MNK-MPR-33



.

DEVELOPMENT OF KELLOGG COAL GASIFICATION PROCESS

Contract No. 14-01-0001-380

April 30, 1967

APPROVED :

Progress Peport No. 33

Project Manager

RESEARCH & DEVELOPMENT DEPARTMENT



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J. SUMMARY

This progress report is the thirty-third since the awarding of the contract. It is concerned with the first phase of the contract and summarizes the progress that has been made in the three principal areas now being studied: process research, chemical engineering studies and mechanical development.

The determination of the effect of sodium sulfate concentration in the melt on the combustion rates of anthracite and bituminous coke has been completed. The significant enhancement of the combustion rates (greater than four-fold) by the addition of 0.5 to 1.5 percent sulfate in the melt can be considered catalytic.

Runs were made to determine the effect of high steam conversions on gasification rates. Due to equipment limitations, these high conversions had to be attained using low steam feed pressures. The results indicate that at these conditions steam conversions up to 70 percent had little, if any, effect on the gasification rate of bituminous coke.

One new gasification run with graphite indicates that this feed has a low enough rate (≤ 12 lbC/hr/CF) to enable its use in the 5 3/4-inch diameter mechanical test reactor - with external heat supply - if pre-pilot bed depth studies are desired.

Additional ash removal studies have been made which indicate that:

- 1. Even if silica and alumina dissolve in the melt, treatment with bicarbonate solution will precipitate most of these materials (which will be removed with the rest of the ash.)
- 2. One atmosphere of carbon dioxide is insufficient to convert the dissolving sodium carbonate into bicarbonate at 210°F for pH control.
- 3. About 20 percent of the silica in the ash is present as soluble silica in the melt, and this amount will be carried through the ash filter and will precipitate with bicarbonate (vs. 35 percent assumed in the conceptual design.)



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4. Alumina as sodium aluminate dissolved in molten sodium carbonate at 1740°F raises the viscosity of the melt. At 30 percent aluminate, the melt is almost solid. However, indications are that even 1 atmosphere of carbon dioxide greatly reduced the solubility of alumina.

The process design of the proposed gasification pilot plant was completed for inclusion in the Summary Report and Proposal now being prepared.

Calculations were made to determine the economic effect of eliminating the power credit from the pipeline gas economics for bituminous coal. If this is done, a substantial amount of equipment associated with energy recovery can be eliminated resulting in a decrease in investment from \$140,000,000 to about \$112,000,000. The net effect, then, is an only slight increase in gas selling price from 50.3 to about 52¢/NSCF.

Cost estimation of the hvdrogen-from-bituminous coal plant has been completed. Estimated capital investment for this plant, capable of producing 250,000,000 SCFD of hvdrogen is about \$77,000,000. Gas manufacturing cost, calculated using the same procedure as for pipeline gas, is estimated to be about ???/MSCF for bituminous coal at \$4 per ton. There are no by-product credits taken in this cost.

A computer program has been completed for the sizing of melt transfer lines and flow predictions. The program has been utilized to size the melt transfer lines for the proposed pilot plant and to predict the performance of the gas-lift circulation systems. Based on these predictions an eight-inch diameter transfer line was selected for the pilot plant.

The cross flow contamination model was modified to allow the testing at downflow velocities in the vessels comparable to those specified in the conceptual design. The results indicate that at such velocities, product contamination may exceed specifications (< 5 percent inerts in pipeline gas), thereby necessitating the use of steam stripping of the melt leaving the combustor. On the other hand, if the gasifier and combustor can be operated with a larger temperature difference (e.g. 200°F instead of 70°F), downflow velocities would be substantially reduced and the contamination problem would be greatly alleviatec.

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Corrosion Test #10, a simulated gasification test designed to study the corrosion resistance of Monofrax A under compression, was completed. After approximately 550 hours, the specimens were found to have corroded at an average rate of about 1.5 inches per year - considerably higher than any previously determined rate at comparable conditions.

Since it was postulated that a possible cause of this result might have been an insufficient carbon dioxide pressure to inhibit the formation of Na₂O, a 140 hour run was made with a sodium carbonate-ash melt (but no sulfur) and one atmosphere of carbon dioxide. No measurable corrosion of the Monofrax A was detected.

After this run an exact duplicate of Test #10 was begun. After 140 hours, the Monofrax samples were removed and examined. A corrosion rate of about 0.2 inch per year was noted - in good agreement with previous corrosion tests made before Run 10. The samples were then returned to the melt and will be checked periodically as part of an attempt to determine the cause of the unusual result of Run 10.



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II. PROCESS RESEARCH

A. Accomplishments

- 1. Combustion Studies
 - a. Effect of Sodium Sulfate on Combustion Rate of Anthracite

The result of varying the concentration of sodium sulfate in the melt from 0.5 to 1.5 percent on the rate of combustion of anthracite was found to be very significant as is shown in Table 1 and the following tabulation:

Rate of Combustion - 1bs C/hr/cu.ft.

% Na_SO.	Temperatu	re - °F	At Average	Temperature	
in Melt	Initial Average			To 1740°F	
0.5	1746	1760	19.7	17.3	
1.0	1746	1808	32.2	21.0	
1.5	1749	1843	43.3	23.2	

The effectiveness of increasing the concentration of sodium sulfate not only is reflected in the average temperature of combustion during the 50% carbon consumed period, but is well documented by the increased rates of combustion.

In order to correlate the combustion rate for the average temperature shown to 1740°F, it was necessary to make a combustion run (Run 192) on anthracite at 1930°F. This run and an earlier run, H-160, gave the slope which allowed the rate to be corrected to 1740°F. THE M. W. KELLOGG COMPANY Research & Development Depertment



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TABLE 1

SUMMARY OF COMBUSTION RUNS IN MOLTEN SODIUM CARBONATE /1/ Run No. H -190 191 192 193 4/3 4/5 4/7 4/11 Date - 1967 - Anthracite-ゝ Feed % Total Carbon 80.0 % Vol. Matter 5.9 % Ash 11.7 Gms. Charge 21.35 Mesh Size 12/20 % C in Melt - Initial 4 Melt Gms. Na₂CO₃ Gms. Ash or Na₂SO₄ % Ash or Na₂SO₄ in Melt 409.9 405.7 407.8 411.9 2.07 /3/ 4.14 /3/ 8.3 6.21 /3/ 0.5 1.0 2.0 1.5 Height - inches 4 LL . 4 4 Conditions 1749 1930 Temp, °F - initial 1746 1746 - average /2/ 1760 1808 1929 1843 - maximum 1790 1828 1940 1874 44.7 44.9 44.7 44.7 Pressure - psia Sup. Gas Vel - ft/sec 0.99 1.01 1.07 1.00 30 45 Run Time - min. 35 10 26.0 26.1 26.8 26.4 Air Rate - liters/min Product Gas Analysis \$ CO₂ - 5 min. - 35 min. 5.2 9.0 3.9 12.5 1.4 1.6 -----1.4 0.7 2.2 - end 0.8 \$ 0₂ 15.2 11.5 8.0 17.0 - 5 min. - 35 min. 19.2 19.5 -19.2 19.6 18.8 - end 19.0 Results Combustion Rate Constant 1.67 2.73 1.22 3.67 Rate - lbs C/hr/cu.ft. at 4% carbon 19.7 32.2 14.3 43.3 **%** C Devolatilized 11.1 10.0 12.7 13.4 % C Combusted 89.9 88.3 84.0 83.6

/1/ Used 2 inch ID Inconel reactor. Feed charged in N₂ at 0.1 ft/sec SGV. and 5 min for devolatilization before air in. /2/ Average temperature in 50% carbon consumed period.

/3/ Na₂SO₄ instead of ash.



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Figure 1 shows the effect of sodium sulfate concentration on the rates of combustion of anthracite and bituminous coke. Although it appears that sodium sulfate above 1% concentration had a greater effect on coke than on anthracite combustion, such a conclusion is questionable because of the large temperature correction which must be made. Certainly, however, the catalytic effect of sulfate is well demonstrated.

Figure 2 shows the effect of temperature on the rates of combustion of anthracite and coke in the presence of 2% ash and in the presence of 0.5 to 2% sodium sulfate. Again this well illustrates the effect of sodium sulfate on combustion.

- 2. Gasification Studies
 - a. Effect of Steam Conversion on Rate of Gasification of Coke

Most of the process research data on gasification has been performed intentionally at low steam conversions i.e., less than 10% of the steam fed. Proposed commercial operation calls for about 70% steam conversion. Naturally, the effect of higher steam conversions on the rate of gasification is desired. However, the bench scale unit is very limited in this respect, mainly because of the low bed height allowed by furnace size and the fixed minimum velocity of about 0.5 ft/sec through the 2 inch diameter reactor. By diluting the steam at low pressure with nitrogen to give a total pressure of three atmospheres, runs up to 66% steam conversion have been obtained. These runs are shown in Table 2.

Plugging difficulties with runs 194 and 195 led to low carbon balances, consequently a procedure modification and testing at 1840°F to speed up the gasification led to runs 196 and 197. The average steam conversion during the 50% carbon consumed period is shown on the table. The results have been plotted on the standard correlation curve for steam pressure versus gasification rate in Figure 3. These results show remarkable agreement with the original line, certainly within the accuracy of the data. This indicates that gasification rates should not be significantly altered by steam conversions to 70%.



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	TABLE	2		
SUMMARY OF GASIFICATION	RUNS IN MO	- LTEN SODIUM	CARBONATE	: /1/
			_	
Run No. H Date - 1967	189 3/30	195 4/17	196 4/21	197 4 /25
Feed % Total Carbon % Vol. Matter % Ash Gms. Charge Mesh Size % C in Melt - initial	Graphite 96.1 3.9 17.26 60/100 4	~~~	Coke VJ 93.2 0.6 6.2 18.5 12/20 4	>
Melt Gms. Na2 ^{CO} 3 % Ash Bed Height - inches	414 0 4	414 0 4	414 0 4	414 0 4
Conditions Temp °F Pressure - psia % Steam in N ₂ Steam Pressure - psia Sup. Gas Velft/sec Run Time - min. cc H ₂ 0 in/hr cc N ₂ in/min	1740 44.7 91.5 40.9 1.03 70 1200 2312	1738 44.7 19.7 8.8 0.40 65 102 8.6	1840 45.0 16.4 7.4 0.50 65 101 10,691	1840 44.7 9.1 4.1 0.51 75 57 11,787
Results % C in Devol. Gas % C in Prod. Gas % C Loss	2.5 98.1 -	2.7 63.2 34.1	6.5 95.4 -	6.3 95.2
<pre>Gasif.Rate Const. k_I - input k₀ - output Rate - lbs C/hr/cu ft at 4% C, output</pre>	0.70 0.70 8.2	0.44 - 5.1	0.72 0.72 8.5	0.60 0.60 7.1
Avg. % Steam Converted -				

/1/ Used 2 inch ID Inconel reactor. Melt presteamed 15 min, flushed with N₂, coal added in N₂ for 5 min devolatilization, then steam started. Run H-194 not tabulated because plugging difficulties did not allow run to be completed.

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	FIGURE	3			
EFFECT OF STEAM	PRESSURE	ON GA	SIFICATION	RATE	
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			<u>CONDITIONS</u>		8
			A COAL © COKE		
			12/20 MESH CARBON INITI		
			NO ASH 4 INCH BED 0.5 FT/SEC SGV		Г 6, 8
			% STEAM CONVER	S1011	6 7
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b. Rate of Gasification of Graphite

Preliminary considerations on the use of the Mechanical Development Group's 5 3/4-inch diameter reactor, with a heat flux limitation of about 12 lbs C gasified/hr x cu. ft. melt, indicated that perhaps graphite may have to be the feedstock if the effect of melt height on gasification rate is to be studied. Run 189 in Table 2 shows a gasification run on an available graphite of 60/100 mesh size at the usual test conditions for feedstocks except no ash was present initially. As shown in the following tabulation, it is about 1/3 as reactive as anthracite of 12/20 mesh size with 2% ash in the melt. Thus graphite meets the rate requirement.

Feedstock	Mesh Size	Gasification Rate - lbs C/hr x cu. ft.
Graphite	60/100	8.2
Anthracite _	12/20	23.6

3. Melt Studies

a. Experiment on Melt Containing Solubilized Ash

A melt was prepared from sodium aluminate, sodium silicate and sodium carbonate which contained approximately 4% silica and 4% alumina. This mix was brought to 1740°F rapidly, then with N, bubbling through at 0.5 ft/sec for 10 minutes the melt was sampled. Nitrogen was replaced by carbon dioxide (an exothermic reaction was noted) and the melt agitated for 30 minutes before a second sample was taken.

Both samples were ground and treated with 9% aqueous sodium bicarbonate in the usual ash separation scheme. The precipitates representing the ash in coal were dried at 140°F and analyzed for Al₂O₃,SiO₂ and Na. It was noted that the volume of the precipitate from the N, test was about 2-3 times that of the one derived from the CO₂ test. This indicates that the melt from the N₂ test upon treatment with bicarbonate solution formed a gelatinous silica and alumina, whereas in the other test all or part of the silica and alumina were precipitated out by the CO₂ added to the melt and this precipitate was dense as expected.



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Analysis on both residues indicated 80% recovery of silica, 100% recovery of alumina, and 4% loss of sodium. The rest of the silica precipitated slowlv (analyzed after 1 month of standing) from the filtrate which had a pH of 9.8, the normal pH where silica starts to separate out from solution.

This experiment shows:

1. Even though some mineralization occurs in the melt, that is, some silica and alumina dissolves, most of the solubilized material will be precipitated by the 9% NaHCO₃ solution.

2. From the standpoint of ease of filtration and washing, it is preferred that no mineralization occur in the melt.

3. If the bicarbonate solution which is used to treat the melt can be maintained below 9.8 pH, preferably at about 9 pH, carryover of silica downstream will be minimal. The indication here is that 20% of the silica may be carried into the second residue (sodium bicarbonate) and perhaps not 35% as previously found in the early work.

b. Attempt to Prevent Silica Carryover in Ash Removal Step

A melt containing 8% anthracite ash was prepared at 1830°F with 1 atmosphere of CO₂ bubbling through the melt for 1 hour before pouring and freezing.

A 30 g. sample of the powdered melt was treated with boiling 9% NaHCO₃ solution while passing CO₂ through the slurry. The attempt here was to try to form NaHCO₃ from the carbonate being dissolved in order to keep the pH down and cause the silica to separate. It was found that the solution did not carbonate at all, in fact, literature indicates the solution will lose CO₂ by bicarbonate decomposition. The filtrate from this operation was then carbonated at room temperature yeilding residue number 2.

Analyses of residues 1 and 2 showed 77% of the silica in residue 1 and 23% in residue 2. This further confirmed the fact that 1 atmosphere of CO, present during solution of the melt is insufficient to completely prevent silica carryover. Notably only 3.0% of the Na was found in residue 1, thus 97.0% of the sodium was recovered.

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A second 30 g. sample treated with boiling bicarbonate solution for one hour but without CO, present gave the same weight of ash recovered indicating the solution did not dissolve any additional silica. This implies that the silica which carried over in both samples was originally present as water soluble silica in the melt.

c. Effect of Aluminate Content of Na, CO, Melt

The question of what happens when alumina dissolves in the melt has been raised. Chemistry indicates that

 $A1_{2}0_{3} + Na_{2}C0_{3} = 2NaA10_{2} + C0_{2}$

thus sodium aluminate forms and presumably dissolves in excess sodium carbonate. In order to answer the question of how this affects the melt, the measurement of viscosity was used with three concentrations of sodium aluminate at 1740°F. The results are given in the follow-ing tabulation.

Viscosity

% NaAlO ₂ in Melt	RPi1	Centipoises
	مين المارية	شين الي مارين إيران ه
10	5,10	400
	(5	1700
20	1 0	1200
	L 20	800
	0.5	~80,000
30 ·	∤ 1.0	70.000
	L 2.5	36,000

It is easily seen that increasing aluminate, especially at the 30% level, has a significant effect on increasing the viscosity of the melt. The 30% NaAlO, melt was almost solid.

By way of additional information, after the viscosity was determined on the 30% mix, nitrogen used for mixing was replaced with carbon dioxide for 1/2 hour. This caused the viscosity to change immediately, about 20,000 centipoises was obtained at 1 rpm and about 14,000 cp at 2.5 rpm. This was a significant drop which indicated CO₂ reversed the solubility and some alumina was precipitated out of the sodium carbonate.

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B. Projections

Additional melt studies employing freezing point to determine the solubility behavior of alumina and silica in molten sodium carbonate under various partial pressures of carbon dioxide will be made. Further investigations of sodium recovery from ash separations must be made. Disposition of sulfur in gasification and combustion will be investigated.

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III. CHEMICAL ENGINEERING STUDIES AND DEVELOPMENT

A. Accomplishments

1. Pilot Plant Design

The process design of the proposed gasification pilot plant was completed and an estimate of plant cost is currently being made. In addition, efforts have been made and will continue in order to prepare a Summary Report justifying Kellogg's position to proceed to this pilot plant stage and to prepare a Proposal for the cost and schedule for proceeding with such a program.

- 2. Flowsheet Studies
 - a. Pipeline Gas

Calculations have been made to determine the economic effect of eliminating the power credit from the pipeline gas economics. As was reported earlier (Progress Report No. 21) a power credit of 7.6¢/MSCF was taken for the bituminous case assuming this plant would operate as a total energy center. If, for some reason, however, power could not be credited, a rather substantial amount of equipment could be eliminated (heat exchangers, turbines, generators, etc.) For the case of bituminous, this corresponds to a reduction in capital investment from \$140,275,000 to about \$112,000,000. Using this figure, new economics have been calculated using the OCR-AGA standard accounting procedure. Total operating expense is estimated to be 45.1¢/MSCF (vs. 41.6¢/MSCF with power credit) and the 20-year average gas selling price is estimated to be 52¢/MSCF (vs. 50.3¢/MSCF.) Thus, insofar as gas selling price is concerned, there is little difference whether a power credit is taken for the excess energy generated in the process or not, but elimination of the power credit does decrease plant investment significantly.

b. Hvdrogen

Cost estimation of the hydrogen-from-bituminous coal plant has been completed. A conceptual flowsheet has been prepared but is not yet completed in its final form. Similarly, heat and material balances as well as utilities requirements have been calculated, although these will not be presented until the flowsheet is completed.



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Briefly, the process consists of a coal preparation section where the coal is received and ground to a size (-12 mesh) acceptable for good gasification and combustion rates. The coal then flows to a gasification section where it is reacted with steam in the presence of molten salt to produce synthesis gas. Ash brought in with the coal is continuously removed in an ash removal section by processing a melt slipstream withdrawn from the combustor. The raw synthesis gas undergoes a single stage of high temperature shift conversion and is then cooled down in preparation for CO, and sulfur removal. The gas purification scheme used is the Fluor Process for CO, and bulk sulfur removal, followed by sponge iron and activated carbon for H.S and organic sulfur removal, respectively. This clean gas is then reheated and reacted with additional steam over a nickel catalyst (reforming) to reduce the methane content of the product gas to about 4 percent. The effluent gas is cocled and undergoes a single stage of low temperature shift conversion, passes through a second Fluor CO, absorber, and finally through a catalytic methanation unit. This unit is designed to reduce the residual carbon oxides to a very low level. Effluent gas is cooled, compressed to 370 psia and leaves the plant at the rate of 250,000,000 SCFD. Gas specifications are given below.

Duantity, SCFD	250,000,000
Temperature, °F	100
Pressure, psig	350
Composition (mole percent):	
Γ H ₂	95.2
CH.	4.1
N_4	0.4
H ² O	0.3
cć + co,	10 ppm max.
S compounds	l ppm max.

The cost of producing 250,000,000 SCFD of hydrogen from bituminous coal according to the sequence just described is calculated in Tables 3 and 4, assuming 90 percent stream efficiency. Since no precedent has yet been set for the method to be used in calculating hydrogen costs, the procedure used here is in accordance with the standards used for estimating pipeline gas costs.

Estimated capital investment is summarized in Table 3. Shift, reforming, and methanation catalysts as well as activated carbon have been included in fixed investment because they have long lifetimes. Total capital investment is about \$77,000.000. THE M. W. KELLOGG COMPANY Research & Development Department



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TABLE 3

INVESTMENT SUMMARY

HYDROGEN FROM BITUMINOUS COAL

Basis: 250,000,000 SCFD of Hydrogen 90% Stream Efficiency

Bare Cost* Title Section \$ 3,384,500 Coal Storage and Preparation 100 21,640,500 Gasification 200 2,353,100 High Temperature Shift Conversion 300 12,505,000 Gas Purification 400 2,122,800 500 Methanation 3,153,600 600 Ash Removal Reforming and Low Temperature Shift Conversion 700 7,614,800 10,768,700 **Offsite** Facilities 1100 \$63,543,000 Total Bare Cost Interest During Construction and 11,183,600 Contractor's Overhead and Profit \$74,726,600 TOTAL FIXED INVESTMENT Working Capital 584,600 30 days Coal Inventory 66,000 30 days Carbonate Inventory 2,000 30 days Catalyst Inventory 28,100 Catalyst Charge Accounts Receivable at 11% of 2,024,000 Total Operating Expense

Total Working Capital 2,704,700

TOTAL CAPITAL INVESTMENT \$77,431,300

^{*} Bare Cost includes materials, freight, construction labor, field administration and supervision, insurance during construction, cost of tools, field office expense, and cost of home office engineering and procurement.

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Estimated operating expenses are shown in Table 4. Bituminous coal is charged at \$4 per ton. Total operating expense is calculated to be 22.4¢/MSCF.

B. Projections

1. Pilot-Plant Design

The Summarv Report and Proposal for the pilot plant program will be completed during Mav.

2. Flowsheet Studies

An attempt will be made to complete the final flowsheet for the hydrogen plant. When this is done, a complete "process package" will be prepared including material balances and utilities requirements. Efforts will also be made to resume work on the synthesis gas plant which has been temporarily suspended due to the press of preparing the Report and Proposal. THE M. W. KELLOGG COMPAN. Research & Development Department

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TABLE 4

ESTIMATED OPERATING EXPENSE

HYDROGEN FROM BITUMINOUS COAL

Basis: 250,000,000 SCFD of Hydrogen 90% Stream Efficiency

Item	\$/Year	¢/MSCF
Bituminous Coal at \$4 per ton	6,404,500	7.8
Sodium Carbonate make-up at 1.55¢ per pound	724,000	0.9
Miscellaneous Chemicals	122,100	0.2
Sponge Iron Make-Up	16,500	0.02
Direct Operating Labor at \$3.20 per man-hour	858,000	1.0
Maintenance at 3% of bare cost	1,905,000	2.3
Supplies at 15% of Maintenance	285,000	0.4
Supervision at 10% of Operating Labor	85,800	0.1
Payroll Overhead at 10% of Operating	08 000	r n
Labor & Supervision	34,400	0.1
General Overhead at 50% of Maintenance +	1.565.000	1.9
Supplies + Operating Labor + Supervision	1,000,000	
Plant Operating Expenses	12,061,300	14.7
Depreciation at 5% of Fixed Investment	3,740,000	4.5
Local Taxes and Insurance at 3% of Fixed		
Investment	2,240,000	2.7
Sub-Total	18,041,300	21.9
Contingencies	360,000	0.5
	18 01 300	22.4
TUTAL OPERALING EXPENSE	T0 340 T 3000	6 6 9 T



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IV. MECHANICAL DEVELOPMENT

A. Accomplishments

1. Mechanical Characteristics Testing

The analysis of deposits found in Test #3, conducted to determine entrainment in the aeration gas exiting a sodium carbonate melt bed, should be available by next report.

2. Melt Circulation

A computer program has been completed for the sizing of melt transfer lines and flow predictions. The program is based on the work previously reported in this area. Figure 4 shows the general configuration and defines the nomenclature used in the program. The program can handle additional bends in the horizontal transfer line to allow layout flexibility. Since the program handles each portion of the system individually and reports pressure drops for each, many other configurations are possible by the use of simple artifices.

The program has been utilized to size the melt transfer lines for the proposed pilot plant and to predict the performance of the gas-lift circulation systems. Figures 5 through 8 show the results for two configurations each for both the gasification (steam) and oxidation (air) gas lifts. Based on these predictions an eight-inch diameter transfer line was selected for the pilot plant.

The cross flow contamination model was modified slightly to allow the testing of cross flow contamination with somewhat higher downflow velocities in the vessels. Figure 9 shows the results of water tests conducted with bed aeration of 2 F.P.S. superficial velocity and gas lift superficial velocities ranging from 2 to 15 F.P.S. Maximum downflow velocities of 9.23 F.P.S. were tested with the aeration nozzle extended four and eight inches into the vessel from the bottom. Such a velocity corresponds approximately to that which will exist in the commercial plant (or pilot plant) with a 70°FAT between gasifier and combustor. If contaminations of the order found here do exist, it will probably be necessary to strip the melt leaving the combustor with steam as discussed in Progress Report No. 32.



All Dimensions in Ft. WL = 35. of Liquid Flowing (Lbs./Sec.) WG = " " Gas " " " RHOG, RHOL, RHOA = Density of Gas, Flowing Liquid, Vessel Liquid (Lbs./Cu. Ft.) VG, VL = Viscosity of Gas, Liquid (Lbs./Ft./Sec.) El, E3. E4 = Surface Roughness (Abs. Ft.) H1, H2, H3, H4, H5 = Pressure Drops (Ft. Liquid) KELLOGG

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On the other hand, if the gasifier and combustor could be operated with a greater temperature differential (e.g. 200°F) these downflow velocities would be correspondingly decreased thereby alleviating the contamination problem.

3. Corrosion Testing of High Temperature Materials

During this report period, Corrosion Test #10 was completed. This was a simulated gasification test which used a blend of gases fed to the test vessel instead of the graphite-steam feed previously used. The conditions were as follows:

G	as Feed	Melt	Com	pos	itio	n
^H 2 =	15.2 Mol %	^{ha} 2 ^{CO} 3	=	87	Wt.	¥
^{CO} 2 =	10 "	Coal As	:h =	10	h	
CO =	4 . 7 "	. Na ₂ S	=	3	**	
^N 2 =	41.7 "	Tota	11	100		
$H_20 =$	28,4 "	· ·				
Total	100.0					

Conditions:

Pressure	=	14.7 psia
Duration	=	549 hours
Temperature	=	1840°F
Superficial Gas		
Velocity	=	1.0 ft./sec.
-		

The specimens included in this test were two cubes of Monofrax "A", each approximately one inch on a side. These were clamped together under a load of 40 psi to simulate a joint in the lining of the proposed reactor. At the conclusion of the test, the blocks were found to have corroded at an average rate of 1.5 in./year with a maximum of 2.4 in./year on one edge of one specimen. These corrosion rates were much higher than any previously determined for this material and further testing was begun immediately to determine the cause.



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Test #11; in an attempt to isolate the cause of the high corrosion rates found in Test #10, was run with 100% CO₂ as the feed and no Na₂S in the melt. All other conditions remained the same as for Test #10. After 138 hours of exposure to the Test #11 environment, the Monofrax "A" was examined and found as sound as when placed in the reactor, with no measurable corrosion. Five explanations of these results of Test #10 and #11 are possible:

- The CO₂ partial pressure in Run #10 was not great enough to inhibit the decomposition of Na₂CO₃ to Na₂O, which is quite capable of autacking Monofrax "A".
- 2. The ratio of CO₂ to H₂O was not high enough to suppress the formation of NaOH.
- The presence of Na₂S instead of the usual Na₂SO₄ caused the high corrosion rate in Run #10.
- 4. Some odd coincidence caused the high corrosion found in Run #10.
- 5. Some combination of the above.

In order to investigate further the unusual results of Run #10, an exact duplicate run was made but for a shorter duration. This run, #12, included two blocks of Monofrax "A" under stress with all conditions as noted for Run #10. In addition, an unstressed sample of Monofrax "A" was included as a control specimen. After a test duration of 140 hours the samples were examined with the following results: The average corrosion rate of the samples was found to be .19 in/yr with a maximum on one face of 0.23 in/hr. These rates are in keeping with those previously determined for Monofrax "A".

As this run, #12, is a duplicate of #10, and its corrosion rates are much lower, on the order of those previously determined for Monofrax"A", the conclusion must be drawn that something unusual occurred during Run #10. What this may have been remains to be determined in further testing of the Monofrax "A".

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B. Projections

1. Mechanical Characteristics Testing

Further work is contemplated to define more thoroughly the carry-over, both entrained and gaseous, as a function of superficial velocity through the bed.

2. Melt Circulation

Not much more work can be accomplished in this area until the actual melt can be circulated. The design and cost of a test facility to achieve this end is currently being investigated.

3. Corrosion Testing of High Temperature Materials

Run #12 will be continued on towards 500 hours to investigate the corrosion rate after lengthy exposure. Based on these results, further testing may be indicated to help determine the cause of the results obtained in Run #10.