summarizes this current work along with previous evaluations of hot jacket and steam recoveries which was summarized in the report noted above.

To accomplish the flush test runs, reactor 4 was set up with a Fetterolf Ram Seal Flush Valve (1 inch size). The design of the valve was rated to deliver near 80 gpm of water at 200 psig. The initial setup incorporated a Sellers water-steam mixing nozzle to develop the 200-250 psig required for the flush valve. This high pressure hot water delivery system was abandoned due to water-steam balance problems inherent in the mixer when used in conjunction with the Fetterolf rinse valve. An inbalance in the system would cause loss of pressure, poor water flow and subjected the recovery system to possible injection of ambient air.

This equipment setup was revised to incorporate the copolymer water charge pump as the source of flush water. For the recovery trials run in R-4, this pump delivered 180-200 psig of cold or hot water to the Fetterolf Flush Valve. Test runs with the reactor flush recovery procedure evaluated the effect of reactor flush on recovery efficiency and minimizing wall fouling. Variables looked at were hot versus cold flush, a water add to the reactor for vapor generation, and reactor jacket tmeperature.

Observation of R-4 after flushing and recovery showed the valve to be ineffective against preventing wall buildup. It was adequate only in washing the loose resin off of the interior walls of the reactor and would not cut accumulating skin buildup. This rate of buildup remained dependent on the reactor jacket temperature, with the higher jacket temperatures promoting an increased rate of fouling. This wall fouling effect was noted through visual observations, control in kinetics, and residual VCM results after recovery. Unfortunately, the residual VCM vapors in the reactor after recovery was very dependent on jacket temperature. This was true of all the recovery procedures evaluated and summarized in Table I. The higher jacket temperatures were needed to produce acceptable residual VCM levels of less than the permitted 2000 ppm. Hot versus cold water usage (60 versus 135°F) in the reactor flush was critical in obtaining complete transfer of the slurry from the reactor to the stripper. Cold water flushing promoted reverse flow of slurry due to the cooling of the reactor vapors. This resulted in large slurry heel remaining in the reactor. With this unstripped slurry heel, residual VCM levels of below 2000 ppm were impossible to obtain in the 20-30 minute recovery time after 22 inches vacuum.

DISCUSSION (continued)

The hot flush prevented this backflow of slurry by promoting the vapor generation needed to maintain the forward flow. It was also critical to maintain a pool of hot water in the reactor during recovery. This pool served as a means to generate carrier vapor for removal of the VCM. Several attempts to eliminate the water add step resulted in unacceptable residual VCM.

Reviewing results summarized in the comparisons presented in Table I, there appears to be little advantage to incorporating the Fetterolf Flush Valve in copolymer EPA recovery process. In all recovery procedures the hot reactor jacket is needed. The total reactor recovery time needed to meet EPA limits is nearly identical for all procedures evaluated. The use of steam is a more positive source of carrier vapor than the dependence on a pool of hot water in the bottom of the reactor. Current copolymer processing does not require a flushout of the reactor between all batches or all product changeovers so that there is minimal savings associated with lost product flushed to the ground or lost airout time. From all observations the capital expenditure required to install the flush valves is not warranted.

Table II summarizes the economics of the proposed EPA recovery procedures. It is recommended that Production go forward with the utilization and installation of steam in EPA reactor recovery. Preferably, this steam should be low pressure (25 psig) with a steam flow rate in the area of 150-300 lbs/hr. This process will account for near a 12.4% loss in productivity. It is also recommended that a high pressure water cleaning system be installed (4,000 to 6,000 psig, 40 gpm) to help regain a portion of the lost time due to the increased cleaning frequency that will be associated with EPA recovery. Productivity loss with this cleaning system will be reduced to near 6.5%. The high pressure water cleaning will have to be on a frequency of 3-5 batches. With cleaning frequencies much beyond this, wall buildup will be too heavy, kinetics problems will be encountered and buildup will peel off in large sheets requiring entry to remove it (entry will involve two men).

In addition to the above it is recommended that Engineering and Production proceed with the formulation for design of closed reactor operation in copolymer production. Not only would this eliminate completely the lost production due to the EPA recovery but it would also result in an estimated gain in production of near 4.5% (see memo, J. W. Poarch to R. E. Harmond, 11/26/77). With closed reactor technology, fouling of the reactor walls due to hot reactor jacket would be minimized and reactor opening for high pressure cleaning would be at an estimated 5 batch frequency. With closed reactor operation the EPA limits would be a minimal problem (approx. 10,000 ppm would be allowable), resulting in less emissions and operator exposure.

L. Medeiros

LM/ib

TABLE I

SUMMARY OF RESULTS

TEST PROCEDURE	JACKET TEMPERATURE, °F		MINUTES: DUMP, RECOVERY, + VAC BREAK							BATCH CLEAN FREQ.	FINAL VCM IN VAPOR SPACE, PPM
	DUMP	RECOVERY	DUMP	FLUSH	to 22" HgVAC	WATER ADD	AFTER 22" VAC	VAC BREAK	TOTAL		
1- CONTROL	–	NO HEATUP	15	0	15	0	0	10	35	15-20	100M - 200M
2- HOT JACKET	180	180	15	0	15	0	15	10	55	5-10	2M - 6M
3- STEAM + HOT JACKET	180	180	15	0	15	0	10	10	50	3-10	2M - 4M
	180	180	15	0	15	0	30	10	70	3-10	800 - 1600
	180	180	15	0	15	0	50	10	90	3-10	400 - 1100
4- FLUSH	NO HEATUP	180	15	3	15	2	20	10	65	*	Greater than
5- FLUSH	180	180	15	3	15	2	20	10	65	5-10	500 - 2000
FLUSH	150	150	15	3	15	2	20	10	65	*	Greater than
FLUSH	160	160	15	3	15	2	20	10	65	*	1300 - 5000

* CANNOT MEET EPA, INSUFFICIENT BATCHES RUN TO ESTIMATE A CLEANING FREQUENCY

TABLE II

PROJECTED IMPACT ON PRODUCTIVITY

FOR EPA COPOLYMER RECOVERY

TYPE 315 RESIN

	(5/28/76) 1977 BUDGET	(7/15/77) 1978 BUDGET PROJECTION	REVISED PROJECTION FOR 1978 AFTER 6/1/78 STEAM WITH ALL MANUAL CLEANING	PROPOSED STEAM WITH H.P. CLEAN	FUTURE PROPOSED STEAM WITH H.P CLEANING AND CLOSED REACTOR OP
YIELD, LBS/BA	10,000	9,950	9,950	9,950	9,950
CHARGE TIME, HRS	1.0	1.0	1.0	1.0	0.7[9]
HEATUP TIME	1.0	1.1	1.1	1.1	1.1
REACTION TIME	7.7	7.3	7.7[3]	6.9[4]	6.9[4]
DUMP TIME	0.3	0.3	0.3	0.3	0.4
SUB TOTAL:RX CYCLE	10.0	9.7	10.1	9.3	9.1
[illegible]ITIONAL OSHA & EPA[10] ([illegible]NCLUDING REACTOR RECOVERY)	0	1.4[2]	0.6[7]	0.6[6]	0.2[5]
WAITING TO DUMP	0.4	0.6	0.8	0.8	0.5
WAITING TO CHARGE	0.8	0.8	0.8	0.8	0.8
WAITING TO CLEAN	0.2	0.1[8]	0.3[7]	0.7[6]	0.4[5]
CLEANING	0.7	0.4[8]	1.2[7]	0.7[6]	0.5[5]
MAINTENANCE	0.4	0.4	0.4	0.4	0.4
SUB TOTAL: DOWN TIME	2.5	3.7	4.1	4.0	2.8
TOTAL CYCLE	12.5	13.4	14.2	13.3	11.9
lbs/REACTOR HR	800	743[1]	701[1]	748[1]	836[1]
% CHANGE	CONTROL	(-7.1)	(-12.4)	(-6.5)	+4.5

NOTES: see Table II (continued)

TABLE II
(continued)

NOTES: (1) Budget projections show a full twelve months operation and were not factored for a split year operation.

(2) Original budget projection with OSHA and EPA effect lumped into one category.

(3) L. P. reduction for kinetics control due to increased wall fouling.

(4) Increased L. P. due to more frequent cleaning with H. P. water system.

(5) Closed reactor operation with opening after every 5th charge, H. P. clean after every 5th charge with possible entry after 20 charges.

(6) H. P. clean after every 3rd charge with possible entry after 21 charges.

(7) Manual clean on an average after every seven charges.

(8) Current cleaning frequency (15-20 charges).

(9) Closed reactor operation with opening and evacuation every 5th charge. (No reactor recovery or vacuum check for four of the five charges.)

(10) No time allotted above for EPA sampling.

(11) The following times were allotted for each H. P. cleaning:

0.25 hour's for opening manway nozzle
0.50 hour's for airout
0.50 hours for H. P. cleaning of walls
0.25 hours for closing manway nozzle.

TEST PROCEDURE # 1 RECOVERY CONTROL

Turn steam switch off, set batch temperature setpoint to 135°F, put reactor on tower water only and transfer the batch to the stripper. After completion of transfer, isolate the reactor and recover down to 22 inches mercury vacuum. Close off recovery and break vacuum with nitrogen.

TEST PROCEDURE # 2 HOT JACKET RECOVERY - NO FLUSH

Turn steam switch on, set batch temperature setpoint to max to heat and maintain the jacket temperature at 180°F minimum while transferring to the stripper. After completion of transfer to the stripper, recover reactor down to 22 inches minimum, then continue to recover for the additional time specified in the test. Close off recovery and break vacuum with nitrogen while cooling jacket.

TEST PROCEDURE # 3 STEAM PURGE - NO FLUSH

Turn steam switch on, set batch temperature setpoint to max to heat and maintain jacket temperature at 180°F minimum while transferring to the stripper. After transfer is complete, open steam purge to reactor. Allow steam to purge out through bottom of reactor for approximately 2-5 minutes. Then close off stripper transfer valve, isolate reactor from dump line and continue to recover reactor with steam purge for specified time period after 20-22"vacuum is attained. Close off recovery, close steam, cool and break vacuum with nitrogen.

TEST PROCEDURE # 4 FETTEROLF FLUSH VALVE

When ready to dump turn steam switch off and leave temperature controller set at reaction temperature. At completion of transfer of the batch to the stripper, flush reactor into stripper with hot water (3 minutes, 135°F, 200-300 gal, 150-200 psig). Isolate reactor and heat the reactor jacket to 180-190°F while recovering down to 22" mercury vacuum minimum. After 22" and while continuing recovery, add 25-50 gallons of hot water to the reactor through the flush valve. Recover reactor for an additional 20 minutes, close off recovery then cool and break vacuum with nitrogen.

TEST PROCEDURE # 5 UTILIZING FETTEROLF FLUSH VALVES

Heat the reactor jacket to specified temperature (150°, 160° or 180°F tests) at the start of the dump and recovery procedures. At completion of the transfer, hot water flush the reactor into the stripper (3 minutes, 135°F, 200-300 gal, 150-200 psig). Isolate the reactor and recover down to 22" mercury vacuum. While continuing to recover, add 25-50 gallons of hot water to the reactor through the flush valve. Recover the reactor for an additional 20 minutes, close off recovery then cool and break with nitrogen.

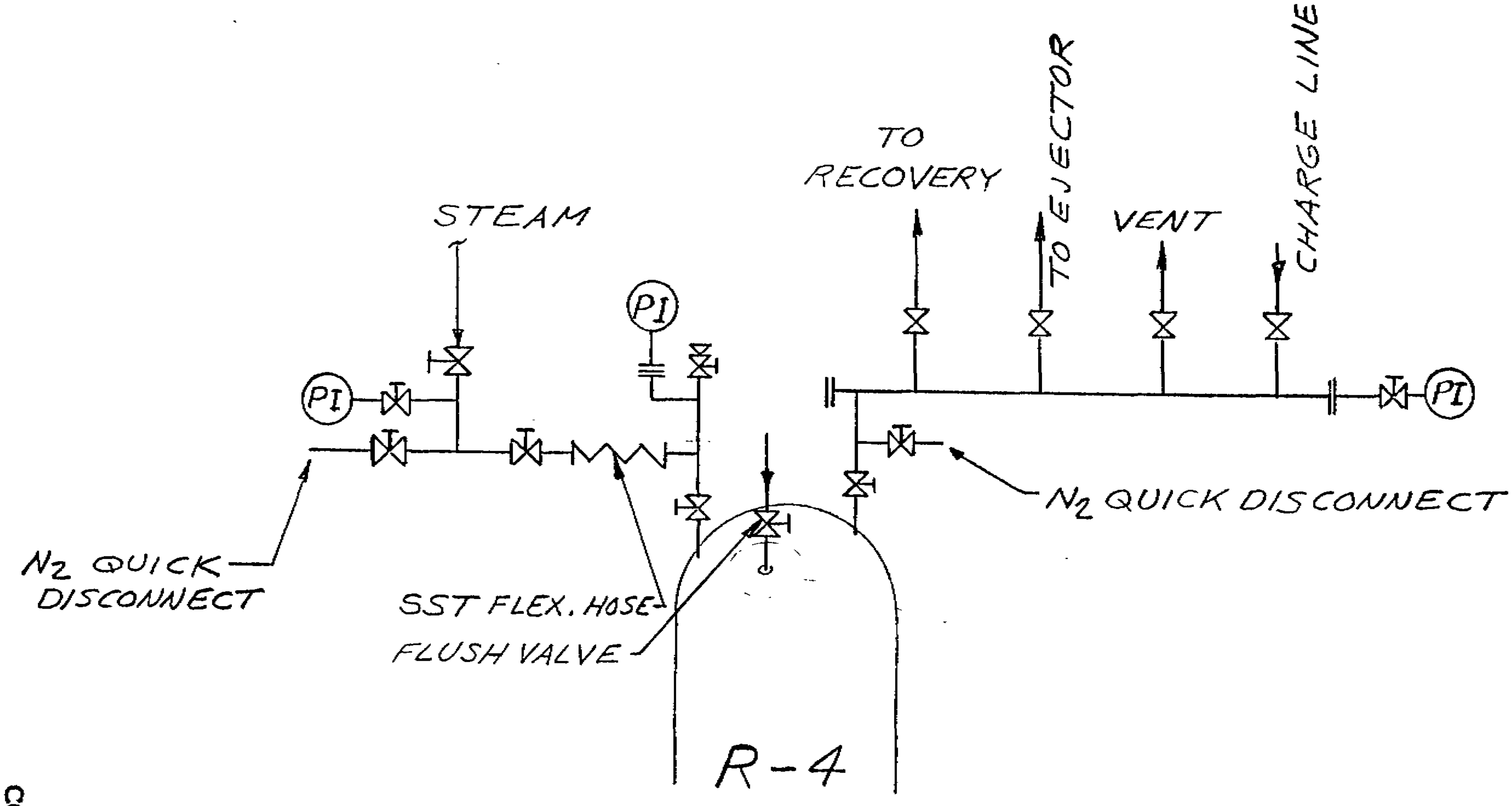

FIG. 1

COPOLYMER REACTOR RECOVERY TESTS