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DEPT OF THE INTERIOR

DEVELOPMENT OF KELLOGG COAL GASIFICATION PROCESS

Contract No. 14-01-0001-380

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Progress Report No. 15

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A DIVISION OF PULLMAN INCORPORATED



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RESEARCH & DEVELOPMENT DEPARTMENT

REPORT NO. 15

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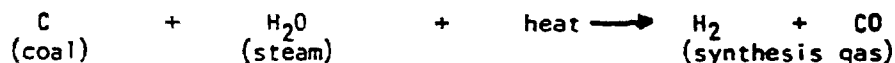
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I. INTRODUCTION

The objective of this contract with the Office of Coal Research is to develop the Kellogg Gasification Process to the point where it will be able, on a commercial scale, to convert coal into pipeline gas at a cost of 50¢/MSCF or hydrogen at 25¢/MSCF. Five raw materials are to be studied -- an anthracite, a high-volatile bituminous coal, a sub-bituminous coal, a lignite, and a char. Although Kellogg's experimental work will not extend beyond the production of raw synthesis gas, the overall project must make engineering evaluations for four ultimate end products -- pipeline gas, hydrogen, synthesis gas, and transport gas.

Basis for the Kellogg Gasification Process is the reaction between steam and fine coal in a molten salt bath to form synthesis gas, a mixture of hydrogen and carbon monoxide, according to the reaction:



The necessary heat of reaction is supplied by circulating a heated molten salt stream. In addition, the molten salt mixture is chosen to catalyze the gasification reaction so that it may be carried out at a relatively low temperature.

The program is divided into three phases of study extending over a five-year period. Phase I, which is now in progress, involves several concurrent efforts:

1. Bench-scale process research -- to investigate melt properties, reaction kinetics, and the effect of process variables.
2. Chemical engineering studies and development -- to determine the optimum process flowsheet and operating conditions and to coordinate experimental work with overall project objectives.
3. Mechanical development -- to find acceptable materials of construction and develop techniques for handling the molten salt and powdered coal.



Phase I will be concluded by the design of a pilot plant to gasify 24 tons of coal per day, if it is found that a pilot plant program is justified by the bench-scale experimentation and economic studies.

Phase II will be devoted largely to the construction and operation of a pilot plant to convert a variety of raw materials into raw synthesis gas. The effect of operating variables found to be significant in Phase I will be investigated to obtain data for design of a commercial plant.

Phase III will involve the detailed process design of a commercial plant to produce 250 million standard cubic feet a day of product gas, including cost estimates and projected economics for those areas of the country that appear to offer commercial possibilities.



II. SUMMARY

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

In a commercial plant employing molten salt gasification, it is proposed to burn coal in direct contact with the melt (molten salt plus coal ash) to provide the heat necessary for gasification. It is important to the economics that this combustion produce CO_2 , not CO , even though it takes place in the presence of excess carbon, and that the heat of combustion be liberated in the melt and not in the gas space above it. A combustion experiment was carried out using a long probe to sample the combustion gas just above the melt surface. It was found that at the beginning of the run, with 5% carbon in the melt, about 75% of the carbon burned was producing CO_2 in the melt, as desired. By the time the carbon concentration had dropped to 3.5%, no more CO was being produced. These results are quite encouraging. The economic effect of the worst condition observed, i.e. 1/4 of the carbon producing CO , will be evaluated.

A second combustion experiment in which silica was added to the molten salt yielded the same combustion rate as for pure sodium carbonate, indicating that the apparent catalytic effect of coal ash cannot be attributed to its silica content.

A question was raised some time ago concerning the true reaction temperature for gasification experiments. Calculation of water-gas shift equilibrium constants for a number of runs indicated that the gasifying medium (steam) may fall short by several hundred degrees Fahrenheit of achieving the melt temperature, which is reported as the temperature of the experiment. The true reaction temperature might then be open to question. This problem has been resolved by modifying the experimental equipment to preheat the feed steam to 1500 - 1600°F before it enters the melt. Gasification rates were not affected by such preheat. It was concluded that the measured melt temperature is, within the experimental limits imposed on temperature measurement, the true reaction temperature.



The effect of bed height on rate of gasification was studied by varying the quiescent melt depth from 4 to 8 to 12 inches while maintaining the same quantity (but not concentration) of carbon in the melt. The specific rate of gasification (pounds gasified per hour per pound present) did not change, indicating no deterioration of rate in deeper beds due to poorer gas-melt contacting. On the other hand, when bed height was increased while holding carbon concentration constant, the specific rate decreased by about 20% although the quantity of carbon gasified was larger. The cause of the decline is not known, whether it be poorer contacting or the effect of lower steam partial pressure due to higher steam consumption. Further study is planned.

The effect of changing feed steam partial pressure from 0.9 to 0.3 atmosphere at 1840°F in an 8-inch bed appears to be smaller than that at 1750°F in a 4-inch bed. This will also bear further investigation.

Plans are in progress for modifying the present Inconel reactor system so that gasification and combustion experiments can be carried out at pressures up to 60 psia. Consideration will be given to a study of the effect of gas composition on gasification kinetics.

The recovery of sodium values from melts prepared at 1700 and 1900°F containing 5 - 10% ash and 0 - 10% bituminous coke was studied. After dissolution of sodium carbonate, the residue of coal and coke was washed with sodium bicarbonate solution and water in ratios simulating commercial operation. Sodium losses in the washed residue, which would be discarded in commercial operation, were much higher than in earlier experiments in which much larger quantities of wash water were used. One of the earlier experiments was then repeated, yielding excellent reproducibility. It was concluded that the manner of washing and quantity of wash liquor used are critical factors that cannot be adequately demonstrated on the laboratory scale. Because the loss of sodium is a vital economic factor, further studies of this problem on a larger scale will be considered.

The process design of a plant to produce 250 million standard cubic feet per day of 900-Btu gas from sub-bituminous coal was substantially completed. Insofar as possible the design is based on recent experimental data. Estimated total investment for the process plant (not including offsite facilities) is about 120 million dollars. Gas production cost, not including any return on investment, from \$2-per ton coal (8¢/MM Btu) is about 40¢/MSCF. The economic effect of changing



several operating parameters was investigated and will be continued next month. A similar design and economic study based on bituminous coal will be continued.

Corrosion tests in crucibles and in the corrosion test unit have shown that zirconia 8-D, the 25%-porosity commercial form, is not a satisfactory material of construction in contact with the melt under gasifier conditions. Nor is a 95% alumina refractory. Samples of high-density, fused 99% alumina from several manufacturers have shown good resistance, however, even in such a severe environment as fused NaOH at 1400° F. Further tests of this material are planned.

Materials considered for use as electrodes in the proposed high-pressure reactor were tested in the corrosion test unit under actual gasification conditions. RA600 appeared to be satisfactory after 48 hours at 1800° F and a current density of 40 amps/in.² of electrode.

The corrosion test reactor vessel failed after 1200 hours of service at atmospheric pressure and is being replaced.



III. PROCESS RESEARCH

A. Accomplishments

1. Combustion Studies

In one combustion run (J-9845), the addition of 4% sodium silicate to the melt was made to determine if silica in ash was responsible for the enhanced combustion rates when ash was present in the melt. The combustion rate constant for this run was 0.37, in good agreement with the standard run constant of 0.43, indicating no positive effect on combustion.

In the second combustion run (J-9851), a probe sampler was employed to obtain samples of gas just above the melt surface in order to establish whether carbon was burning to carbon monoxide or to carbon dioxide in the melt. In previous runs only carbon dioxide appeared in the exit gas from the unit when sufficient combustion air was present. The results of the run are given in the following tabulation.



Time min.	Operation	% in Probe Product			Ratio CO ₂ /CO	Approximate % Carbon Left in Melt
		CO ₂	CO	O ₂		
-5	1st Coke charge					
0	Air in	-	-	-	-	100
5		6.5	2.0	15.0	3.3	95
15		5.0	1.0	14.5	5.0	82
25		5.5	tr	15.0	55 ⁺	70
40	Air out,					
	N ₂ only	-	-	-	-	52
142	N ₂ @ 0.05 FPS	0.5	4.5	0	-	-
152	N ₂ @ 0.25 FPS	0.5	3.0	0	-	36
155	2nd Coke charge	-	-	-	-	135
160 (C)	Air in	-	-	-	-	135
6		13.0	7.0	5.5	1.9	126
23		12.5	2.0	7.0	6.3	97
32		11.5	0.5	7.5	23	83
44		11.5	tr	7.0	115 ⁺	65



The probe worked well and showed that carbon monoxide was being evolved from the melt but that it decreased to a trace in time while the carbon dioxide stayed at a high level. Two coke charges were made which behaved essentially the same except for the change in level of the gases and the time to reach a trace of carbon monoxide. This was probably due to the higher carbon concentration after the second coke addition. It does appear that when the carbon concentration reached about 3.5% in the melt (70% of 5% initially in the melt) the production of carbon monoxide ceased. Also noted is the reaction of carbon with sodium carbonate to produce carbon monoxide when only nitrogen is bled through the melt. Some of the carbon monoxide during combustion may be produced by this reaction. These results lead to the postulation that under steady state conditions the major combustion product will be carbon dioxide and only a minor amount of carbon will go to carbon monoxide.

2. Gasification Studies

The data on eight gasification runs are presented in Table I. Essentially the effect upon gasification rate of feed steam preheat, bed height, and steam concentration were studied.

The unit was modified as follows: steam as prepared in the generator was passed into a 20-ft. long $\frac{1}{4}$ " O.D. tubing coil placed in a 3" I.D. by 12"-long furnace, then into the reactor through a transfer line which was wound with resistance wire to maintain temperature. A thermocouple was placed into the preheated steam-nitrogen gas stream just at the entrance of the reactor. Another safety couple measured the skin temperature of the transfer tubing.

The results of using maximum preheat, giving a steam-nitrogen temperature of about 1500 to 1600°F going into the inlet tubing in the reactor, are shown in runs 9843, 9844 and 9846. The specific gasification rate constants are compared in the following tabulation with those of earlier runs without preheat. The results show that no major effect of preheat, as applied, appears evident from the data at 1730 and 1840°F.



Table I
Gasification of Bituminous Coal in Molten Na₂CO₃ /1/

Run No. J-	9843	9844	9846	9847	9848	9849	9850	9852
Date of Run-1965	9/30	10/5	10/11	10/13	10/15	10/19	10/21	10/27
Feed -----Bituminous Coal-----								
% Total Carbon	81.3							
% Volatiles	37.3							
% Ash	3.9							
gms. charged	20				40	20	20	20
mesh size	12/20							
gms. Na ₂ CO ₃	414		828		828	828	1242	828
% C in melt	3.8		1.9		3.8	1.9	1.3	1.9
Bed Ht.-inches	4		8		8	8	12	8
Conditions								
Temp. °F.	1730	1840	1640	1840	1840	(1840)	1876	1840
% steam in N ₂	90				30		90	30
Preheater Temp. °F	1520	1580	1560	780	670	730	660	750
Min. to 0% CO	85	55	175	55	85	95	55	85
Total Run-min.	85	65	175	65	85	95	55	85
Results								
% C to CO + CO ₂	80.2	86.6	129.8	108.3	96.9	109.9	112.5	111.6
% C to C ₁ -C ₃	8.0	4.8	14.8	3.1	4.8	12.5	14.6	4.8
% C to tar, etc.	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5
Total % C	94	97	150	117	107	128	133	122
Spec. Gasif. Rate Constant								
Basis input	0.67	1.13	0.67	1.29	1.00	1.13	1.80	0.95
Rate								
lbs. C gasif./hr/CF								
at 10% C in bed	21	35	22	40	31	35	56	30
at 5% C in bed	10	17	10	19	15	17	27	14
Salt carryover-gms.								
	8.2	13.0	19.4	11.4	12.0	9.0	10.4	4.7

/1/ Atm. Pres., 0.5 ft/sec. superficial gas velocity.



Gasification Rate Constant

<u>Temp. °F.</u>	<u>Without Preheat</u>	<u>With Preheat</u>
1640	0.2 /1/	0.67
1730	0.55	0.67
1840	1.29	1.13

/1/ Estimated

It is indicated that at 1640°F preheat may show an advantage, however the very high carbon balance on this run complicates the result. The high carbon balance is the result of an abnormal release of carbon dioxide from the melt.

Calculations based upon the water gas shift reaction and comparison to known equilibrium data were used to attempt an insight into the actual gas phase temperature. The water gas shift reaction is:



The results of direct comparisons of preheat versus non-preheat runs are shown in Figure 1. At 1840°F the results indicate the approach to equilibrium to be about the same. However, at 1740°F, the preheated inlet gas run shows a definite advantage in approaching water gas reaction equilibrium. It is indicated therefore, that preheating of the steam may have a beneficial effect at lower salt temperatures in this system. Based upon this work and the use of higher temperatures in our studies, further runs were made dropping the preheat temperature to about 700°F in order to ease the freezing of fittings and difficulties associated with high temperatures in transfer lines.



EFFECT OF PREHEAT ON THE WATER-GAS SHIFT REACTION DURING COAL GASIFICATION

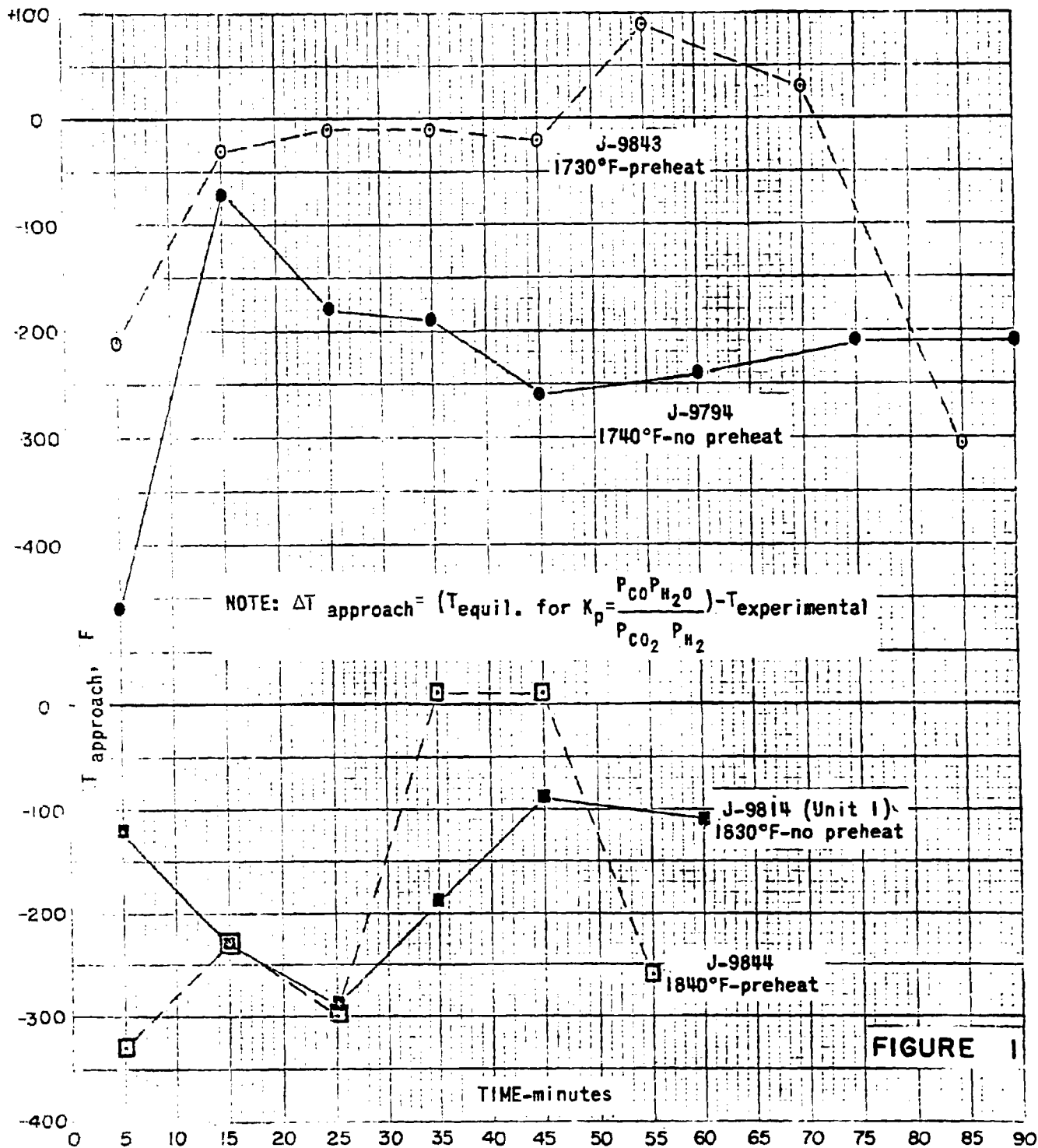


FIGURE 1



The next series of runs studied the effect of bed height on the gasification rate constant. Since some of the earlier simulated gas-liquid contacting experiments indicated formation of large bubbles a few inches above the gas inlet, in a 3-inch tube, the bed height was pushed from 4 to 8 to 12 inches in the 2-inch reactor with good success in operation. Although the run at highest bed height was at a higher temperature due to a faulty thermocouple which was discovered in time, a correlation of temperature and rate constant allowed comparison of this run with the others. As shown in the following tabulation, the rate constant was constant for the three bed heights indicating no adverse effects of gas-melt contacting on the rate up to the 12-inch level. A temperature profile indicated an actual expanded bed height of 15 to 16 inches for a bed which is only 12" deep when quiescent.

<u>Molten Salt Bed Height-inches</u>	<u>Gasif. Rate Constant k</u>
4	1.29
8	1.29
12	1.24

In run J-9848, the initial carbon concentration in the 8-inch molten salt bed was made equal to the normal concentration in a 4-inch bed, namely 3.8%. The reaction rate constant for this run was only 78% of the standard, indicating an effect due to the higher steam consumption, which gave a lower concentration of steam. This matter will be investigated further.

With an 8-inch bed, steam pressure was lowered to 0.3 atmosphere in run 9849. The specific gasification rate constant of 1.13 appeared to be high based on prior data and since in the next run, the thermocouple was found to be reading low, the true temperature of this run became suspect. The run was then repeated with new thermocouples in J-9852, and the rate constant for 0.3 atm. steam at 1840°F in an 8-inch bed was 0.95, in reasonable agreement with the 1.13. Further work is contemplated for the effect of steam pressure on the rate. It does appear that changing steam pressure from 0.3 to 0.9 atmosphere at 1840°F in an 8-inch bed does not have the effect it did at a lower temperature and bed height.



3. Viscosity Measurements

At the request of Chemical Engineering, viscosities of melts containing 4 and 8% bituminous coal ash with and without bituminous coke present at 1700 and 1900°F were estimated from the data presented in Progress Report 13 (August 31, 1965) and the literature values for pure Na_2CO_3 ⁽¹⁾. The viscosities for this non-Newtonian system apply to shear rates equivalent to 10 to 50 RPM on the Brookfield viscometer and are to be considered as approximate only. The data are presented in the following table. These data are considered to be sufficiently reliable for use in preliminary design studies.

<u>% Ash</u>	<u>% Coke</u>	<u>Viscosity in cp at</u>	
		<u>1700°F</u>	<u>1900°F</u>
4	-	11-19	5-6
8	-	44-100	21-30
4	5	60-140	30-44
8	5	190-415	100-155

4. Sodium Carbonate Recovery

Additional sodium recovery tests were run on melts containing coal ash and carbon using commercial design quantities of wash solution. The melts were prepared by mixing molten sodium carbonate with bituminous coal ash using carbon dioxide gas for about six hours, then nitrogen replaced the CO_2 and bituminous coke was added in small amounts as rapidly as the melt would allow without frothing over. This took about 30 minutes. This melt was solidified and ground to give 12/20 mesh material for the wash tests. First the solid was treated with 9% NaHCO_3 solution in the normal manner (6/1 liquid to solid ratio at the boiling point for 10 minutes) and filtered. The residue was washed with 3/1 hot water to solid ratio in one case and in a second case it was treated with a similar ratio of 9% NaHCO_3 solution followed by

(1) Molten Salt Data by G. J. Janz, A. T. Ward, and R. D. Reeves, Rensselaer Poly. Inst., 1964.



hot water. This residue 1 solid was dried, weighed, and analyzed for sodium. Thus the percent of sodium lost in residue 1, a product to be discarded in the commercial scheme, can be calculated. The conditions of interest and the results are given in the following table.

Melt Temp. °F	Matter % Ash	Added % Coke	Wash Conditions	% Sodium Lost in Residue 1
1700	10	5	H ₂ O	8.16
1700	10	5	NaHCO ₃ and H ₂ O	6.55
1900	10	5	H ₂ O	19.2
1900	10	5	NaHCO ₃ and H ₂ O	19.2
1700	10	10	H ₂ O	7.47
1700	10	10	NaHCO ₃ and H ₂ O	5.02
1700	5	-	H ₂ O	2.43
1700	5	10	H ₂ O	3.12

A number of tentative conclusions can be drawn from these data. The amount of sodium retention in residue 1 was most severe with the higher temperature melt. The double wash was better than the single wash at the lower temperature only. Sodium recovery was better from a lower ash content melt with or without coke present. The effect of 5 versus 10% coke in a melt containing 10% ash was slightly better on sodium loss at the higher coke level, an unexpected result which may be within the range of experimental error.

All the results at 10% ash gave less than 95% sodium recovery, which is probably uneconomic. To determine whether better sodium recovery could be obtained, two additional runs were made. First, an x-ray pattern on residue 1 from the high-temperature melt showed Na₂CO₃·H₂O to be present. This material should have dissolved in the wash, thus indicating the wash treatment was insufficient. It was reasoned that the addition of coke using nitrogen may have allowed ash-sodium carbonate reactions to occur to a greater extent than if carbon dioxide was present to depress this reaction.

The 1900° F runs were repeated using carbon dioxide throughout the melt-ash-coke mixing period. Washing residue 1 with hot water at 3/1 ratio resulted in 8.95% sodium loss, while the double NaHCO₃-H₂O wash each at 3/1 ratio gave 8.35% sodium loss. Neither one of these residues



were considered to be washed properly. The favorable presence of carbon dioxide during mixing is well demonstrated. However, the still high sodium retention by the residue was believed to be due to the poor washing restriction. Residue 1 showing 8.95% sodium loss was washed with a large amount of hot water (200 ml. of H₂O for about 4 grams of residue). Weight loss was assumed to be due to sodium carbonate only. This indicated the sodium loss to be only 3.2% and demonstrated that suitable sodium recoveries are possible providing washing is properly done. It is believed that under the restricted Laboratory conditions and the small-scale, commercial-type washing cannot be simulated or achieved, thus poor sodium recoveries will be inherently obtained as shown above. Large-scale work on sodium recovery is indicated.

5. Corrosion Tests

Additional small static corrosion tests were made on coupons of zirconia Y-1027, the dense non-porous form, and on 8-D, the commercial 25%-porosity form. Zirconia Y-1027 corroded at a rate of 0.09 IPY in an 1800° F sodium carbonate melt, with a CO₂ atmosphere, for 95 hours. Silicon nitride, which was also present, dissolved. The same zirconia Y-1027 corroded at a rate of 0.3 IPY in an 1800° F melt of sodium silicate with a minor amount of sodium carbonate and in a CO₂ atmosphere for 26 hours. These and the previous tests quoted in last month's summary led to the conclusion that alkali attacked and dissolved the zirconia in Test 4 reported by Mechanical Development. Gross amounts of alkali appeared to be liberated by reaction of the melt with some of the other substances present such as silicon nitride, molybdenum, and tungsten.

Coupons of the porous zirconia 8-D exhibited much less resistance to corrosion than Y-1027. A sample of 8-D in 95% Na₂CO₃-5% NaOH melt for 17 hours at 1800° F corroded at a rate of 0.7 IPY and appeared to be soft and friable. A similar sample completely disintegrated in a sodium hydroxide melt at 1400° F in 17 hours. These results indicate that 8-D zirconia should not be used in contact with the molten salt bath in the design of the high-pressure gasifier.

Two alumina coupons were evaluated for resistance against sodium hydroxide, the most reactive constituent in the molten salt. Monofrax M, a 95% alumina ceramic material, was badly corroded by sodium hydroxide in 16 hours at 1400° F. Only 36% by weight of the coupon remained, the rest disintegrated through swelling and decrepitation during washing.



This indicates that this material will not be satisfactory for reactor service. The second ceramic, Coors fused 99% alumina, showed remarkable resistance, only 0.53 IPY corrosion rate in molten sodium hydroxide in 17 hours at 1400 F. This material remained structurally hard and appears to be a good choice for reactor service.

A magnesia crucible material was evaluated for resistance to alkali and sodium carbonate in overnight tests at 1400°F and 1800°F, respectively. In sodium carbonate the magnesia ceramic withstood corrosion satisfactorily, but in sodium hydroxide it appeared that the binder material was dissolved and a weak, crumbly structure was left. From this work, it is believed that magnesia is also a possibility for reactor service but the magnesia should not be bound by siliceous matter.

B. Projections

Present plans call for utilizing the present Inconel reactor system with proper safety and pressure modifications to its maximum pressure of 3 atmospheres gage (60 psia). Further combustion studies will be made to determine heat transfer relationships through temperature profiles automatically recorded. Consideration will be given to a study of the effect of recycle gas on the kinetics of gasification.



IV. CHEMICAL ENGINEERING STUDIES AND DEVELOPMENT

A. Accomplishments

Preparation of the process flowsheet for pipeline gas from sub-bituminous coal, utilizing recent experimental data, has been substantially completed. The finding reported last month, that so much excess steam is available from the plant as to influence the choice of optimum gas purification process, has turned out to be erroneous. The Fluor Solvent CO₂ Removal Process is still the most economical, as concluded in Progress Report No. 11⁽²⁾. Excess steam will be available, but not enough to dictate selection of the Shell Sulfinol Process, which can compete with Fluor in this application only if steam is free.

A preliminary estimate of investment for a plant producing 250 MM SCFD of 900 - Btu pipeline gas from sub-bituminous coal is presented in Table II. Estimated gas production cost is presented in Table III for the following basis:

1. melt temperature in gasifier = 1830°F
2. melt temperature in combustor = 1900°F
3. 4% coal in melt
4. 8% ash in melt
5. 95% carbon utilization
6. 2.4% Na₂CO₃ loss per pass through ash removal system
7. volumetric rate of gasification and combustion = 15 lb/hr ft.³
(k = 1.6)
8. steam/carbon ratio in gasifier = 2 lb/lb

(2) Progress Report No. 11, June 30, 1965, p. 5.



TABLE 11

ESTIMATED CAPITAL INVESTMENT

PIPELINE GAS FROM SUB-BITUMINOUS COAL

Basis: 250 MM SCFD of 900-BTU Product Gas

<u>Section</u>	<u>Description</u>	<u>Investment, millions of dollars</u>
100	Coal Storage and Preparation	13
200	Gasification	58
300	Water-Gas Shift	7
400	Gas Purification	22
500	Methanation	11
600	Ash Removal	3
	Interest during Construction	<u>5</u>
	Total Fixed Investment	119
	Working Capital	<u>5</u>
	Total Capital Investment	124



TABLE III

ESTIMATED GAS PRODUCTION COSTS

PIPELINE GAS FROM SUB-BITUMINOUS COAL

Basis: 250 MM SCFD of 900-BTU Product Gas
90% stream factor
Higher heating value of coal = 12,720 BTU/Lb.

<u>Item</u>	<u>¢/MSCF of Product Gas</u>
Coal at \$2/ton	13.6
Utilities:	
Boiler feed water	2.7
Cooling water & process water	1.6
Catalysts & Chemicals:	
Na ₂ CO ₃ at 1.55¢/lb.	1.7
Methanation catalyst	1.4
Gas purification chemicals	0.4
Operating labor at \$3.20/man-hour	1.6
Overhead at 80% of operating labor	1.3
Fixed charges at 11.15% of fixed investment	<u>16.2</u>
Total Gas Production Cost	40.5



9. combustion directly to CO_2 in combustor melt
10. flue gas leaves combustor at 2200°F

The method of calculation is that suggested by OCR⁽³⁾, but in abbreviated form for these preliminary figures.

Actually such a plant would be completely self-sufficient, having facilities for providing steam, power, cooling water, boiler feedwater, fire protection, etc. The cost of such facilities has not yet been estimated and would, of course, increase total plant investment. On the other hand, the unit charges made for utilities in calculating production cost would no longer be necessary; this would tend to balance the increased investment.

Several alternatives to the above base case have been studied so far:

1. If ash concentration in melt is reduced from 8 to 4%, investment and utilities for the ash removal system increase and more coal is lost from the system, resulting in a 1.3¢/MSCF increase in gas production cost.
2. If rates of combustion and gasification are found to increase with the square root of total pressure (no experimental data yet available at high pressure), gas production cost should be 1.5¢/MSCF lower.
3. If steam/carbon ratio in the gasifier is 3 lb/lb instead of 2, gas production cost will increase by 4.5¢/MSCF.

B. Projections

The process flowsheet for pipeline gas from sub-bituminous coal will be completed. A more-detailed estimate of capital costs for this case will be prepared. The effect of several other process assumptions and operating parameters will be investigated.

Calculations for a similar case based on bituminous coal will be continued.

(3) Office of Coal Research, "Tentative Standard for Cost Estimating of Investor-Owned Plants for Producing Pipeline Gas from Coal," June 4, 1965.



V. MECHANICAL DEVELOPMENT

A. Accomplishments

I. Environmental Testing of High-Temperature Materials

Further evaluation of the Monofrax "A" samples from Corrosion Test #4 indicates little corrosion. These samples seem as strong as before the test and show only a discoloration to differentiate the tested samples from the "as received".

During this report period, a program was undertaken to determine a material to be used as a heating electrode in the proposed high-pressure reactor. Test #5 was conducted using a 3/16"-diameter carbon electrode placed in the melt and carrying a current of 40 amps/in.² of electrode submerged. This electrode suffered an almost immediate mechanical failure at a point where the carbon was joined to a supporting metal rod.

Test #6 was begun immediately, using a 1/4"-diameter rod of RA 600 as an electrode. This again carried 40 amp/in.² of submerged electrode area. In addition, three samples of zirconia 8-D were placed in the reactor for Test #6. After approximately 48 hours at temperature, the electrode and samples were removed and examined. The diameter of the electrode had not changed, and there was no indication of appreciable corrosion along its length. It had, however, shortened in overall length, due probably to arcing at the liquid interface as the surface of the melt fell below the electrode. (Due to buildup of splashed melt on the upper walls of the reaction, the level of the melt slowly falls. This partially solid deposit must periodically be scraped back into the melt to restore its proper level.)

Of the zirconia 8-D, only the sample that had been in the gas space remained; the others had completely disappeared. This one remaining sample was badly corroded.

Shortly after the completion of Test #6, while the reactor was being cleaned and recharged with fresh melt, several fairly large holes opened in the sides of the reactor above the melt. This damage is such that it is not feasible to repair the reactor. Total usage of this reactor was approximately 1,200 hours.



2. Coal Feeding Studies

The bench-scale coal feeder inlet seal and rotor assembly were modified to better handle the largest particle sizes (10 mesh) anticipated and provide a better "blow-by" seal for the smallest particles (200 mesh). Full range calibration at pressure and temperature is currently underway. Preliminary results indicate the minor difficulties with feeding the largest particle sizes have been eliminated.

3. Effect of Physical Properties on Bubble Size

Several observations have been made and photographed using the test setup described in the last report.

A new tower using a 3" I.D. plastic tube as the test section has been constructed, more nearly simulating the proposed high-pressure reactor. Several observations of bubbling in the test fluid have been made in this new tower, and have been photographed.

B. Projections

1. Environmental Testing of High-Temperature Materials

Parts for a new reactor are on order and as soon as they are received fabrication will begin. As soon as the new reactor is ready, Test #8 will commence. Samples presently scheduled for Test #8 are:

- a. McDanel AP-35 High-Purity Alumina
- b. Coors AD-99 High-Purity Alumina
- c. Zirconia 8-D

2. Coal Feeding Studies

Full-range calibration tests of the bench-scale coal feeder at proposed operating pressure and temperature will be completed.

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3. Effect of Physical Properties on Bubble Size

Several new spargers are being fabricated for observation of bubble patterns in the 3" test tower. As soon as these are ready, testing will continue.

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VI. MANPOWER AND COST ESTIMATES

Figure 2 shows the projected manpower breakdown for Phase I for 1965 as well as the actual effort that was made. It can be seen that a 13.3-man effort was made during October.

Figure 3 shows the expenditures during October. For the month \$24,309 was expended, not including fee and G & A. The total expenditures through October were \$267,482. Including fee and G & A the total expenditures were \$309,350. This is 51.5% of the encumbered funds.

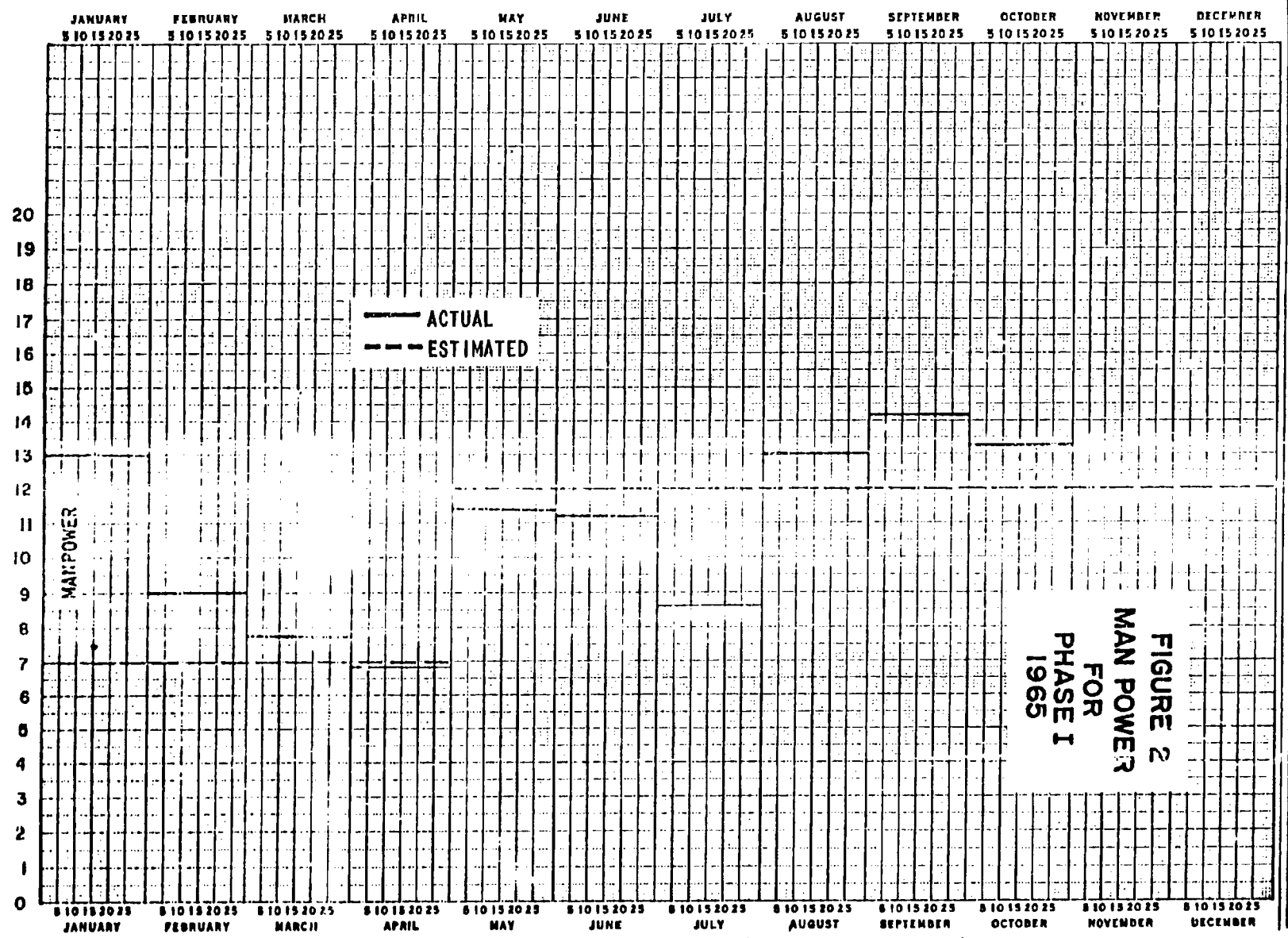


FIGURE 2
 MAN POWER
 FOR
 PHASE I
 1965

