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

Interim Report for the Period
April-June 1976

by
R. H. Moore
D. G. Ham
G. E. Stegen

July 1976

Prepared for the Energy Research
and Development Administration
under Contract E(45-1)-1830

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PACIFIC NORTHWEST LABORATORY
operated by
BATTELLE
for the
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Under Contract E(45-1)-1830

Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22151
Price: Printed Copy \$5.00; Microfiche \$2.25

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Battelle
Pacific Northwest Laboratories
Richland, Washington 99352

QUARTERLY ~~SUMMARY~~ REPORT
APRIL - JUNE 1976
PROCESS FOR CLEANING AND REMOVAL OF
SULFUR COMPOUNDS FROM LOW BTU FUEL GASES

I. OBJECTIVE AND SCOPE OF WORK

Phase IV. In this phase of work it is proposed to remodel the PDU by incorporation of appropriate sub-systems 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) ~~As~~ a consequence of the redirection of this program which occurred in March 1976, a revised work plan was prepared and submitted near the start of this report period.
- (2) A conceptual process design has been outlined in considerable detail.
- (3) Detailed design calculations for critical elements of the PDU including:
 - (a) The stripping column for salt regeneration,
 - (b) The extraction column,
 - (c) Salt cooler, and
 - (d) Miscellaneous piping runs, ceramic lined venturi, and process vesselshave been completed and design drafting to prepare drawings and procurement specifications is in progress.
- (4) A detailed analysis of the process design problem and resultant solution is presented.
- (5) The PDU, currently configured in batch mode, has been partially disassembled in preparation for revisions to existing components and installation of added equipment required for conversion to continuous mode.
- (6) Referring to the schedule shown in Figure 1, Task 2 is complete and Task 3 is approximately 50% completed which implies a slippage in the schedule of 4-6 weeks. Tasks 4 and 5 are delayed as a result. Delays

• Long lead time procurements

FIGURE 1 Schedule Of Work

must be attributed to the unusual complexity of the design problem and to a considerable degree of optimism in the initial scheduling.

- (7) Two invention disclosures have been submitted during the report period.
- (8) A Quality Assurance Plan has been formulated in cooperation with our quality assurance specialist.

III. TECHNICAL PROGRESS

A. Conceptual Process Design

Battelle's molten salt scrubbing process has been operated in batch process mode with continuous recirculation of the extracting salt from the de-entrainer to the venturi scrubber. In this configuration, operation began with contact of the gas with clean salt and terminated when the extraction capacity of the salt was exhausted. The salt was subsequently regenerated, again in batch mode, or was replaced with fresh salt. Highly efficient extraction of sulfur compounds and particle scrubbing was demonstrated and it was shown that a vertically oriented venturi scrubber was an efficient device with which to circulate the molten salt scrubbing liquid. No mechanical pump was required. It remains to be demonstrated that the salt can be continuously regenerated simultaneously with extraction of sulfur compounds and particles.

To achieve continuous operation with simultaneous extraction of sulfur compounds, particle removal, and regeneration of the extraction capacity of the salt a substantially revised process has been conceived. As before, a venturi scrubber is to be used to lift a gas salt dispersion to a de-entrainer. This is situated at an elevation such that the de-entrained salt returns by gravity to the venturi inlet after passage through various process steps. No mechanical pump is required. Figure 2 illustrates the process flow diagram with stream flows shown in Table I.

As illustrated in Figure 2, raw gas from Battelle's fixed bed gasifier will enter the venturi scrubber where the gas is accelerated to 250 ft/sec (or higher) in the throat area. Salt also enters the venturi in the throat area and is dispersed into a fine mist by energy transferred by the gas phase. This dispersion is lifted to the de-entrainer where >98% of the salt

*STREAMS ARE IDENTIFIED BY NUMBERS IN SQUARE BRACKETS, Cf TABLE I

FIGURE 2. Process Flow Diagram*

TABLE I Typical Process Stream Properties (Estimated) for Continuous Hot Gas Cleaning. Basis 75 SCFM Feed Gas 1.3% H₂S

Stream	Temp. °C	Pressure psig	Flow Rate, lb-moles/hour					Salt Phase Sulfur
			Flow Rate H ₂ + CO + N ₂	CO ₂	H ₂ S	H ₂ O	Salt	
1	750	2.25	10.5	0.63	0.164	1.25	0	0
2	700	1.5	10.5	0.75	0.046	1.37	8.56	1.167
3	700	1.33	10.5	0.75	0.046	1.37	*	*
4	700	1.33	--	--	--	--	7.32	0.998
5	700	1.33	10.5	0.75	0.046	1.37	7.32	0.998
6	700	1.33	--	--	--	--	1.24	0.169
7	550	1.33	--	--	--	--	1.24	0.169
8	600	1.0	--	--	--	--	1.24	0.011
9	110	1.0	--	0.49	--	0.98	--	--
10	500	1.0	--	0.49	--	0.98	--	--
11	580	0	--	0.332	0.158	0.822	--	--
12	700	0.75	--	--	--	--	1.24	0.011
13	700	1.29	--	--	--	--	1.24	0.052
14	700	0	10.5	0.79	0.006	1.41	--	--
15	700	2.25	--	--	--	--	8.56	1.05

* Salt carryover in stream 3 is considered part of stream 4.

coalesces to a bulk liquid phase. A portion of the de-entrained salt flows through a cooler and thence to a regeneration column where it is contacted counter currently with a steam + CO_2 mixture. This treatment converts sulfur compounds in the salt to gas phase H_2S which may subsequently be converted to sulfur in a Claus process." The regenerated salt flows on to the fresh salt make-up tank where it is reheated and fed to the top of the absorption column. Here the clean salt again encounters gas flowing from the de-entrainer and additional sulfur compound extraction occurs, this time in counter current flow. Salt leaving this column flows to the venturi feed tank.

A packed bed de-entrainer and an absorption bed sit on top of the extraction column. The de-entrainer will consist of a shallow bed (12-18 in. deep) of 1/2 in. Al_2O_3 rings, similar to Rashig rings, but of the type produced by Carborundum Corporation for catalyst supports. The de-entrainer packing removes entrained salt by a mechanism of inertial impaction resulting from the tortuous flow. This may also be true of absorptive materials such as active alumina, silica gel, or active carbon which are contemplated for use in a shallow polishing bed above the de-entrainer packing. Chemisorption may also contribute to the removal mechanism in the latter case.

B. Process Design Basis

In the continuing evolution of this molten salt scrubbing process it was decided to strive for a design which tends toward an economic optimum and which will provide data sufficient for scale-up, process optimization, and economic evaluation. Very little existing equipment will be discarded. Where possible, the various equipment items will be separately operable to simplify unit testing and plant shakedown.

The process is designed to remove sulfur compounds from low BTU fuel gas at temperatures up to 725°C (higher if satisfactory materials performance is indicated). Gas flows up to 75 SCFM will be processed with removal

* PNL is not required to demonstrate this conversion step. After condensation of steam, it is anticipated that H_2S will be combusted and disposed of with other stack gas.

of >90 percent of the sulfur compounds and with production of a gas containing <0.001 grains/SCF of particles 2-6 microns diameter. The use of mechanical pumps for circulating the molten salt was avoided in the present PDU and is to be avoided in the revised system.

Major process variables are listed below together with a qualitative description of the effect of changes in these variables. Where appropriate the design basis value of the variable is discussed.

(a) Inlet Gas Sulfur Content

As the inlet gas sulfur content increases, the regenerant gas H_2S concentration, regenerated salt sulfur content, and product gas sulfur content all tend to increase. If the product gas sulfur content is to be held constant despite an increase in the inlet gas sulfur content, this can be accomplished through some combination of increased salt flow, increased regenerant gas flow, reduced regeneration temperature, or an increase in number of absorption and regeneration stages.

For the purpose of the present design, the inlet H_2S concentration was set at 1.30%. This is roughly twice the concentration typically obtained by air gasification of high sulfur coals and near the upper limit for oxygen gasification of such coals.

(b) Outlet Gas Sulfur Content

This is a dependent variable and is proportional to the inlet gas sulfur content. It decreases with increased salt flow, decreases with increasing number of extraction stages, and decreases as the sulfur content of salt from the regenerator is reduced. Increased salt flow requires greater regeneration efficiency (increased number of regeneration stages, lower regeneration temperature, or increased steam + CO_2 flow).

For design purposes the outlet H_2S concentration has been set at 0.045%. This is the equivalent of less than 1/2 of the present EPA limit for SO_2 emissions from plants burning solid fuels (based on 150 BTU/SCF in the product gas and a standard of $1.2 \text{ lb } SO_2/10^6 \text{ BTU}$). This represents a removal efficiency of 96.5%.

(c) Regenerator Temperature

The regeneration reaction is exothermic and the position of the equilibrium is increasingly favorable at lower temperatures. Lowering the temperature for regeneration reduces the steam + CO_2 requirement and allows production of a gas of higher H_2S content. Decreased regeneration temperatures, however, require larger salt cooling capacity and/or installation of interstage cooling. In addition, increased heat input is required to reheat the salt to the absorption temperature. There is, of course, a low limit for the regeneration temperature due to the freezing point of the salt.

The pure ternary eutectic mixture of Li_2CO_3 , Na_2CO_3 , and K_2CO_3 melts at 394°C . Tests with Na_2S substituted for all of the Na_2CO_3 show adequate liquidity at 500°C . It seems reasonable to expect, however, that ash constituents solubilized by the salt may act to raise the freezing point of the mixtures. For design purposes a salt temperature of 550°C was assumed at the regenerator inlet and the column was assumed to operate adiabatically. Provision has also been made for cooling the top stage of the regenerator.

(d) Absorber Temperature

The extraction reaction is endothermic and the position of the equilibrium is increasingly favorable to extraction of sulfur compounds at higher temperatures. Higher temperatures result in increased corrosion-erosion of materials. A practical upper limit for the "alloyed" steels to be used here appears to be about 750°C . For purposes of this design calculation a temperature of 700°C was selected.

(e) Number of Theoretical Stages in Regenerator and Absorber

The number of theoretical stages in the absorber sets an upper limit on the regenerator outlet H_2S concentration (assuming gas product purity is fixed). An increase in the number of absorption stages allows attainment of a predetermined product gas purity despite an increase in the sulfur content of the extracting salt. This increased sulfur content, in turn, allows an increase in regenerant outlet gas H_2S concentration or a reduction in the number of regeneration stages. An increase in the ratio of absorber to regenerator stages will also result in decreased salt flow rates--a consequence of higher salt loading.

Because of the higher temperature and higher gas flow rate in the absorption section, it is anticipated that absorption stages will be more expensive than regenerator stages. In the present design, the number of absorption stages has been reduced somewhat by increasing the number of regeneration stages. This also lowers the regeneration gas flow rate.

(f) Regeneration Gas Rate and Composition

The regeneration gas (67% steam + 33% CO_2) rate is a dependent variable. The rate is increased when the ratio of absorber to regenerator stages increases...a consequence of lowered sulfur content in the salt. The rate is also increased by increased liquid flow rate or by increased regeneration temperature.

Since the cost of regeneration gas can be substantial it is important to minimize its use. It is also desirable to produce a gas high in H_2S concentration as feed to a Claus process. For design purposes, the regenerator off-gas has been specified at 12% H_2S (prior to steam condensation ...which yields 30% H_2S after steam is condensed).

(g) Liquid Flow Rate

Liquid flow to the regenerator and absorber is small in comparison to the gas flow...a consequence of the high capacity of the salt for extraction of sulfur compounds. The required flow would be insufficient for efficient particle removal in the venturi scrubber so a substantial recycle flow is utilized. The recycle stream does not pass through the regenerator or through the absorber columns. The recycle stream carries the sulfur extracted by the co-current flow contact in the venturi scrubber. For design purposes, the recycle flow rate has been set at 3 liter/min.

(h) Particle Removal

Particle removal (from the salt) will be by means of a bleed stream. Salt will be bled off at intervals to maintain a constant particle burden and/or a salt freezing point at a desired temperature. If, as is planned, the salt is the eutectic mixture of alkali carbonates, this can be freed of particles by dissolving the bleed salt in water, filtering to remove insoluble matter, and evaporating to effect crystallization of the mixed carbonates.

Particle loading in gas entering the venturi scrubber depends on the characteristics of the gasifier and any precleaning equipment (cyclones, etc.) in use. At present, insufficient data are available to specify a bleed rate required, though it is anticipated to be very low.

C. Specific Design Detail

(a) Absorber and Regenerator Design

The absorber design is based on a nominal gas flow rate of 75 SCFM and an operating temperature of 700°C. A product gas containing no more than 0.045% H_2S and an inlet gas of 0.37% H_2S is assumed.* The salt entering the absorber will contain 0.009 mole fraction alkali sulfide and should exit at a mole fraction of 0.042 at which concentration it flows into and mixes with recycle salt to be returned to the venturi inlet. The 700°C equilibrium curve and the venturi and absorber operating lines are shown in Figure 3. The absorber requires 1.5 equilibrium stages. Salt flow rate is 0.0206 lb-mole/min (approximately 470 ml/min).

The regenerator inlet and outlet salt contains 0.136 and 0.009 mole fraction alkali sulfide respectively. The inlet gas is 67% H_2O and 33% CO_2 and the outlet gas contains 12% H_2S .

Heat of reaction cannot be neglected in designing the regenerator. A tray-by-tray calculation was made taking the following assumptions into account:

- (1) Regenerant gas enters at 500°C.
- (2) Salt enters at 550°C.
- (3) The heat of reaction is 25 kcal/g mole.
- (4) Operation is adiabatic.
- (5) The average temperature of the gas leaving each plate is assumed to be equal to that of the salt on the plate.

The calculation then proceeds by simultaneously solving the heat balance, mass, balance, and equilibrium relations for each theoretical stage. This

* It should be noted that partial extraction of H_2S from gas entering the venturi at 1.30% H_2S has been achieved before the gas enters the absorber.

FIGURE 3. Equilibrium Curve at $700^\circ C$ and Venturi and Absorber Operating Lines.

calculation indicates that three theoretical stages are sufficient to provide the required regeneration efficiency. The maximum salt temperature inside the column is predicted to be 617°C and the outlet salt will be at 600°C. The required regeneration gas rate is estimated to be 8.8 SCFM, however, the column has been sized to allow operation at slightly more than twice this flow rate.

In designing both the absorber and regenerator it has been assumed that the chemical reaction is essentially instantaneous, i.e. mass transfer is rate controlling. Equilibrium data (Figure 3) were those determined in laboratory studies of the behavior of the $\text{Li}_2\text{CO}_3\text{-Na}_2\text{CO}_3\text{-K}_2\text{CO}_3$ eutectic system.⁽¹⁾ It was assumed, as indicated by the data, that NaCO_3 was the active species for reaction with H_2S .

A number of different types of contactors are in common use for gas scrubbing. Several types were considered for this application including columns with random dumped packing, bubble cap tray columns, sieve tray columns, spray towers, baffle plate columns, venturi scrubbers, and Doyle scrubbers. In selecting a contactor, one must consider the very low ratio of liquid to gas flow which characterizes this process. Because of the low liquid rate, a packed column is a poor candidate for the absorber. The regenerator, on the other hand, operates at the same liquid rate, but at about an eighth of the gas flow rate so a packed column might be feasible for the regenerator. Unfortunately, packed columns operate efficiently only over a narrow range of gas flow rates. This flow must be such as to offer resistance to liquid flow but not sufficient to reverse the liquid flow...a condition known as flooding. In order to study the desired range of gas flow rates and salt flows, it might be necessary to provide several columns of different diameter and packing size. As will be shown below, we have provided for brief study of packed columns.

Bubble cap tray columns provide considerable flexibility and maintain high efficiency over a wide range of liquid and gas flow rates. A large amount of test data has been published on their operating characteristics which allows fairly reliable design without extensive preliminary development work. Their major disadvantages are high pressure drop and relatively complex (and costly) fabrication problems.

Sieve tray columns tend to weep at low liquid flow rates and the small plate perforations may plug when the liquid contains suspended solids.⁽²⁾ Spray towers would be difficult to operate because of the low liquid head available. Also, erosion and plugging of small nozzles could be a problem. The Doyle scrubber is designed primarily for particle removal;⁽³⁾ however, it could also provide efficient sulfur compound absorption because of the intimate gas-liquid contact it provides. A series of venturi scrubbers could also be used. The venturi scrubber provides high stage efficiency but produces a fairly high gas pressure drop. A total of three venturi scrubbers would be required. One will be employed in the proposed process to attain the necessary salt circulation. It was felt that operation of the PDU will generate sufficient data regarding the performance of this venturi to permit design of a cascade of multiple venturi scrubbers and the project objectives interests would be better served by using a different type of contactor to provide the additional (2) absorption stages required. Two bubble cap trays will be provided for this purpose.

The regenerator is being designed with a short packed bed de-entrainment section above the top bubble cap tray. By lengthening this section and by providing a suitable salt inlet line (which can be blanked off when not in use) this packed section can be used as a packed column. To do so, the center section of bubble cap trays would be removed and the packed section would bolt directly to the bottom section. This will allow evaluation of a few packing configurations at a column diameter of 6 in. for comparison with the performance of the bubble cap trays.

(b) Tray Efficiency

A number of empirical correlations have been published for estimating tray efficiencies. Unfortunately these concern systems which are quite different from ours and can hardly be expected to apply. Theoretical relations were used to estimate tray efficiencies. The relations proposed by Geddes⁽⁴⁾ indicate a Murphree vapor efficiency of 95.7% in the regenerator and >99% in the absorber. Those proposed by Bakowski⁽⁵⁾ predict 78.5% for the regenerator and 98.82 for the absorber. In calculating the number of stages required, the more conservative estimates have been used. These high efficiencies are in agreement with our laboratory data taken in a bubbler contactor.

(c) Liquid Entrainment

In a bubble cap column, the gas stream may entrain a small amount of liquid carrying it up the column. This has the effect of reducing the efficiency of the column. The extent of entrainment was estimated using two methods given by Perry,⁽⁶⁾ Both are in essential agreement (eq. 18-19 and Fig. 18-19C) indicating that approximately 11% of the gross liquid flow through the absorber will be entrained and carried from the bottom stage to the top stage. This entrained liquid causes the effective vapor efficiency of the top stage to be reduced from 98.8% to 88%. Assuming the same amount of liquid entrained from the knock-out drum, the efficiency of the bottom plate will also be reduced to 88%. For the regenerator the entrainment is predicted to be less than 2% of gross liquid flow reducing effective gas phase tray efficiency from 78.5% to 77.4%.

(d) Determination Actual Number of Stages Required

A tray-by-tray calculation indicates that with a tray efficiency of 77.4%, four actual stages are required. Because of various uncertainties and extrapolations necessary in the calculations, it would be wise to introduce a considerable degree of conservatism in the design. In the case of the regenerator this has been accomplished by providing eight actual stages. In addition provision will be made for cooling the liquid phase on the top stage where maximum heat generation is occurring.

Equilibrium considerations led to prediction of 1.5 equilibrium stages needed for the absorber. This is in addition to the venturi. With a vapor efficiency of 88%, two actual stages are required.

(e) Column Design Details

The absorption column is a 10.75 in. OD cylinder (10 in. schedule-40 pipe) containing two bubble cap trays with four 2.875 in. OD caps per tray. The downcomer weir extends 3.25 in. above the tray and provides 2 in. of static liquid seal. Fifteen slots (0.25 in. wide x 0.75 in. high) are cut into each cap. The gas riser is 1.9 in. OD (1.5 in. Schedule-40 pipe) and the downcomer is 1.315 in. OD (1 in. schedule 40 pipe). Spacing between trays is 19.375 in. With outlet connections included, the column has an overall height of 76.31 in. It is to be fabricated in two sections using 10 in.,

150 lb flanges for connection of the two sections. A similar flange will connect the upper half section to the de-entrainer.

The regeneration column contains eight bubble cap trays contained in a 6.625 in. OD cylinder (6 in. schedule-40 pipe). Each tray contains two bubble caps. Nine slots (0.25 in. wide x 0.5 in. high) are cut into each cap. The gas riser is 1.315 in. OD (1 in. schedule-40 pipe) and the downcomer weir extends 1.4 in. above the plate.

The cap slots have been sized to provide gas velocities within the range recommended by Davies,⁽⁷⁾ e.g.:

$$\rho_g^{-0.5} \cdot 3.4 < V_{\text{slot}} < 12 \cdot \rho_g^{-0.5}, \quad (2)$$

where V_{slot} is the velocity of the gas coming through the slot, ft/sec, and ρ_g = gas density, lb/ft³. The limits for the two columns are:

Regenerator: $23 \text{ ft/sec} < V_{\text{slot}} < 81 \text{ ft/sec}$
 @ Design flow, $V_{\text{slot}} = 31 \text{ ft/sec}$

Absorber: $25 \text{ ft/sec} < V_{\text{slot}} < 90 \text{ ft/sec}$
 @ Design flow, $V_{\text{slot}} = 57 \text{ ft/sec}$

Schematic drawings of the absorption and of the regeneration columns are shown in Figures 4 and 5.

(f) Pressure Drop and Flooding

Pressure drop in the columns results mainly from contraction losses in the riser, losses due to flow reversal in the cap, resistance to flow in the cap slots, and the pressure due to the liquid seal above the slots. Methods for estimating these losses are given by Peters and Timmerhaus.⁽⁸⁾ Results of this estimate are given in Table II.

If the pressure imposed across a tray exceeds the height of the downcomer, the liquid will not flow down the downcomer. This condition is known as flooding. As evident from Table II, we are well below flooding in both columns.

(g) Salt Knock-out Drum (De-entrainer)

Referring to the flow diagram (Figure 2) the equipment item number 1 is the Salt Knock-out Drum or coarse de-entrainer. This unit serves a

FIGURE 4. Schematic of Absorption
Column

FIGURE 5. Schematic of Regeneration
Column

TABLE II. Pressure Drop in Regenerator and Absorber

A. Absorber, (Flow 75 SCFM, 700°C)

<u>Source</u>	<u>ΔP (psi)</u>	<u>ΔP (in. liq)</u>
Riser and Cap	0.0785 (Per Tray)	1.087 (Per Tray)
Slot Blow	0.0498	0.69
Liquid Seal	0.1 444	2
<u>Total</u>	<u>0.2727</u>	<u>3.777</u>

B. Regenerator, (Flow 8.85 SCFM, 630°C)

Riser and Cap	0.0382 (per Tray)	0.53 (Per Tray)
Slow Blow	0.0274	0.38
Liquid Seal	0.0361	0.50
<u>Total</u>	<u>0.102</u>	<u>1.41</u>

number of functions. It serves to de-entrain most of the salt from the high velocity gas stream. It is the high point in the system from which salt flows by gravity to the regenerator and subsequently to the absorber. It has been sized so it can contain sufficient salt to allow two hours operation of the regenerator at design rate. This will permit independent testing and shakedown of the salt cooler and regenerator.

Only a small fraction (10-20%) of the salt entering the knock-out drum will be fed to the regenerator. The balance will flow over a weir and pass down the recycle line to the venturi feed tank. Tars and oil droplets or particles of lower density than the salt phase will not reach the submerged outlet to the regenerator. These will track with the recycle salt flow and float on the liquid in the venturi feed tank. With the salt at 700°C oils and tars should undergo coking reactions to produce a char and low molecular weight hydrocarbons. The char will react at a low rate with water vapor and CO₂ but will probably be formed faster than it is destroyed. It will also go over the weir and end up in the venturi feed tank.

Material of higher density than the salt (>2.0) will tend to sink. The rate of flow over the weir is 85-90 percent of the total salt flow rate, so there will be little time for settling to occur. It seems probable that particles of higher density will tend to remain suspended and will slowly dissolve if subject to chemical attack.

(h) The Venturi Feed Tank

This tank receives incoming salt from the absorber and from the knock-out drum. It is also equipped with a weir to permit overflow of excess salt. By operating with a trickle of salt overflowing the weir, the venturi will operate at constant head. This salt overflow will comprise the bleed necessary to maintain salt quality. Once a steady state is attained (constant recycle rate and constant feed to the regenerator) the bleed rate can be controlled by adjustment of flow from the salt make-up tank to the absorber and thence to the venturi feed tank.

Any increase in flow from the salt make-up tank (over what is entering from the regenerator) will result in an increase in the volume of the circulating salt which can only be relieved by overflow at the weir in the

venturi feed tank. Obviously, fresh salt will have to be added at the salt make-up tank to compensate for this loss and maintain a desired level in this tank. There will be sufficient capacity in the salt make-up tank so salt additions can be intermittent; Salt bleed can also be intermittent.

(i) Flow Meters

Two siphon type flow meters will be installed, one at the base of each column. These, in fact, are an integral part of the column construction, in that the salt reservoir emptied by the siphon is an integral part of the column structure. A gamma ray densitometer will sense the filling and emptying of the salt reservoir caused by the siphon and the flow of salt. At the predicted flow rate for the absorber and regenerator the reservoirs will exhibit about six cycles/hr. The cycle rate will be calibrated in terms of salt flow rate. These meters will yield average flow rate but will not be responsive to rapid flow changes. It is anticipated that salt flow rates to these columns will be fairly constant.

In addition a commercial flowmeter made by Thermal Instruments, Inc. will be used to sense total flow to the venturi. This instrument will also require calibration.

(j) Salt Cooler

Regeneration of the extraction capacity of the salt by contact with steam + CO_2 in excess of equilibrium concentration is favored by reduced temperatures. Isothermal extraction-regeneration is possible, but at the expense of excessive steam consumption. In the present flowsheet, extraction is at (nominally) 700°C and regeneration is at 550°C . A salt cooler is desired which will cool the salt to a somewhat lower temperature than 550°C .

Design of this cooler has turned out to be more of a problem than was anticipated. Such heat transfer data as is available indicates that the heat transfer coefficient of the solid salt (in pure conduction) is about an order of magnitude higher than that of the liquid salt. This means that if a coolant such as air is used at a significantly lower temperature than the freezing point of the salt, complete solidification of the salt would be difficult to prevent. Pre-heating the coolant air sufficiently to avoid this

problem reduces the AT and increases the area of heat transfer surface required. With co-current flow of salt and air and with a jacketed 1.5 in. pipe through which 470 ml/min salt flow is established, preliminary calculations indicate 31 feet of pipe is required to cool this salt 200°C, (assuming a 450°C freezing point).

In part, this result arises from the fact that viscous flow is closely approached. It would be helpful if turbulent flow could be achieved, but this would require a very small diameter pipe.

The best alternative may be to pack the heat exchanger so as to obtain mixing. In effect this will promote turbulence and provide an alternative to purely conductive heat transfer. Attempts are being made to evaluate this procedure analytically.

IV. PROPOSED WORK DURING THE NEXT QUARTER

During the next quarter, design drafting should be completed, bid packages should be prepared and submitted to vendors, and all major equipment procurements initiated. Work on modifications to existing equipment can get under way.

V. OVERALL PROJECT STATUS AND CONCLUSIONS

During this quarter the Battelle Fused Salt Scrubbing Process has been subjected to exhaustive engineering analysis and optimized for operation in continuous mode. Engineering analyses are nearly complete and design drafting is well underway and should be completed in the next quarter. Detailed engineering analysis has proved somewhat more time consuming than originally envisioned and the project is some 4-6 weeks behind the planned schedule. It is hoped part of this lost time can be regained during the next quarter.

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