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Quarterly Summary Report October - December 1977

Process for Cleaning and Removal of Sulfur Compounds from Low Btu Gases

January 1978

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QUARTERLY SUMMARY REPORT
OCTOBER - DECEMBER 1977

PROCESS FOR CLEANING AND REMOVAL
OF SULFUR COMPOUNDS FROM LOW BTU GASES

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January 1978

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PROCESS FOR CLEANING AND REMOVAL OF SULFUR
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I. OBJECTIVE AND SCOPE OF WORK PHASE IV

In this phase of work the Process Development Unit (PDU) is to be remodeled by incorporation of appropriate subsystems to permit operation in continuous process mode. The PDU will be operated for a period of time sufficient to demonstrate process viability.

II. SUMMARY

- 1) Construction of the PDU was completed during the final week of October 1977.
- 2) A detailed hazards review and safety analysis was prepared and presented to the Battelle Safety Review Committee.
- 3) Initial startup of the PDU occurred on November 14, 1977. A power failure caused by a blown fuse on the Richland PUD high voltage line resulted in emergency shutdown after about 24 hours' operation.
- 4) Abnormal chatter in magnetic reactors feeding power to trace heating circuits was corrected and miscellaneous repairs were made and the PDU was restarted on November 17, 1977. Minor difficulties with the coal conveyor delayed full operation for a few hours, but this run had to be aborted due to failure to achieve salt flow to the venturi.
- 5) To conserve funds, test operation of the PDU using air to circulate the salt was attempted to see if proper flow sequence could be established. In two such runs severe difficulties were experienced in achieving or in maintaining

flow to the venturi scrubber and in both cases efforts had to be terminated with the venturi feed line plugged at some point.

6) These preliminary runs disclosed the presence of a sludge composed of miscellaneous tramp materials together with corrosion-erosion products which had combined with added sulfide. This material, of generally higher density than the bulk salt tends to accumulate and settle out in the lowest spot, in this case, the venturi feed line. Small openings in, e.g., the venturi orifice or the 1/4-in. diameter, Z-shaped flow passage through the bellows sealed valves became plugged. The valve was usually the offender.

7) To ameliorate this problem, standpipes were installed in the venturi feed tank and salt makeup tank to provide a reservoir into which heavy solids could settle. A fresh salt inventory was weighed up and charged to the salt makeup tank to replace salt jettisoned to permit this modification. Maintenance on flow meters, pressure sensors and sampling lines was performed.

8) On December 16, 1977 the PDU was again started. Salt circulation, initially with air only, was successful, so the secondary burner and gasifier were started. This run was continued until midnight of December 19, 1977. Over two tons of coke and sub-bituminous coal were gasified to supply low Btu gas at ~75 SCFM to the gas cleaning facilities. The extraction section of the plant performed well with sulfur recoveries exceeding design (>95%). Simultaneous operation of the regeneration column was not achieved, although the column was operated for a brief period without simultaneous operation of the venturi scrubber. The PDU was shut down for the holiday season and to permit time for data analysis and planning for improvements and for scheduling repairs.

III. TECHNICAL PROGRESS

During the month of October 1977 a maximum effort was put forth to complete the construction phase of the project. Major tasks coming to completion included installation of insulation by E. J. Bartell Company and wiring of trace heaters. Numerous smaller tasks included:

- Wiring of multiposition motor driven valves which direct sample flows to the gas chromatographs.
- Wiring of gamma ray densitometers used for flow sensing.
- Wiring and installation of H_2S sensors and alarm bells.
- Wiring and installation of floodlighting.
- Installation of a small "hot shot" steam generator and steam tracing on all sample lines.
- Installation of purge gas systems to keep all AP sensor and analytical sampling lines salt free.
- Leak testing and repair of all leaks found.
- Installation of a tar collection system.
- Installation and calibration of the gas chromatographs, infrared CO and CO_2 analyzers, and the digital data logger for monitoring 60 thermocouple sensors.

On about November 1, 1977 400 lb of a salt mixture comprised of 33.3 wt% each of Li_2CO_3 , Na_2CO_3 , and K_2CO_3 was weighed, blended in a small cement mixer, and charged to the salt makeup pot. Eight to 10 hours was required to melt this salt which was then sampled in a test of sampling procedures.

One hundred pounds of this salt mixture was also placed in the lower pot furnace and melted to provide a liquid seal at the salt return flow line. Without this seal gas can bypass the venturi scrubber and escape through the extraction column.

Following approval of a safety and hazards review, the PDU was started up for the first time on November 14, 1977 at 8:00 a.m. When the trace heaters were brought up to temperature, a process requiring several hours, a problem which would potentially be serious was noted. There was excessive chatter in the large magnetic reactors (relays) which in conjunction with solid state proportioning controllers feed power to the trace heaters. Analysis with the aid of an oscilloscope showed that a large voltage spike which appeared when one of

the reactors opened a circuit would act through the controllers to actuate a dozen or more reactors at a time.

This has two adverse effects: 1) It places an excessive load on the primary transformer, and 2) it causes excessive wear on the silver-plated contact points. These can weld shut when wear becomes excessive to keep a heater powered and out of control. Nothing could be done about this problem except to keep the operation under close observation. Following shutdown the problem was solved by installation of an electrical filter across the reactor terminals to reduce feedback of voltage spikes through the controllers.

During the early morning hours of November 15, 1977 the gasifier was started on a charge of 1000 lb of coke. It was not up to temperature by 6:00 a.m. when the day shift arrived, so an additional 800 lb of coke was charged to ensure a long run. About 10:30 to 11:00 a.m., gas of good fuel value was being produced and salt flow to the extraction column was initiated.

In early tests with water and air, starting flow of water to the extraction column led to a problem. This, it may be recalled, is a 10-in. diameter column with two trays containing four bubble caps per tray. In starting water flow to the column, the first tray sealed off properly (as indicated by ΔP measurements) but because the airflow rate was too high the second tray did not seal off.

This time molten salt flow was initiated with only CO_2 purge gas flowing. Pressure drops indicated both trays sealed off properly and the gamma ray densitometer indicated the siphon reservoir had filled with salt. Gas flow was then valved to the venturi scrubber and extraction column and the valve which admits salt to the venturi scrubber was opened wide. No pressure drop response across the venturi was observed and it was concluded salt flow had not occurred. This was confirmed in 20 to 25 minutes when salt overflowed the wier in the venturi feed tank. This made it necessary to valve off salt flow to the extraction column from which salt flows to the venturi feed tank.

Efforts were still in progress to initiate salt flow when a fuse blew on the Richland PUD high voltage line causing loss of one phase and necessitating

an emergency shutdown. A motor on a high pressure air compressor within the supporting laboratory single phased and burned out and this was the only damage sustained.

The power surges caused by the action of the magnetic reactors was probably the cause of the outage. The decision was therefore made to shut down, correct this problem by installation of electrical filters, and have the Richland PUD fuse their line with larger fuses (which was done the next morning). The burned-out motor was replaced and beaded wire heaters were installed at points on the venturi salt feed line to eliminate possible cold spots thought to be responsible for difficulty in initiating salt flow. The gasifier was also discharged and readied for another run.

On November 17, 1977 a second startup was initiated and this time when the heaters came up to their set temperatures the filters effectively reduced the chatter in the magnetic reactors to reasonable levels. Contact life will be appreciably extended.

During efforts to charge the gasifier that night the chain in the drag chain conveyor parted after 800 lb of coke had been added. Repairs were made the following morning, but during the three hours required, it was necessary to operate the gasifier at minimum output and pressure with gas bypassing the PDU via a 4-in. line to the secondary burner. This prevented startup of the molten salt scrubber until late afternoon.

This time flow to the venturi scrubber started immediately but could not be maintained. About 10 % of salt was dispersed by the venturi action, and carried to the coarse de-entrainer, but after 10 to 15 minutes, flow was completely blocked and could not be restarted. It was necessary to terminate the run.

Examination of the line to the venturi following cooldown disclosed a plug of high-melting sludge at the bellows sealed valve. This is a needle valve with a 1/4-in. diameter Z-shaped flow path which is fairly easily plugged. The sludge is believed to be tramp material and residues of the alonizing powder scoured off the pipe and vessel walls.

To reduce staff requirements and costs, it was decided to test operate the PDU using air to circulate the salt. The gas pre-heater is just barely able to heat air to 700°C at the required flow rate (75 SCFM). Operation was initiated to see if reliable valve function and salt flow control could be maintained.

In two attempts (November 29, 1977 and December 6, 1977) difficulties in maintaining reliable salt flow were experienced. As before this was found to be due to heavy solids separating at the lowest point in the system, the venturi feed line. Clearly, more drastic measures must be taken.

To solve the problem standpipes were fabricated for installation in the salt makeup tank and venturi feed tank to provide a dead space into which heavy solids could settle. This is an interim solution. Design of a filter trap for later installation in the venturi feed line is in progress.

In order to install the standpipes as much of the salt inventory as could be removed was drained from the system. When these were installed run C-5 was initiated at 8:00 a.m., December 16, 1977.

A. Operations on Shift (1) 8:00 a.m. - 6:00 p.m. December 16, 1977

Five hundred lb of salt was mixed, charged to the salt makeup pot, melted and brought to a temperature of 1320°F by 2:30 P.m. All trace heaters were brought up close to predetermined set points. The secondary burner was started and the gasifier slide valves and conveyor tested and found ready to operate. All sample lines and pressure sensor lines were leak tested and found okay (however, see below). A supply of coke was transferred from the stockpile to a point near the conveyor using a small front end loader.

B. Operation on Shift (2) 6:00 p.m. - 6:00 a.m. December 16-17, 1977

Operation was started using air to circulate the salt. Immediate salt flow to the extraction column at 0.89 ℓ/min was observed by stick readings on the salt makeup pot, but flow response to the venturi scrubber was slow and the upper standpipe in the venturi feed tank overflowed at 9:10 p.m. and flow to the extraction column had to be valved off. The ΔP at the venturi was 1.25 - 1.4 indicating some flow and the coarse

de-entrainer had been filling and showed 8 ℓ at 9:20 p.m. This would indicate an average flow through the venturi of about 160 ml/min. If this is subtracted from the 890 ml/min through the extraction column, the net inflow to the venturi feed tank was 730 ml/min which is sufficient to fill the venturi feed tank to the point of overflow in 38 min which is about the elapsed time at which overflow was noted.

In the interval from 9:20 to 9:30 the level in the coarse de-entrainer increased to 10.5 in. (13 ℓ) indicating flow to the venturi had increased to 500 ml/min and was increasing (as shown by ΔP readings). Flow was now initiated to the regeneration column and also to the absorption column. It was found possible to balance the flow into and out of the salt makeup pot, a very desirable situation.

Difficulty was experienced in getting reliable AP readings across the trays in the regeneration column. It was believed some of these small lines may have become plugged with salt from earlier testing. In any case, all systems were in simultaneous operation and it was decided to proceed with operation on fuel gas.

At 1:00 a.m. charging of 1000 lb of coke to the gasifier was completed and at 2:40 a.m. preparations to initiate salt flow were complete. At 3:15 a.m. salt flow to the venturi was valved on and AP readings indicated full salt flow was established almost immediately. At 3:20 flow to the extraction column was initiated and stick readings indicated immediate flow response at 0.64 ℓ /min.

At 3:50 a.m. attempts were made to start the regeneration column. Steam was coming off the superheater at only 100° to 105°C and when this entered the steam-CO₂ preheater and regeneration column, operating at 550°C, it flashed causing severe flooding in the regeneration column. Difficulties in adjusting steam flow to the system contributed to the problem. Flooding was so severe that salt was ejected from the column and found its way into the H₂S exhaust line which operates at only 150°C. Here it froze plugging this line completely. To the operators it appeared the regeneration column had plugged and steam-CO₂ flow to this column was

shut down when pressures reached 6 psig. Flow to the extraction column was valved off about 4:30 a.m. and with the venturi still operating all salt collected in the salt makeup tank by 5:30 a.m.

Analyses of product gases and gas samples taken throughout the extraction column showed COS and H₂S concentrations in the range of 9 to 12 ppm each. Inlet H₂S and COS concentrations were measured at 135 and 99 ppm respectively. This inlet H₂S concentration is abnormally low, but may be the result of the early stage in operation of the gasifier. Whatever the reason, the overall sulfur recovery during this period of operation averaged 91.3% which is a little below the design level of 95%. This is not serious in view of the low concentrations of H₂S and COS in the product gas.

C. Operation on Shift (3) 6:00 a.m. - 6:00 p.m. December 17, 1977

The gasifier was continued in operation via the bypass line while efforts were made to locate the obstruction in the regeneration column. By measuring pressure drops from point to point along the column it was soon discovered that the column itself was not plugged but that the exit line was plugged. Sometime later the actual plug (about 1 lb of frozen salt) was located by the laborious process of disassembling the line. This salt was chipped and washed out of the line which was then reassembled, a process which required about 8-10 hours' work.

It was also discovered that one of the heaters on the regenerator siphon had failed and this was spliced for temporary repair. Efforts were made to get all sample lines and ΔP sensor lines to the regenerator cleared but these efforts were only partly successful.

At 4:30 p.m. 800 lb of coke and 200 lb of sub-bituminous coal was charged to the gasifier and by 6:00 p.m. the PDU was again ready to operate.

D. Operation on Shift (4), 6:00 p.m. - 6:00 a.m. December 17-18, 1978

Efforts were continued to clear lines to the regenerator sample and pressure sensors and 30 lb of fresh salt was added to the salt makeup tank to make up inadvertent losses due to overflow at the venturi feed tank.

At 10:30 p.m. salt flow was initiated to the venturi and a few minutes later to the extraction column and with immediate flow response. During the next hour salt flow to the extraction column seemed somewhat variable and it was necessary to open and reset the valve to this column frequently. At 12:00 midnight the salt feed to the extraction column was turned off.

At 1:30 a.m. pure H_2S spike gas was added, bringing the inlet H_2S concentration to 2355 ppm (from 504). Salt continued to circulate in the venturi-coarse de-entrainer loop to allow buildup of sulfur compounds in the salt phase. At 4:00 a.m. salt flow to the regenerator was started using only CO_2 as a strip gas. The steam flow to the superheater was erratic and no steam was admitted to the column until the condition could be corrected. At 5:02 a.m. salt flows were shut down and gas flow bypassed to permit addition of coal to the gasifier. By 5:30 a.m. 400 lb of coke and 400 lb of coal had been added. As a result of frequent blowdowns, water in lines leading to the superheater was about cleared out and steam temperatures were climbing.

During the period from 10:30 to 5:00 a.m. a continuous effort to analyze as many gas samples as possible was in progress. The results are collected in Table 1. The inlet gas sample is taken just ahead of the venturi scrubber. The sample taken from the head space of the coarse de-entrainer, when compared with the inlet sample, measures the effectiveness of the co-current flow, single stage contact in the venturi scrubber and exhaust line. Samples taken at the top of the extraction column (or in the clean gas exit line) measure the effectiveness of the two additional stages of counter current flow contact in this column.

Throughout this period of time the venturi scrubber gave better overall sulfur recovery than would be predicted from the design basis. This was doubtless due to the fact that sulfur loading in the salt was below that used in design calculations. Salt samples were taken but analytical data is not yet available.

The additional extraction stages in the column raised the overall sulfur recovery to levels exceeding the design recovery. The last sample taken

TABLE 1. Gas Analyses and Performance of the Extraction Section,
Shift No. 4, December 17-18, 1977

Time 12/17/77	At Venturi		At Coarse De-Entrainer			At Top of Extraction Column			Remarks
	H ₂ S in ppm	COS in ppm	H ₂ S out ppm	COS out ppm	Overall S Extraction %	H ₂ S out ppm	COS out ppm	Overall S Extraction %	
22:35	504	98	--	--	--	--	--	--	Start salt flow
23:30	504	98	121	76	70.7	--	--	--	
24:00	504	98	85	10	83.8	--	--	--	
00:15	504	98	--	--	--	13	9	96.8	Turn off salt flow to extrac- tion column
00:30	504	98	--	--	--	11	9	97.1	
00:45	504	98	95	10	82.0	--	--	--	
01:30	2355	100 est	--	--	--	--	--	--	
02:00	2355	100	158	125	90.5	--	--	--	
02:30	2355	100	260	90	87.1	--	--	--	
02:45	2355	100	--	--	--	24	13	98.7	
02:55	2355	100	194	78	90.1	--	--	--	
03:40	3762	100 est	--	--	--	--	--	--	Start salt flow to regenerator column
04:22	3762	100	434	85	87.4	--	--	--	
05:02	3762	100	--	--	--	203	105	93.1	

at 5:02 a.m. is an exception, but it must be recalled that salt flow to the extractor had been off since midnight and the sulfur loading in the static salt phase held up on the trays (which won't drain so long as gas is flowing through the column) now is probably above the level employed in design calculations. On the whole, these data show the extraction section performed extremely well. Unfortunately, sampling difficulties prevented getting data on the effect of CO₂ strip gas on salt flowing through the regeneration column. The exact nature of the difficulty being encountered was not clear at the time, but proved to be due to frozen salt obstructing flow in sample lines.

E. Operation on Shift (5), 6:00 a.m. - 6:00 p.m. December 18, 1977

At the start of this shift, dry steam was available to the regeneration column which was started up with steam flow followed by salt flow and, finally, by CO₂ flow. Startup of the regeneration column was achieved at 7:42 a.m. after minor difficulties in starting salt flow were overcome.

After considerable difficulty in getting flow to the venturi started (it was necessary to apply a torch to the stem of the Nupro valve in the venturi feed line) flow was achieved about 8:20 a.m. This was just in time to prevent the feed supply to the regeneration column from becoming exhausted.

At 8:33 a.m. attempts were made to initiate salt flow to the extraction column (which in turn supplies the venturi scrubber) but no flow could be obtained. As efforts were continued, the reservoir of salt in the venturi feed tank emptied and flow to the venturi slowed and finally stopped. In turn, the reservoir of salt in the coarse de-entrainer, normally kept filled by the venturi scrubber, drained through the salt cooler and through the regeneration column to the salt makeup tank. Thus, all of the salt inventory collected in the salt makeup tank behind this recalcitrant valve.

Efforts to unplug this valve finally succeeded about 3:30 p.m. and, as if to add insult to injury, the valve froze in the open position as salt crept up into the bellows and froze around the stem. Immediate steps

were taken to open the valve to the venturi scrubber, but for unknown reasons, flow to the venturi scrubber could not be established immediately. Some salt was lost at the venturi feed tank overflow standpipe before the valve from the salt makeup tank to the extraction column could be reclosed.

To this point, 4:30 p.m., operating difficulties had accumulated to the point where run termination was being seriously contemplated. The venturi AP gauge was beginning to indicate a trickle of salt flow and as the next shift came on it was decided the run would be terminated unless the venturi would start within a few hours. Within a half hour it did start.

F. Operation on Shift (6), 6:00 p.m. - 6:00 a.m. December 18-19, 1977

At 6:30 p.m. flow to the venturi scrubber was achieved and was maintained until 7:15 p.m. when flow was valved off to permit charging coal to the gasifier. At 8:30 p.m. 1000 lb of sub-bituminous coal had been charged to the gasifier and flow of salt to the venturi scrubber re-established. The venturi was continued in operation while efforts were directed at starting salt flow to the regeneration column. Sporadic flow to this column was obtained starting at 11:25 p.m. and with moderate success was continued until 1:00 a.m. when gas flow was bypassed.

Meanwhile at 11:15 p.m. H_2S spike gas flow was started and allowed to flow into the venturi scrubber section until 11:53 p.m. Flow to the absorber was never initiated except for one brief pulse just after the H_2S spike gas was turned off.

The total pressure drop across the venturi scrubber-extraction column had exceeded 4 psig at this point and it appeared the static salt on the trays of the extractor and the salt in the coarse de-entrainer had become viscous. When this condition was observed the valve to the absorber was opened to add fresh salt to the trays of the extraction column and it appeared that the ΔP did drop, but gas flow was bypassed, shutting down the PDU.

In summary, during the period from 8:30 to midnight, a small fraction of the total salt inventory was circulated and recirculated in the venturi

scrubber loop. Only a small portion of this salt flow ever drained off to the regeneration column. About 20 ℓ of salt was circulating and about 6 to 7 ℓ of this flowed to the regeneration column and back to the salt makeup pot.

Toward the end of this shift, 300 lb of coke was charged to the gasifier to allow gasification to continue long enough to drive off tars from the coal-coke bed. Samples of all tanks were collected.

G. Operation on Shift (7), 6:00 a.m. - 6:00 p.m. December 19, 1977

Operation of the gasifier was continued until late in the shift. It was shut down about 4:30 and cooled under N_2 and steam, but it was necessary to call in a man from the other shift to keep it under observation until about midnight of the 19th. At that time the secondary burner and steam generator were valved off and secured. All trace heaters had been turned off and the salt was sitting in the molten state at 600° to 650°C in the salt makeup pot.

This run was not entirely successful in that simultaneous operation of both the extraction and regeneration sections was not achieved. Many operational problems were encountered. The unfortunate severe flooding of the regeneration column encountered at the onset of the run fouled the analytical lines and pressure sensor lines with frozen salt, a condition not corrected during the run. Little or no data was obtained on the behavior of this unit.

On the other hand, modest success was achieved in operation of the extraction section. Design recoveries were met or exceeded for H_2S and CO_2 and data has been obtained which will define the limits of sulfur loadings in terms of plant operability.

The Battelle gasifier operated very successfully on sub-bituminous coal and could have been continued in operation for a few more days. It is probable that five to six day runs will be attainable.

The experience gained in this run indicates some changes should be made. It is planned to modify the sample and ΔP sensing systems on the regenerator and extraction column to reduce the incidence of fouling by salt.

All of these lines are ~~much~~ too small and by enlarging ~~them~~ at points adjacent to the columns, salt plugging can be reduced.

In addition, a substitute for these bellows sealed Nupro valves is being sought. A valve that can be opened to the full-line diameter for an instant, to clear obstructions, would be feasible. At the moment it appears it ~~may~~ be possible to modify available gate valves to obtain what is needed.

The PDU was shut down at midnight December 19, 1977 and remained down over the holidays. ~~Due~~ to severe weather and the need for modifications as indicated above, restart is planned for sometime in February.

IV. PROPOSED WORK ~~DURING~~ THE NEXT QUARTER

It is planned to resume operation of the PDU. Test runs will demonstrate the effectiveness of various ~~demister~~ (chemisorber) additions to the packed bed de-entrainer. Tests of salt compositions containing up to 20 mole % CaCO_3 are planned. These ~~may~~ be less corrosive than compositions which are CaCO_3 free.

V. OVERALL ~~PROJECT~~ STATUS

Delays in completion of the PDU forced operations into the holiday and winter season with consequent further delay. Completion of the program is now anticipated late in ~~May~~ 1978.

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