**Quarterly Summary Report
July-September 1976
Process for Cleaning and Removal
of Sulfur Compounds from Low
BTU Fuel Gases**

by
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October 1976

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Pacific Northwest Laboratories

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CONTENTS

I.	OBJECTIVE AND SCOPE OF WORK PHASE I V	1
II.	SUMMARY.	1
III.	TECHNICAL PROGRESS	1
	A. Plant Design	1
	B. Stress Problems.	6
	C. Extraction and Regeneration Column Design Details	8
IV.	PROPOSED WORK DURING THE NEXT QUARTER	.12
V.	OVERALL PROJECT STATUS AND CONCLUSIONS	.12
VI.	REFERENCES	.12

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I. OBJECTIVE AND SCOPE OF WORK PHASE IV

In this phase of work it is proposed to remodel the PDU 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) Detailed design of the remodeled PDU is complete insofar as items which have effect on the schedule is concerned. These items include the piping support structure and piping and process vessels or equipment which must be "alvanized" for corrosion protection.

2) Major procurements have been initiated. Orders have been placed with low bidders for procurement of the extraction and regeneration columns and for the major portion of the trace heaters needed. The bid package for piping, coarse ammonia, salt cooler, venturi flash tank, and venturi (all of which will require "aluminizing") has been submitted to our Purchasing Department for distribution to selected vendors.

3) A comprehensive stress analysis on piping and support structures has been conducted with the aid of well known computer codes by personnel from our Engineering Department.

4) Portions of the PDU have been dismantled and some site preparation work has been completed to facilitate impending construction.

III. TECHNICAL PROGRESS

A. Plant Design

The reader can obtain a good impression of what the remodeled PDU will look like from the Piping Isometric (Figure 1) and from the Assembly Elevations

FIGURE 1. Piping Isometric

(Figure 2). Including ground, there are five accessible operating levels which contrasts with but two operating levels in the present PDU.

The large furnace, E-1, situated at ground level contains a 24 in. diam "aluminized" pot which will hold about 30 gal of molten salt. In the batch configuration of the PDU, the sulfur loaded salt was transferred by gravity induced flow to this pot where it was treated with steam + CO_2 , introduced via a sparge ring, to regenerate the salt. This pot will no longer be used for this purpose. In the remodeled PDU it will serve for emergency storage of the salt.

Elevation A-A (Figure 2) shows this furnace. Above the pot cover a tee is shown with an entering 4 in. line. This line carries the entering feed gas from the gasifier. Immediately above the tee is the venturi orifice. A 1 in. line containing a bellows sealed valve enters the venturi from the venturi feed tank (E-3, Figure 1). This line introduces the salt which is dispersed in the gas by the accelerating effect of the venturi orifice.

The dispersed salt is carried via a 2-1/2 in. line to the coarse de-entrainer (E-6, Figure 1). This line is sized to ensure a climbing film of molten salt along its inner surface and throughout its length. A baffle and reduced flow velocity in E-6 is expected to result in 98 to 99% removal of the entrained salt from the gas phase.

The gas flows out of E-6 down a 4 in. line to E-3 where it passes over the pool of salt in this venturi feed tank and then to E-4. The de-entrained salt collected in E-6 flows out through a standpipe (weir) and into the 4 in. line through which the gas is flowing. The lower gas velocity in this line will not result in appreciable re-entrainment of the salt, the bulk of which is thereby recycled to the venturi feed tank. The standpipe in E-6 serves a dual purpose. It provides a constant head to facilitate control of the small flow through the salt cooler (E-7, Figure 1) of 400 to 600 cm^3/min . This must be controlled by the bellows sealed valve between the salt cooler and the re-generation column, E-5. The standpipe also skims off floating particles which will be washed back to the venturi feed tank. The latter contains a similar standpipe through which excess salt can be bled off. The bleed will carry away accumulating particles.

ELEVATION B-B

ELEVATION A-A

FIGURE 2. Support Structure

As was pointed out in last quarter's report,⁽¹⁾ a larger volume flow of salt is required for efficient particle removal than is needed for sulfur removal. The recycle flow from the coarse de-entrainer to the venturi feed tank will be 3.0 to 3.4 liters/min, some 6 to 8 times the flow to the regeneration column.

Returning now to consideration of the gas flow, gas entering E-4 will encounter two bubble cap trays as it passes upward through this extraction column. Regenerated salt, combined with fresh makeup salt, flows downward through this column counter to the flow of gas. These two additional extraction stages further reduce the sulfur content of the gas which next passes through a 12 in. diam packed bed de-entrainer which comprises the upper section of E-4. The gas emerging from E-4 is the clean product gas and will be combusted in the secondary burner for disposal.

E-7, Figure 1, shows the position of the salt cooler. Preheated air will cool salt flowing through 1.5 in pipes. Only the straight sections of pipe will be cooled and only these sections will be packed with 0.5 in. x 0.5 in. hollow Al_2O_3 cylinders (center hole 3/16 in. diam). The overall length of the packed tube heat exchanger portion of the salt cooler will be 19 ft. Air and salt will be in cocurrent flow with the inlet air at $\sim 400^\circ\text{C}$ and inlet salt at 700°C . The exit salt temperature will be 500°C and, depending on flow rate, the exit air will approach the salt temperature as a maximum. At present we are considering the use of an injector to recycle hot air and will control the inlet temperature by means of a bleed.

The purpose of the packed tube is to produce turbulence in the small salt flow and thereby obtain a higher effective heat transfer coefficient. Without the packing the salt cooler is appreciably larger (~ 31 ft long).

To form the shell around the packed tubes, a sheath of stainless steel sheet of about 16 gauge will be used. End fittings will be formed to fit loosely around the tube. This fitting can be packed with "Kaowool" insulation and secured with a clamp to hold the slight air pressure. Trace heaters must be installed over the shell so inadvertently frozen salt within the packed tube can be remelted if required.

The cooled salt will flow to the top tray of an eight tray bubble cap column, E-5, and downward counter to a stream of steam/ CO_2 (67/33). Sulfur stripped from the salt emerges at a H_2S concentration of $\sim 12\%$ which could be increased to $\sim 30\%$ following condensation of the steam (condensation is not contemplated). This gas will be combusted and vented via the stack. Salt from E-5 flows to E-2, the salt makeup tank. The pot cover on the 24 in. diam pot in this furnace is being remodeled to permit easy addition of makeup salt. The atmosphere over this pot will be at the same pressure and of the same composition as the gas entering E-5 (67/33 steam/ CO_2). A pressure of 1 to 2 psig may be present. Release of this pressure would upset flow of gas in E-5 and of salt to E-4. Design of a small lock hopper is being considered as a solution to this problem.

Salt flows out of this pot to E-4 which is a 10 in. diam, two tray bubble cap column. After extraction of H_2S from the counter currently flowing gas, the salt flows to E-3 and combines with the recycle flow from E-6.

B. Stress Problems

The 2.5 in. pipe leading from the venturi to the coarse de-entrainer, E-6, is to be fabricated in three sections due to limitations on the length of pipe sections which could be "alonized." This line is approximately 30 ft long and upon heating to 700°C will grow about 5 in. in length. Such expansion could produce intolerable stresses in this pipe or in connections to E-6. In addition, any distortion (bending) could have a destructive effect on brittle trace heaters. Similar considerations apply to the 4 in. line connecting E-6 and E-3.

Stresses in these and other pipes and vessels have been analyzed for thermal and deadweight loadings using the Swamson Analysis Code ANSYS. This code is extremely well suited to piping analysis. Specific piping components and supports can be modeled quite exactly from its wide element library. The code is noted by many authorities as the industry standard for sophisticated structural analysis.

To maximize system integrity, two basic criteria for piping stresses were considered. The first criteria is that primary stress, i.e., stress due to deadweight and external loads, be kept below approximately one-fifth yield strength at operating temperature. Material strength at 730°C was estimated at about 11 ksi. Deadweight loading primary stress intensities nominally calculated by the ANSYS program were therefore kept below 2000 psi.

The secondary stress criteria for thermal loading was two pronged. Secondary stresses from thermal loading are self-relieving for ductile materials, a fact long recognized by established piping codes. What this means is that stresses calculated on the basis of elastic analysis can exceed the elastic limits of the material for certain loadings and the integrity of the system will in no way be compromised. A system will, in fact, shakedown to where it is cycling completely elastically under thermal expansion loadings if elastically calculated stresses are kept below twice the high temperature yield strength of the material. The system will "relax" itself. For this reason a combined maximum stress intensity limit was set at approximately 1-1/2 times the high temperature yield strength of the material. The second prong of this secondary stress critical deals with the stresses in the 2-1/2 and 4 in. long vertical risers. These runs, being very long, displace significantly due to thermal expansion, driving the coarse de-entrainer vessel nearly 5 in. For these reasons it was decided to limit maximum stress intensity due to combined loading in these runs to approximately one-half the high temperature yield stress. These values seem to indicate fairly high stress values but they are the combination of direct stresses and peak nominal bending stresses. The vast majority of the stresses indicated in the piping was due to pipe and elbow flexure accommodating length changes and does not reflect direct tensile or compressive stresses due to restraint. Direct stresses were carefully monitored so that large loads would not be transmitted to the support structure. For example, direct stress in the vertical runs was adjusted to be less than 100 psi under combined loading.

Pipe and vessel supports were selected from standard manufacturers equipment. To accommodate the large displacement of E-6, this vessel will be suspended from a constant rate support. This is a compression spring mechanical

device which yields a constant supporting force at all positions throughout its travel. Additional spring type supports on the 25 in. line just above the venturi and on the 4 in. line near the bend leading to E-3 derive from the modeling study. Trace heaters on these long 25 in. and 4 in. pipe runs will be interlocked electrically to prevent development of stresses in the event the heaters should fail in one line but not in the other.

A review of the piping support structure was conducted. Some minor detailing and bracing changes were made to reflect standard structural practice and to aid in the erection of the structure. The structure is quite tall for its base width, but is deemed adequate for a conservatively applied 20 psf wind loading. The deadweight design is conservative as to floor beam spans and column stresses. The item that seemed to cause the most concern was the lack of footings for such a tall structure. Calculations show that the 6 in. slab provides adequate footings for this structure under wind loading both at minimum structure weight and at a maximum structure weight equivalent to 100 psf loading on all the gratings.

C. Extraction and Regeneration Column Design Details

Orders have been placed to the low bidder for the extraction and regeneration columns E-4 and E-5. Details of their construction are illustrated by Figures 3 and 4.

The extraction column is to be built in two sections to facilitate "alonizing." The column shell is 10 in. schedule 40, 304 stainless steel. Each tray contains four bubble caps and a downcomer of 1 in. schedule 40 pipe. Provision for sampling the vapor space above each tray via 3/8 in. schedule 80 pipe has been made. The inlet salt composition can be sampled at E-2 and the outlet salt composition can be sampled at E-3.

The bottom section of the column is formed from a 10 in. pipe cap welded to the pipe. Passing through this cap is a section of 5 in. schedule 40 pipe. This is the flowmeter reservoir. A radioactive source and Geiger-Mu1ler detector will be used to sense the filling and emptying of this reservoir due to siphoning through a siphon leg (not shown, cf. Figure 1). The rate of filling and emptying will be proportional to the average salt flow rate, to be established by calibration.

The packed bed de-entrainer is being remodeled to fit on top of the upper section, (14), of the extraction column. The 12 in. I.D. column portion has been cut off the old pot cover (which will be discarded). A 10 in. flange will be welded onto the de-entrainer at the appropriate spot on the reducer where the diameters match. This requires machining away the "aluminumizing" in the vicinity of the weld. The unit, including the weld zone will be "realuminumized."

The scrubbing action of the two bubble cap trays will help to remove particles and entrained salt generated by the venturi which were not removed by the coarse de-entrainer. Subsequent passage through a packing of 1/2 in. x 1/2 in. Al_2O_3 cylinders, each having a 3/16 in. diam hole down the center, is expected to reduce entrained salt to requisite levels.

Figure 4 illustrates details of construction of the 6 in. diam, eight tray bubble cap column to be used for salt regeneration. Salt from the cooler, E-7, flows to the top tray of this column which is designed to permit air cooling. The regeneration reaction is exothermic and the largest heat effect occurs on this first tray. Cooling may not be required, but the capability to do so has been incorporated into the design.

The regeneration column has been split into three sections to facilitate "aluminumizing." As with the extraction column, interstage gas sampling is provided for. Inlet and outlet salt compositions can be deduced from salt samples taken at E-6 and E-2.

The bottom section of the regeneration column forms the reservoir of the salt flowmeter which will fill and empty cyclically due to the action of a siphon leg through which the salt leaves this column (not illustrated). Here too, a gamma ray densitometer will sense the condition of the reservoir.

Both columns will be encased in trace heaters, insulation, and weather-proof outer covering. All lines and portions of vessels which carry or contain a bulk salt phase will be fitted with safety troughs to collect and divert the salt to containers should a line or vessel rupture or develop a serious leak.

IV. PROPOSED WORK DURING THE NEXT QUARTER

Design of electrical circuitry, control systems, and instrumentation should be well underway. Support structure construction should be completed and preassembly of piping and process vessels preliminary to "aloning" should be nearly completed if reasonable delivery of the piping and associated vessels can be obtained.

V. OVERALL PROJECT STATUS AND CONCLUSIONS

During this quarter detailed design and drafting has been completed except for a few days remaining work on the support structure. To say that the design is complete should be qualified. This is true only for those items which effect the time line. Numerous design details remain to be completed, but there will be time for this while pipe is being fabricated, installed, or "aloned." On the whole, we appear to be about 2 months behind the schedule set forth in the work plan submitted in March 1976. This has been due to evolution of a process design of considerably greater complexity and capability than envisioned earlier.

VI. REFERENCES

1. R. H. Moore, D. G. Ham, and G. E. Stegen, "Process for Cleaning and Removal of Sulfur Compounds from Low Btu Fuel Gases," Interim Report April-June, 1976, BNWL-2040-2, UC-90c, July 1976.

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