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**Quarterly Summary Report
January-March 1977
Process for Cleaning and
Removal of Sulfur Compounds
from Low Btu Gases**

April 1, 1977

Prepared for the Energy Research
and Development Administration
under Contract EY-76-C-06-1830

Battelle

Pacific Northwest Laboratories

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QUARTERLY SUMMARY REPORT
JANUARY-MARCH 1977
PROCESS FOR CLEANING AND REMOVAL
OF SULFUR COMPOUNDS FROM LOW BTU GASES

by
R. H. Moore
D. G. Ham
D. H. Mitchell
R. J. Robertus

April 1, 1977

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I. OBJECTIVE AND SCOPE OF WORK PHASE IV

In this phase of work the 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) During the month of January 1977 Tri-Cities Machine and Fabrication completed assembly and erection of the new support structure for the PDU. This work included removal of portions of the old structure, relocation of one large pot furnace, and addition of 20 ft to the stack.

(2) In February 1977, all pipe and equipment fabricated by Metaltronics Inc. was received and during the latter part of February and through March this was assembled and mounted on the support structure. This was a preliminary check of fit. At the end of March, all of this equipment was disassembled and crated and shipped to Alon Processing, Inc. (Tarentum, PA) for "aluminumizing," a process which provides corrosion protection.

(3) Procurement activities continued in parallel with this effort. On a low-bid basis orders have been placed for all electrical gear with which to power the trace heaters (all of which have been ordered), all electrical controllers, and all electric signal measuring equipment. In addition a tar trap, all Grayloc fittings, all gaskets, all thermocouple wire, ceramic beads, and ceramic thermocouple sheaths have been ordered. Much of this equipment has been received.

- (4) Requirements for gas and liquid sampling have been analyzed. It is planned to assemble the analytical equipment and analyze synthetic samples to resolve problems and train personnel.
- (5) Theoretical analysis of this process to devise a computer model useful in data analysis and operating guidance is proceeding.
- (6) Revisions to the Precedence Diagram indicate additional schedule slippage occurred due to delays in receipt of Grayloc flange components and difficulties with preliminary assembly of equipment.

III. TECHNICAL PROGRESS

A. Construction and Assembly

Early in January Tri-Cities Machine and Fabrication assembled the support structure shown in the photograph (Figure 1). The first deck above grade was part of the original structure. All above this deck is new. The stack has been extended an additional 20 ft in height which necessitated taking it down, welding on a segment, erecting it, and adding additional guy wires. In addition, a large pot furnace weighing about 2000 lbs, which originally was installed on the first level above grade, was relocated to the second level above grade. Except for minor painting touchup (which could not be done in prevailing cold weather) this task was completed in mid-January.

Shortly thereafter, pipe and equipment fabricated by Metaltronics Inc. (Portland, OR) began arriving. Their shipment was completed near the end of February having been delayed by slow delivery of Grayloc hubs. As this material accumulated, work was begun on its assembly and mounting on the support structure. It turned out to be inefficient, if not actually impractical, to assemble a few pieces when other key pieces had not yet been received. This is a mobile suspension with the long runs of pipe and the coarse de-entrainer hung from "constant rate" support devices. Thus, these have to be positioned to allow for thermal expansion relative to the extraction and regeneration columns and the venturi feed tank, which were "fixed" by their mounting brackets, which attached to the support structure. It

FIGURE 1. Process Development Unit Support Structure and Stack

proved necessary to essentially complete the assembly before we could be certain that everything fit together correctly and was able to expand properly without developing stresses or distortion. This taxed the skills of our crafts and engineering personnel but was accomplished with but a slight schedule overrun, some of which could be traced to adverse weather.

When assembly was completed photographs were taken and critical measurements were made and recorded. It was difficult to get a good perspective in the photographs (Figures 2-5) because the photographer had to be too close (in the absence of a sky hook). Figure 2 shows a view taken on the uppermost level of the structure showing the yoke on which the "constant rate" support devices are mounted. Two slender 112-in. threaded rods connect these devices to the support bracket from which the coarse de-entrainer hangs. A 2-in. and a 4-in. line, both of which are partly supported by additional spring-loaded hangers also are supported by these devices. Up to 5.5 in. of expansion in the 2-in. and 4-in. lines is anticipated and can be accommodated.

figure 3 is a view of the regeneration column, viewed from top to bottom. The column, actually about 11 ft in overall length, appears somewhat elongated in this photograph.

Figure 4 is a view which shows the 4 in. line extending across the first deck above grade and entering the venturi feed tank. A 4 in. line also emerges from the top of this tank and enters the lower portion of the extraction column. Figure 5 shows an overview. The 4-in. line hanging from the "constant rate" supports is also supported by a spring hanger near the bend where it turns and comes across to the venturi feed tank. The length of this line and the problems of supporting it are clearly illustrated by this photograph.

Though leaving much to be desired it is hoped that these photographs together with drawings in an earlier report⁽¹⁾ will give the reader some conception of the physical size and complexity of this PDU. Its construction is proving to be challenging.

⁽¹⁾Moore, R.H. et al., "Quarterly Summary Report July-September 1976. Process for Cleaning and Removal of Sulfur Compounds from Low Btu Fuel Gases," BNWL-2040-3, October 1976.

FIGURE 2. Constant Rate Supports to Which Pipe and Coarse De-entrainer Are Attached

FIGURE 3. View Looking Down on the Regeneration Column Showing Taps for Sampling and for Pressure and Temperature Measurement

FIGURE 4. View Showing 4 in. Jumper Connecting the Extraction Column and the Venturi Feed Tank

FIGURE 5. Overview Showing 2.5 in. and 4 in. Lines and Coarse De-entrainer Hanging from the Constant Rate Supports. The extraction column and packed bed de-entrainer appear at lower left.

With completion of measurements and photographs all of this pipe and equipment, which had been assembled with considerable difficulty, was disassembled and lowered to the pad in a day. It was then crated and shipped to Alon Processing Inc., Tarentum, PA where it will be made corrosion resistant by "aluminizing." This is a process for diffusing Al into the metal surface to produce Fe-Al intermetallic compounds which are highly resistant to corrosion by sulfide bearing melts.

B. Process Design and Procurement

Paralleling these construction activities, extensive procurement activity has continued. Mr. Don H. Mitchell, working with Mr. Lee Higginson, has assumed the responsibility for procurement of all electrical gear, electrical control equipment, and measurement equipment.

The electrical trace heating requirements are far more extensive in the remodeled PDU than in the old plant. There are 29 circuits as opposed to 4 in the old plant. Type K chromel-alumel thermocouple sensors and solid state proportional controllers will actuate magnetic reactors (relays) to control the trace heaters.

Extra control thermocouples will be peened into the pipe and equipment walls to facilitate changes required if failures occur. In addition, extra thermocouples will be spaced along the pipes and on equipment and scanned with a digital data logger to sense and report the temperatures. This device will alarm and shut down a circuit if an overtemperature situation is detected. Eight circuits on the more critical equipment items will be protected in this manner. All required electrical gear, housings, transformers, wiring channels, controllers, and the data logger are now on order.

All thermocouple wire has been received and beaded thermocouple assemblies are currently being fabricated. Approximately 8000 ft of thermocouple lead wire will be required and has been received.

A supply of preheated air to cool salt flowing from the coarse de-entrainer to the regenerator is required. The blower which supplies air to the gasifier and to the secondary burner (in which gases are combusted prior to disposal via the stack) is adequate to supply this air requirement.

Previous calculations on the salt cooler design problem show a maximum air rate of 80 scfm (with air entering at 400°C). To heat this much air from ambient temperatures to 400°C requires approximately 17.5 kW. For beds with 1-in. SS Pall Ring packing, heat transfer calculations indicate a bed 8-in. dia by 4-ft long will be adequate.

Furnaces on the preheater will be controlled by a 3 mode "on-off" controller with a thermocouple which senses exit air temperature. Air flow control is by a thermocouple sensor monitoring salt temperature as the salt leaves the cooler. This thermocouple signal actuates a proportioning controller which operates a motor driven butterfly valve in the air supply line.

At present, trace heaters for this preheater are to be ordered. We are awaiting delivery on the controllers and the motor driven valve. Miscellaneous piping and fittings will be ordered as needed.

The steam-CO₂ (2:1 mole ratio) supply to the regeneration column (design basis: 1.5 lb-moles/hr) must be preheated to 500"-550°C. In order to accurately measure steam flow, the steam must be superheated. Steam from our steam generator will be heated to about 400°F in a heater traced section of 1-in. stainless pipe 8 ft in length. Superheated steam flow will be measured by an Annubar flow sensor and Magnahelic pressure gauge.

CO₂ will be supplied from a 6-ton liquid CO₂ tank. Its flow will be measured with a rotometer.

The combined steam-CO₂ streams will flow to a preheater which consists of a 24-in. deep bed of 1-in. SS Pall Rings in an 8-in. dia schedule 40 stainless steel pipe. A thermocouple sensor in the gas stream exiting the heater will provide input to controllers (3 mode, on-off) which control the trace heaters for this unit.

Bids for the CO₂ supply are currently under review. We are awaiting delivery of the Annubar flow meter, controllers, and trace heaters. We need to place orders for pressure gauges, the CO₂ rotometer and miscellaneous pipe, valves, and fittings.

Both the steam-CO₂ preheater and air preheater will be fabricated locally and assembled onsite. This work will be underway within a few weeks.

The gasifier will be operated on metallurgical coke to bring all systems to proper temperature, then a switch to a non-caking coal will be made. This will result in the production of tars and oils with which we have not previously had to contend. It is planned to install a tar trap in the 6-in. line immediately below the gasifier outlet. This is expected to remove only a portion of the tars and oils, but will reduce the problems of fouled and plugged lines leading to the venturi scrubber where first encounter with the molten salt occurs. We anticipate no problem with tars beyond this point.

The tar trap contents will be drained to steel drums for sampling and ultimate disposal via landfill. The trap is presently on order.

Bid packages have been prepared for procurement of gamma ray densitometers for use in measurement of salt flow. With the aid of these devices the siphoned emptying and subsequent refilling of chambers at the bottom of the extraction and regeneration columns will allow measurement of the average salt flow rate.

At present, procurements are about 90-95% complete. Items which remain are mainly small items which will be procured as needed. An exception is the coal, which will be ordered when the plant nears operational status.

C. Theoretical Studies

Modeling effort has concentrated on developing and checking computational algorithms to be used in the final computer code. The checking procedure uses simplified forms of the equations (i.e. ideal stages and isothermal operation in the venturi and extraction column) and programs these on an HP-67 calculator. This allows checking for consistency in signs and convergence of iteration schemes. This seemingly double effort has already paid off in terms of savings in computer costs for debugging.

Another reason for developing the calculator codes is to have them available as guides for operation of the demonstration unit this summer. The calculator programs would be available at all times. Thus, when an upset is occurring they could be used to determine magnitudes of changes that should be made to bring the system back in line.

To date two such calculator programs have been completed. The first describes just the venturi. With inlet conditions completely specified (i.e. flows and compositions for gas and liquid streams) it will predict gas and liquid outlet compositions. Effects of changing temperature, pressure and efficiency can also be determined.

A second calculator program uses the same algorithm for the venturi (except it is assumed to be one equilibrium stage) then steps off two ideal stages in the extraction column. This fixes all conditions for the column. A simple material balance then determines conditions out the regenerator. Memory limitations restrict the program to consideration of a system with no fresh input or bleed streams of molten salt.

Effects of changes in process variables can be determined for a wide range of conditions. Some examples are shown in Table 1. The first case represents design conditions. All other cases change only one or at most two values from the design level. All data shown are for 1 atm and 700°C conditions in the venturi and extraction column. (Other temperatures and pressures can be specified.) The reader should note that the first six columns of the table are required input information. Outputs are gas composition out the extraction column and regenerator plus liquid purity required out the regenerator.

The input values shown in the table are all $\pm 10\%$ from the design case. Output values are to be used here only to show trends. Absolute values are not accurate because of the assumptions of ideal stages in the venturi and absorber. The trends are however quite interesting.

Consider all the cases showing a zero m.f. $M_X S$ out the regenerator. All of these represent physically unachievable situations. The diagram for the extraction column describes what is happening (Figure 6).

Point B on the diagram is known. The value for Y_{in} is the gas concentration leaving the venturi. A material balance at the venturi feed pot fixes X_{out} . The slope of the operating line is also known. There are two stages in the extraction column so stepping off two equilibrium stages gives inlet liquid and outlet gas compositions. Negative values for X are physically meaningless. The calculator program checks for negative X values but uses $X=0$ to complete the regenerator material balance.

TABLE I. Results of Calculator Analysis of Computer Codes

Venturi					Regenerator			
Gas Comp In m.f.H ₂ S	Gas Flow In Moles/Hr	Liquid Comp In m.f.H ₂ S	Liquid Flow In Moles/Hr	Liquid Recycle Ratio	Gas Flow In Moles/Hr	Gas Out Reg m.f.H ₂ S	Comp. Out Extr m.f.H ₂ S	Liq. Comp. Out Reg m.f.H ₂ S
0.0131*	12.54*	0.1227*	8.56*	5.90*	1.47*	0.1235*	0.0002*	0.0067*
0.0144	12.54	0.1227	8.56	5.90	1.47	0.1325	0.0005	0.0000
0.0118	12.54	0.1227	8.56	5.90	1.47	0.1212	0.0004	0.0202
0.0131	13.79	0.1227	8.56	5.90	1.47	0.1322	0.0005	0.0000
0.0131	11.29	0.1227	8.56	5.90	1.47	0.1079	0.0004	0.0202
0.0131	12.54	0.1350	8.56	5.90	1.47	0.1208	0.0004	0.0209
0.0131	12.54	0.1104	8.56	5.90	1.47	0.1183	0.0003	0.0000
0.0131	12.54	0.1104	9.42	5.90	1.47	0.1215	0.0004	0.0191
0.0131	12.54	0.1227	7.70	5.90	1.47	0.1175	0.0005	0.0000
0.0131	12.54	0.1227	8.56	6.49	1.47	0.1192	0.0005	0.0000
0.0131	12.54	0.1227	8.56	5.31	1.47	0.1213	0.0004	0.0197
0.0131	12.54	0.1227	8.56	5.90	1.62	0.1108	0.0002	0.0067
0.0131	12.54	0.1227	8.56	5.90	1.32	0.1395	0.0002	0.0067
0.0144	12.54	0.1104	8.56	5.90	1.47	0.1200	0.00019	0.0000
0.0118	12.54	0.1350	8.56	5.90	1.47	0.1047	0.00066	0.0347
0.0144	12.54	0.1227	9.42	5.90	1.47	0.1377	0.000178	0.0070
0.0131	13.79	0.1227	9.42	5.90	1.47	0.1375	0.00018	0.0068

Design Condition

FIGURE 6. McCabe-Thiele Diagram for the Extraction Column

When the situation occurs in the detailed computer analysis, a flag will be set and a new minimum permissible liquid flow rate will be calculated and used in the subsequent regenerator analysis. Note that in some cases, just using an efficiency less than one will give results which (in theory at least) are realistic. Raising the liquid flow will increase the slope of the operating line. (Granted a decrease in gas flow would also do the same, but in a commercial plant, adjusting gas flow rates would be less desirable than changing liquid flows.) Then, the stepping procedure will give reasonable values at the top of the extraction column.

The number of zeros in the last column suggests that design conditions are very close to minimum allowable liquid flows in the regenerator extraction column loop. Likewise the concentration of sulfur in the salt entering the venturi is close to the lower limit achievable. Fortunately, however, the system is flexible and more than one variable can be changed to control an

upset. The last series of cases in Table I illustrate effects of changing more than one variable at a time. One can see that both a 10% increase in gas flow or a 10% increase in sulfur content of the gas can be compensated for by a 10% increase in liquid flow. On the other hand, a 10% increase in sulfur content of the gas cannot be controlled by just a 10% decrease in salt sulfur content. Some increase in flow would also be necessary.

The reader can deduce other more or less expected trends by examining the rest of the table. Again, it must be realized that these numbers are for a highly idealized description of the process.

The computer code will definitely account for nonisothermal operation in the regenerator and also, should it occur, in the extraction column or venturi. Plans are to handle two types of efficiency calculations. One is simply to input a guess then compare predicted outputs with measured ones. Different guesses can be made for efficiency until the two data sets agree. The other method is to calculate an efficiency from tray configurations and flow properties as was done in the actual design stages.

The computer program is, however, to be an operational tool more than a design aid. Hence, detailed theoretical analysis such as determination of mass transfer coefficients is not anticipated in the early stages, at least.

Time permitting, effects of entrainment in the extraction column and regenerator will be modeled. Pressure drop correlations are not seen as an important part of the analysis with the possible exception of the venturi. Hence, they will be deferred until a very theoretical treatment is warranted.

The total modeling project is estimated at 35-40% complete. Computer coding should proceed rapidly after logic is debugged using the calculator routines.

A computer code has also been developed to facilitate calculation of temperatures in the regenerator. The following assumptions are made:

- 1) Salt inlet temperature and composition plus outlet composition are known.
- 2) Gas inlet temperature and composition plus outlet composition are known.

- 3) Column operating pressure is specified.
- 4) Equations for equilibrium data are available (we have them for the ternary eutectic and for a quaternary system containing CaCO_3).
- 5) For an arbitrarily small increment of volume in the column, the temperature of the gas leaving the volume is the same as the temperature of the salt leaving the volume, i.e., thermal equilibrium is achieved. (Attempts will be made later to verify or refute this assumption.)

The analysis then proceeds as follows using a basis of unit time:

- 1) The total amount of sulfur removed is calculated.
- 2) The column is divided into "N" arbitrary increments of equal sulfur removal.
- 3) Enthalpy balance equations are generated for the "N" increments (shown below).
- 4) The resulting "N" equations are solved to give values of temperature (gas or liquid) leaving each element.
- 5) Values of $Y_{\text{H}_2\text{S}}$ and $X_{\text{M}_x\text{S}}$ for the operating line are calculated from a material balance on each element.
- 6) Equilibrium values $X_{\text{M}_x\text{S}}$ are calculated for the selected $X_{\text{H}_2\text{S}}$ and corresponding $X_{\text{M}_x\text{S}}$ eq temperature just calculated above.

The following equations and definitions apply:

Reaction Occurring

Total Sulfur Removed

$$\Delta S_T = L_{N+1}X_{\text{M}_x\text{S}_{N+1}} - L_1X_{\text{M}_x\text{S}_1} = G_NY_{\text{H}_2\text{S}_N} - G_0Y_{\text{H}_2\text{S}_0} \quad (2)$$

Material Balances

$$L_1 = L_2 \dots = L_{N+1} \quad (3)$$

$$\Delta S_{INC} = \Delta S_T / N \quad (4)$$

$$G_i = G_0 - i * \Delta S_{INC} \quad (5)$$

Enthalpy Balance

Input = Output + Accumulation

$$G_{i-1} C_{P_G} (T_{i-1} - T_{REF}) + L_{i+1} C_{P_L} (T_{i+1} - T_{REF}) =$$

$$G_i C_{P_G} (T_i - T_{REF}) + L_i C_{P_L} (T_i - T_{REF}) + \Delta S_{INC} * \Delta H_{R_{EAX}} \quad (6)$$

Equilibrium Expression

$$K_{EQ} = \exp(B_0 - E'/T) = \frac{x_{M_X S}}{x_{M_X CO_3}^* - x_{M_X S}} \frac{y_{CO_2} y_{H_2O}}{y_{H_2S}} P_T \quad (7)$$

Where:

- G's are molar gas flow rates
- L's are molar liquid flow rates
- X's are mole fractions in liquid phase
- Y's are mole fractions in gas phase
- N = total number of increments chosen (arbitrary)
- $\Delta H_{R_{EAX}}$ = heat liberated per mole of H_2S reacting
- C_{P_L} = heat capacity of liquid
- C_{P_G} = heat capacity of gas
- $\Delta S_{INC} = \Delta S_{Total} / N$
- x^* = mole fraction "active" carbonate in salt, i.e., (Na + Ca values)
- P_T = total pressure of system.

Computed temperature profiles are shown (Table II) for three cases:

- I. 1 atmosphere operation with ternary eutectic
- II. 20 atmosphere operation with ternary eutectic
- III. 20 atmosphere operation with CaCO_3 added to the ternary mixture.

For Case I, data in Table II show the regenerator is highly nonisothermal. This is due to the low salt flow combined with high heat of reaction. Most of the temperature change is in the top of the column (i.e., the first stage when the equilibrium plot is drawn).

For Case II (20 atm) very high salt flow rates allow nearly isothermal operation. Adding CaCO_3 (Case III) at 20 atm decreases the required salt flow, and again the column operates nonisothermally. Stage requirements were determined graphically.

TABLE II. Temperature Profiles for the Regeneration Column

<u>Case I</u>		<u>Case II</u>		<u>Case III</u>	
NC	T-DEG C	NC	T-DEG C	NC	T-DEG C
1	621.437	1	557.091	1	595.331
2	639.411	2	556.483	2	601.744
3	641.427	3	555.923	3	600.793
4	638.393	4	555.363	4	598.321
5	633.774	5	554.803	5	595.540
6	628.667	6	554.244	6	592.699
7	623.418	7	553.685	7	589.849
8	618.133	8	553.126	8	587.000
9	612.846	9	552.567	9	584.155
10	607.568	10	552.009	10	581.314
11	602.301	11	551.450	11	578.477
12	597.047	12	550.892	12	575.645
13	591.806			13	572.816
14	586.578			14	569.992
15	581.362			15	567.171
16	576.159			16	564.355
17	570.969			17	561.542
18	565.791			18	558.734
19	560.626			19	555.930
20	555.474			20	553.130
3.0*		2.0*		2.4*	

* Number of theoretical stages required.

D. Program Management

The Precedence Diagram shown in last quarter's report has been updated (Figure 7). It is estimated the schedule shown here has slipped one month from that shown in last quarter's report. This was due primarily to slow delivery of material. Difficulties encountered during preliminary assembly also contributed. The schedule shown on Figure 7 is highly dependent on the ability of Alon Processing, Inc. to perform as indicated.

IV. PROPOSED WORK DURING THE NEXT QUARTER

As much of the electrical work as can be completed before the "alonized" pipe and equipment are returned will be undertaken during this quarter. This work is also somewhat contingent upon availability of materials now on order.

Pipe to be utilized for the air and steam CO₂ preheaters does not require alonizing so fabrication of these units can proceed. This work will be performed locally. Remaining procurements will be completed during this quarter and computer modeling should be completed.

V. OVERALL PROJECT STATUS

Procurement of materials and equipment is about 90-95% complete. All pipe and equipment to be "alonized" has been preassembled and shown to fit properly and is now on its way to Alon Processing, Inc. for treatment. With return of this equipment, anticipated about June 1, final assembly and completion of the PDU can begin in earnest.

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FIGURE 7. Precedence Diagram

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