

DEVELOPMENT OF KELLOGG COAL GASIFICATION PROCESS

Contract No. 14-01-0001-380

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Progress Report No. 21

APPROVED:

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RESEARCH & DEVELOPMENT DEPARTMENT

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RESEARCH & DEVELOPMENT DEPARTMENT

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1. 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 toal, 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:

C
$$\div$$
 H_2 0 \div heat \longrightarrow H_2 \div C0 (coal) (synthesis gas)

The necessary heat of reaction is supplied by circulating a heated molten sult 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 pariod. Phase I, which is now in progress, involves several concurrent efforts:

- Dench-scale process research -- to investigate melt properties, reaction kinetics, and the effect of process variables.
- 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.

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These I will be concluded by the design of a pilot plant to gasify 24 tons of coal per day.

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 II! 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.

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II. SUMMARY

This progress report is the twenty-first 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.

A gasification experiment at 10 atmospheres pressure, the highest pressure tested since the program began, was completed successfully during the month. Other conditions included: a temperature of 1740°F, a superficial gas velocity of 0.5 ft/sec, a quiescent melt depth of 4 inches, and an initial carbon concentration in melt of 4%. Eight other data points are available at these conditions, except for the steam partial pressure, which ranges from 5 to 55 psia. Ten additional points are available at 1840°F and varying pressures. A plot of these data on logarithmic coordinates showing gasification rate constant versus steam partial pressure yields two straight lines (one for each temperature level), which, it is felt, can be extrapolated confidently to the 400-psia region at which it is proposed to operate the process commercially.

Studies of the effect of melt depth in 2-inch ID and 4.25-inch ID reactors were continued. Two successful runs, at 4-inch and 8-inch depths, were made in the larger unit after several attempts failed because of heating problems. The measured gasification rate in the deep bed was only helf that in the 3-inch bed, and it was concluded that the melt had expanded out of the heated zone. If so, the effect of bed depth is confounded.

The position of the 2-inch ID reactor in its furnace was varied in several attempts to obtain similar temperature profiles for different bed depths. It was concluded that bed depth does have a significant effect on gasification rate in the 2-inch ID reactor. The rate decreases with increasing bed depth, apparently levelling off at about 50% of the value measured for the 3-inch depth. The decrease is probably caused by poorer contacting in deep beds of small diameter. Unfortunately, beds deeper than 8 inches cannot be tested in present equipment. (It should be noted that all bed depths mentioned above refer to depth of quiescent melt; as noted in Progress Report No. 20, the melt may expand as much as three-fold under gasification conditions)



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The effect of superficial gas velocity on gasification rate was studied in the 2-inch 10 reactor at 1740°F, 3 atmospheres, 4-inch melt depth, 2, ash in melt, and 4% initial carbon concentration. The rate doubles as superficial velocity is raised from 0.5 to 1.5 ft/sec. It increases another 10% at 2.0 ft/sec, but seems to be levelling off. Inefficient mixing of carbon in the melt at low velocities is proposed as the cause.

A complete "process package" is presented for the production of 250 million standard cubic feet of pipeline gas a day from bituminous coal. The package includes a process flowsheet, process description, material balance, utilities summary, capital cost estimate, and computation of gas production cost and selling price. Estimated capital investment is about 140 million dollars. Cas selling price is about 50¢/MSCF, based on \$4/ton coal and the CCR standard procedure for calculating gas cost. The plant also produces about 160 megawatts of electricity which is sold as a byproduct at 5 mills/kwh, yielding a credit of 7.6¢/MSCF of pipeline gas.

A specific gasification rate constant of 2.3 hr⁻¹ was used in the flowsheet design, resulting in a volumetric gasification rate of 21 pounds of carbon per hour per cubic foot of melt. This is compared in Table 1 with recent experimentally-measured rates.

Unfortunately, no experiment has yet been made to completely simulate commercial conditions (nor is this possible in present equipment). However, by using one experimental run (J-9814) as a base case and making the necessary corrections using correlations derived from other experimental data, one may conclude that the actual rate constant may be 5.9 hr.-1, about 2 1/2 times the rate used in design. This calculation assumes that the correction factors are well defined and that the corrections are additive, both of which require further investigation. The effect of gasification rates both higher and lower than that used in the design will be discussed next month.

Oxidation Test #3, a long-term corrosion test under simulated combustor conditions, was interrupted after 846 hours by a furnace failure. Monofrax \underline{A} and Inconel 600 have both shown excellent corrosion resistance. The test will be resumed as soon as possible and continued to 1000 hours.

A series of tests was conducted in the 4 1/4-inch ID reactor to measure bed expansion as a function of superficial velocity, gas composition, carbon concentration, ash concentration, and continuous coal feeding. Superficial gas velocity is a major factor, with melt depth almost doubling as velocity

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is increased from 0 to 1.0 ft/sec. Gas composition shows a second-order effect, with expansion decreasing slightly as N_2 , CO_2 , and steam are u.d as the fluidizing medium. Carbon concentration has little effect on equilibrium bed expansion, and the effect of ash concentration is not clear. Bed expansion with continuous coal feeding and gasification using a super-ficial gas velocity of 1.0 ft/sec is about 100%, the same as when using this superficial velocity in the absence of gasification.

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TABLE 1 COMPARISON OF GASIFICATION RATE USED IN FLOWSHEET DESIGN

WITH EXPERIMENTAL RESULTS

Flowsheat	Experimental
Design	(Run J-9814)
Bitum lous coal	Bituminous coal
1830	1830
250	مار
8	0
% 4	4 (initial)
0.9	0.5
15 feet	4 inches
2.3	1.3
	Design Bitum: ous coal 1830 250 8 4 0.9 15 feet

Corrections to experimental rate to match flowsheet operating conditions:

<u>Parameter</u>	Correction Factor
<pre>!ncrease avg. steam pressure to 250 psia (Figure 1)</pre>	X 4.2
Increase ash concentration to 8% (Report No. 15, Figure 5) Increase superficial velocity to	x 1.8
0.9 ft/sec (Figure 3)	X 1.2
increase bed depth to 15 feet (Figure 2)	÷ 2
Resultant specific rate constant, k, hr1 = 1.3 X 4.2 X 1.8 X 1.2 ÷ 2	= 5.9

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!!!. PROCESS RESEARCH

A. Accomplishments

Three major areas of concern have been the effect of decreasing specific gasification rate constant with increasing bed height, and the effect of pressure and superficial velocity on the rate constant. It is believed that the results this month answer these questions to a much higher degree than was previously possible. The run results are summarized in Table II.

First, the effect of pressure has been determined to 10 atmospheres absolute for the first time (run 9995). This run was made at 1740°F with maximum steam generation and condensation capacity at 0.5 ft/sec superficial velocity using a 4-inch bed of sodium carbonate and 4% carbon from bituminous coke IV. A brand new Incone: reactor, which ran very smoothly and gave no operational difficulties was employed.

The kinetic rate constant for this run and all the other pressure runs at 0.5 ft/sec, 4-inch bed height, and 1740°F have been plotted in Figure 1. It appears that a linear relationship best describes the data from 10 psia steam pressure and up. For convenience, the coordinate on the right side gives pounds of carbon prolified per hour per cubic foot of melt charged at a concentration of 4% carbon in the bed. Data for 1840°F, with all other conditions identical, are presented also. Thus, a reasonable extrapolation to the desired commercial pressure of 400 psia can now be made.

In connection with the study of bed height, two runs were successfully performed in the 4.25-inch reactor of the Mechanical Engineering Division (J-9987 and 9988). These runs were made at two bed heights, 4 and 8 inches, using 1740°F, 1.0 ft/sec superficial velocity, atmospheric pressure, 2% ash in the melt, and 4% initial carbon from Coke V. The rate constant at the low bed height was 1.38, about 20% better than

TABLE 11

GASTITICATION OF BITUMINOUS COKE IN MOLTEN SODIUM CARBONATE (1)

Ran No. J- Date - 1966	9985	9986	9987	99 <u>88</u> 478	9989 4/12	9990 4715	. 9991 81VA	9992 4720	9993 4721	9994	9925
Food	ζ-			Coke V	1/			ruser os sir	(•	Coke IV	
7 Total Carbon	. X =			93.2	i		1		` '	93.2	
% Vol. Natter	1	[0,6				ļ	i	0.6	1
7 Ash	38	19	176	6.2 88	28.5	14.3	38	19	19	6.2 19	19
gms, charged mesh size	200		1/5	12/20	20.5	111.3	ا تار	,,		' ' ' '	
11.12.003 - 9ms	311.4	405.7	39007		603.6	304.3	812.4	405.7	405.7	405.7	414
Ash - gms	16,6	8.3	<i>[</i>]	5	12.7	6.4	16.6	8.3	8.3	8.3	0
Zin melt	2	2	2	2.	2	2	2	2	2	2.	0
C in melt init.	4	14	14	4	4	1;	4	4	14 14	14	4
Sed Height - in.	8	14	8	4	6	3	8	- 1	"	14	4 ;
Conditions		1		·		ar - 1 m m - 1 m - 1 m - 1 m - 1					
Γemp °Γ	1740	1740	1740		17/10	1740	1732				1740
Pressure - psia	45.0	44.7	14.7	14.7	45.2	44.5	45.2	45.5	45.2		149.7
Steam Prespsia	40.4	39.6	13.4	13.2	40.4	40.2	41.0	40.1	39.7	10.3	132.6
Gas in Steam Ft/sec Stm. & Gas	1.02	1.05	1,07	1.01	1.04	N ₂	1.03	1.56	0.51	2.01	0.51
Minutes to 0% CO	40	30		65	35	25	45	20	45	25	25
Total Run-min.	45	35	85	75	35	35	50	25			
cc H ₂ O in/hr			1756			1173		1738			1886
cc N ₂ in/min	2268	3108	3668	3985	2900	2600	2468	4870	1351	/i501	5050
Posults	 									•	i
Total % C to Coxide	99	97		100	97	100	101	92	100	92	100
Gasif. Rate Constant	\$			}							
Basis Input	1.68						1.31				1.80
Basis Output	1.70	2.14	0.69	1.38	1.94	2.40	1.29	3.16	1.57	3.05	1,80
Rate-Ibs.C/hr/CF a 4% C in bed	20.1	25.3	8.1	16.3	22.9	28.3	15.2	37.3	18.5	36.0	21.2
Salt Carryover-gms	5.1	12.3	6.4	8.1	4.7	13.8	5.4	15.7	6.3		2.2
Hotes	(2)	(2)	(4)	(4)	(2)	(2)	(3)	(2)	(2)	(2)(5)	(2)
1					<u> </u>	<u> </u>	<u> </u>	<u> </u>		ļ	

(1) Island Creed #27 bituminous coal coked at 950°C (cokes IV and V); 2-inch ID inconel reactor except rins 9987 and 9988; coke charged in nitrogen atmosphere at 0.05-0.1 ft/sec.

⁽²⁾ Three and four-inch beds had reactor bottom 8 inches from bottom of furnace; six-inch bed was 5.5 inches from bottom, eight-inch bed was 2 inches from bottom.

⁽³⁾ Bottom of reactor was 8 inches from bottom of furnace.
(4) Runs made in 4.25-inch diameter reactor by Mech. Eng. Division.
(5) Rate constant corrected from 1710°F (for first 15 min.) to 1740°F gives 3.5.

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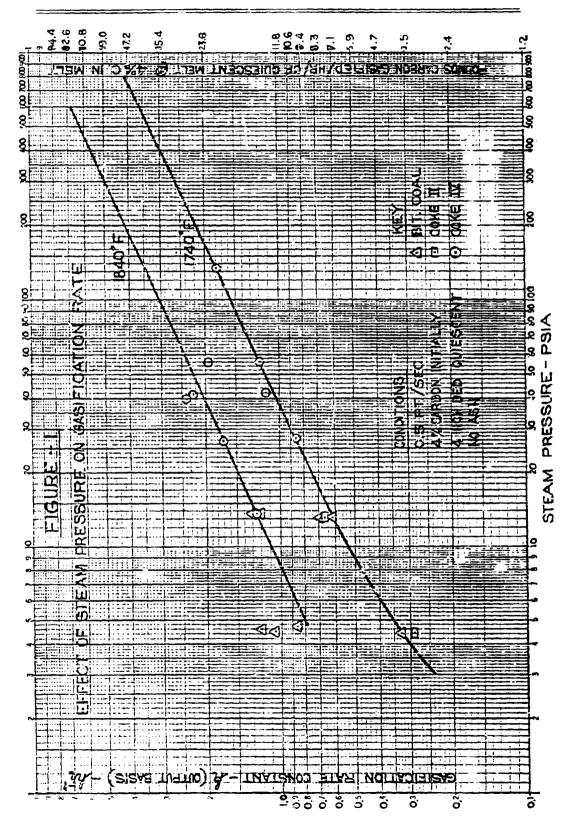
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rate results from almost equivalent runs in the 2-inch diameter reactor. Unfortunately, the rate constant for the high bed was 0.69, half the above value; despite twice the quantity of carbon available, only the same amount reacted as in the low bed. Based upon expansion studies, described in last month's summary, the 8-inch bed during gasification would have exceeded the heating zone of the furnace. Consequently, isothermal conditions hardly could have prevailed, and the poor rate shows it.

In order to eliminate the effect of temperature on reaction rate at different bed heights, the position of the 2-inch diameter reactor in the furnace was varied, thus attempting to obtain similar temperature profiles. Temperatures for two runs at 3- and 6-inch bed heights showed identical rising profiles. Thermocouples above the molten salt were reading 70°F higher than the molten salt. The reaction rates and operating conditions for these runs are given in Figure 2 and Table II (1-9985, 9986, 9989, 9990).

The effect of increasing bed height, at least from 3 to 6 inches, was a 19% loss in rate constant for runs in the 2-inch diameter inconel reactor. The run at 8-inch bed height expanded during gasification beyond the isothermal zone of the furnace despite having the reactor at its lowest possible position. Thus this point (k=1.7) was ignored in drawing the curve for Figure 2.

A deliberate expansion of a bed out of the Kanthal furnace was made with an 8-inch bed placed at the usual position for a 3 or 4 inch bed (J-9991), thereby raising the bottom of the reactor 8 inches above the bottom of the furnace. Gasification showed a 40% drop in rate constant from the result for a 4-inch bed under identical conditions. This, of course, adds further weight to the conclusion that this system cannot test an 8-inch bed without temperature effects. Thus, bed heights of 8 inches and higher must be tested in larger furnace and reactor equipment.

It can be concluded that bed height has a significant effect on reaction rate in the 2-inch diameter reactor. This effect is most probably due to decreasing efficiency of contact between steam and carbon with increasing bed height. It is suspected that carbon distribution is at fault.

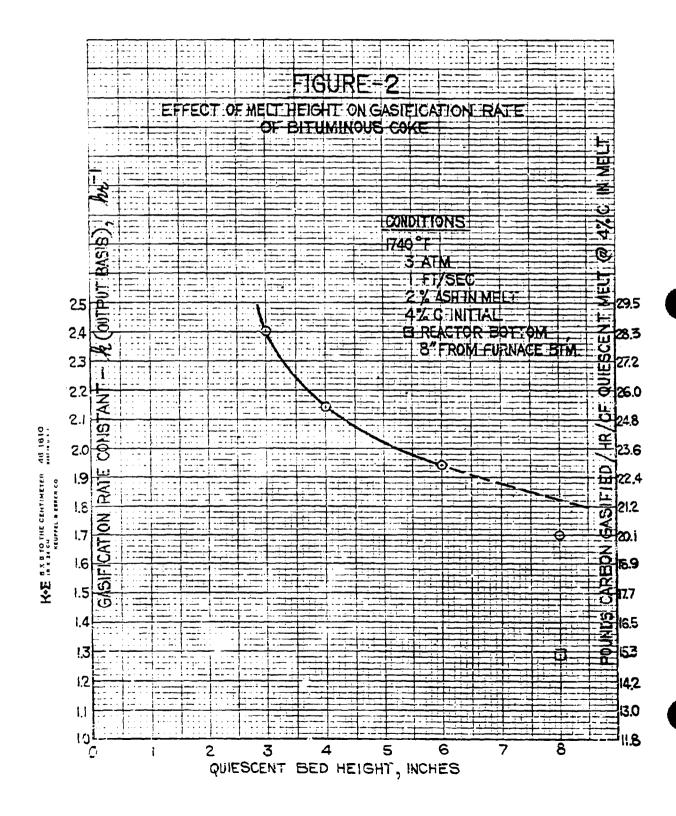
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The next phase of the work considered the effect of superficial gas velocity on the rate of gasification. Up to now tests above I ft/sec have always plugged. With the new technique of introducing the carbonaceous solid into the melt with only nitrogen flowing, devolatilizing for 5 or 10 minutes, then gasifying with steam, runs can be made at higher velocities without the plugging complications previously experienced. Runs have been made, as shown in Figure 3, from 0.5 to 2 ft/sec superficial velocity. The run at 2 ft/sec taxed the heat duty of the Kanthal furnace, as well as the steam generating and condensing capacity of the unit. Temperature during the first fifteen minutes was 1710°F, and by a previous correlation the measured rate constant was corrected to 1740 F. Increasing rate of gasification with increasing velocity strongly indicates that contacting of steam and carbon is the principal problem. It does appear that very little in rate is to be gained by velocities above 1.8 ft/sec. Thus far it appears that distribution of carbon in the molten salt using the 2-inch diameter reactor is the main problem. It is expected that this problem will be diminished in larger diameter reactors.

Although correlations of salt carryover have not been made yet, it is of interest to point out that salt carryover was lower in the 4.25-inch reactor setup than in the 2-inch reactor. Also, during gasification at 10 atmospheres pressure and no ash in the melt, the lowest salt carryover ever recorded was obtained, namely 2.2 grams.

B. <u>Projections</u>

Gasification studies to define effects of carbon concentration, feedstock, particle size, and ash concentration have to be made. Return to combustion studies, especially under pressure, is contemplated.

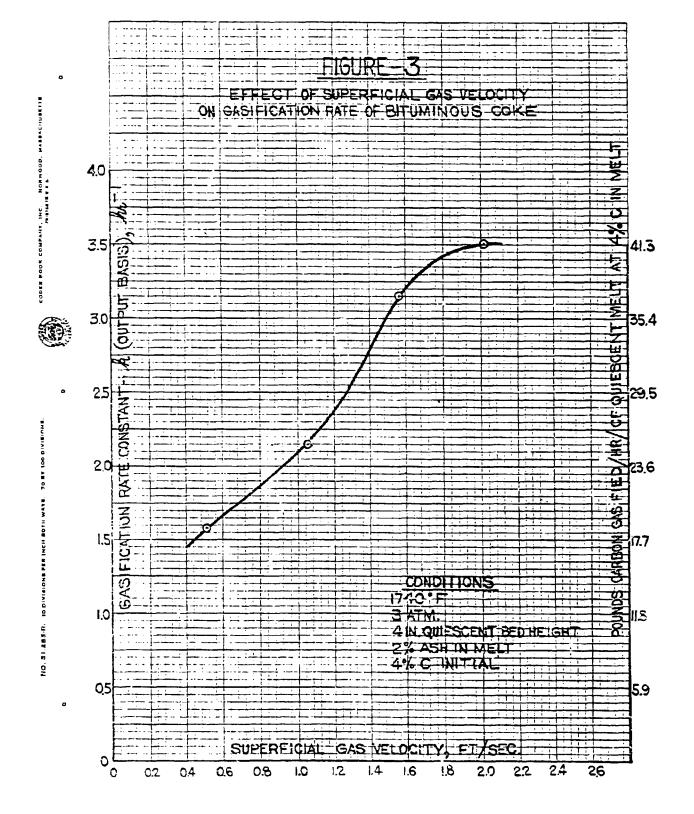
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IV. CHEMICAL ENGINEERING STUDIES AND DEVELOPMENT

A. Accomplishments

A complete "process package" has been developed for the production of 250 million standard cubic feet of pipeline gas a day from bituminous coal using the Kellogg Molten Salt Gasification Process. The package includes a process flowsheet illustrating the conceptual design of the plant. a capital cost estimate, a computation of gas production cost and selling price, a material balance, a utilities summary, and a tabulation of bases and assumptions incorporated in the design. These elements are presented in the following sections.

Process Description

The process flowsheet for a plant capable of producing 250,000,000 SCFD of pipeline gas from bituminous coal is presented as Figure 4. Flow rates and compositions of the various numbered streams on Figure 4 are shown in Table III. In addition, a section-by-section material balance is given in Table IV. A brief description of the flowsheet follows.

a. Section 100 - Coal Storage and Preparation

During eight hours each day, coal is received by truck or conveyor belt from an adjacent coal mine at the rate of 1650 tons per hour. The raw coal travels by belt conveyor to a coal distributing center, where about 550 tons per hour is dispatched for immediate use and the remainder is conveyed to the storage area.

Coal is distributed to several storage piles by a shuttle conveyor. These piles contain enough coal to permit the plant to operate for 30 days at normal capa- . city in the event the coal supply is cut off.

During the 16 hours each day that the mine is not operating, coal is reclaimed from a ready storage pile for use in the process.

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Coal from storage flows by belt conveyor to a Bradford Breaker, where it is reduced in size to about minus 2 inch. This coal is then fed to four parallel hammermills designed to reduce the coal particle size to minus 12 mesh. The crushed coal travels by conveyor belt to three parallel coal storage bunkers, from which it is carried by conveyor belt to Section 200.

b. Sections 200 and 600 - Gasification and Ash Removal

Bituminous coal from storage, ground to approximately -12 mesh in Section 100, is fed to a set of lock hoppers, F-201, a & b, whose purpose is to receive the coal at atmospheric pressure and to deliver it to the process at the operating pressure of about 415 psia. Each of the lock hoppers operates on a 30-minute cycle comprising the following steps:

- (!) Filling with coal at atmospheric pressure
- (2) Pressurizing to about 450 psie with synthesis gas or product gas
- (3) Discharging the coal into the steam line
- (4) De-pressurizing

The lock hopper system is designed so that while one hopper is being filled and pressurized, the other is discharging coal into the process stream. This provides a continuous flow of coal to the dasifier.

Coal from F-201 a & b flows by gravity at the rate of 1,100,000 pounds per hour into a steam line where it is mixed with 420 psia steam superheated to 1000°F in preheater C-201. The amount of steam used as a carrier gas is such that the coal will not be heated above about 500 F, thus preventing the coal from becoming plastic and sticky in the lines. Another stream of this 1000°F steam is used to pick up a recycle stream of NaHCO3-Na2CO3 from lock hoppers F-202 a & b. The two solids-containing steam streams then flow to the gasification section of gasifiercombustor D-201 together with the remainder of the 1000 F steam. The total steam fed to the gasifier is 1,000,000 pounds per hour. Of this, 920,600 pounds per hour is generated in waste heat boilers C-204, C-206, C-207 and 79.400 bounds per hour is generated in waste heat boilers in other sections of the plant.

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In the gasifier, the steam and coal are heated to 1830°F by intimate contact with melt (a mixture of molten sodium carbonate, coal ash, and coal) and react according to the following reactions to produce systhesis gas.

The raw synthesis gas leaves the melt at about 1830° F and 405 psia and flows through separator G-201 designed to remove entrained coal particles and melt droplets. The gas is then cooled to 700° F in exchangers C-201, C-203 and C-204 and leaves the gasification section at the rate of 118,105 moles per hour.

The primary gasification reaction (reaction 1) is highly endothermic; the required heat of reaction is supplied as sensible heat of circulated melt. In the version shown in Figure 1, the gasifier-combustor is divided into two sections by a vertical wall which is perforated below the liquid level. By proper choice of gas velocity, a difference in degree of aeration of the melt in the two sections is induced. This causes meit to circulate from the synthesis gas section, where it provides the reaction heat, to the heating or combustion section. In this latter section heat is added to the melt by direct contact with hot combustion gases.

The flue gases which supply heat to the combustion section of the gasifier-combustor are generated by combustion of coal with air. Air for this purpose is compressed at the rate of 4,285,200 pounds per hour to 420 psia in compressor J-201 and is preheated to 1,000°F in exchangers C-202 and C-203. Combustion occurs in direct contact with the melt, transferring heat to the reacting system efficiently.

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The hot flue gases leave the bed at about 2200°F and 405 psia and pass through separator G-202 to remove entrained coal and melt. The gas then flows through exchangers C-206 and C-202 where it is cooled to 1500°F. This stream is expanded to substantially atmosphere pressure in J-202, which provides all the power required for air compression plus an additional 76,800 km of electricity in generator J-204. Expanded flue gas at 615°F and about 18 psia is cooled to 180°F in exchangers C-207, C-208, and C-209 and is vented to the atmosphere at the rate of 4,655,300 pounds per hour.

The ash left in the melt by the combustion and gasification of the coal is allowed to build up to a level of 8 weight percent. A slipstream of the ash - carbon -NapCO3 mixture is continuously withdrawn from D-201 and flows to E-601, where it is quenched to 444°F with a portion of a recycle solution saturated with NaHCO3 at 100°F. Solid melt particles in the resulting slurry are ground in L-603 to facilitate dissolution of the melt stream. The remainder of the recycle NaHCO2 stream is then mixed with this slurry, cooling the mixture to 228°F. This stream is then flashed to 16.1 psia in F-601, where sufficient holding time is provided to dissolve the Na₂CO₂. The bottoms slurry-from F-601 is filtered in 1-601 to separate the ash and carbon (and some undissolved NagCO₂) from the solution. This residue is sent to disposal.

The filtrate from L-601 is pumped up to 30 psis in J-602 and is fed to carbonation tower E-602. In this tower the Na_2CO_3 is reacted with CO_2 from the gas purification system according to reaction (6).

$$Na_2CO_3 + CO_2 + H_2O = 2NaHCO_3$$
 (6)

Overhead gas from the tower at 200°F is cooled to 95°F in C-601 to condense water, a portion of it is purged to remove the impurities brought in with the $\rm CO_2$ stream, and the remainder is recycled through J-604 to the tower. Fresh $\rm CO_2$ is added to the tower at the rate of 10,396 moles per hour.

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The operating temperature at the bottom of E-602 is $100\,^{\circ}$ F. At this temperature the N_aHCO_3 (along with some ash) is precipitated. The resulting slurry is filtered in L-602, the N_aHCO_3 solution being pumped up to $400\,^{\circ}$ psia and recycled to E-601. The filter residue (N_aHCO_3) is dried by hot gases flowing through the drying hood and is returned to lock hoppers F-202 a & b to be recycled to the gasifier-combustor.

Because of the large volumes of gases processed, Sections 200 and 600 consist of nine parallel trains of operating equipment.

c. Section 300 - Shift Conversion

Synthesis gas leaves Section 200 at the rate of 118,105 moles per hour and flows to Section 300, where about 68 percent of it is fed to shift converter D-301. Boiler feed water at 200°F is fed between beds of catalyst at the rate of 146,980 pounds per hour to absorb the heat of reaction. The remainder of the synthesis gas is bypassed and is combined with the shift effluent. This stream, at 735°F and having a H2/CO ratio of about 3.15/1 (in preparation for methane synthesis), is cooled to 255°F in exchangers C-301, C-302, and C-303. Condensed water is separated from the gas in F-302, and the gas is further cooled to 100°F in C-334. The gas then flows to scrubber E-301, where the gas is countercurrently scrubbed with clean water to remove trace amounts of ammonia which might be present.

Section 300 is designed as five parallel operating units.

Section 400 - Gas Purification

The gas purification section is designed to reduce the $\rm CO_2$ concentration in the shifted synthesis gas to 1.0 mole percent and to reduce the total sulfur concentration to about 0.004 grains/100 SCF of gas. The purification sequence consists of the "Fluor Solvent $\rm CO_2$ Removal Process" for $\rm CO_2$ and bulk sulfur

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removal, followed by sponge iron (iron oxide) and activated carbon for residual $\rm H_2S$ and organic sulfur removal, respectively. All organic sulfur is assumed to be carbonyl sulfide (COS). The selection of the Fluor process for use here was based upon an economic comparison with various alternate schemes. The quantitative results of this comparison were reported earlier (1).

The shifted synthesis gas from Section 300 is cooled to 35°F in exchangers C-401, C-402 and C-403. Hydrate and ice formation is prevented by injection of lean ethylene glycol into the gas. The glycol is subsequently separated from the gas in F-402. The chilled gas is then fed to absorber E-401, where it is countercurrently contacted with lean Fluor solvent. Rich solvent leaving the bottom of E-401 is cassed through a power recovery turbine on J-402 to a 20 osia flash drum, F-401, for partial desorption of gases. This is followed by a vacuum flash at 3 psia for complete regeneration of the solvent. Lean solvent from the vacuum flash step is recirculated to E-401 through J-401. Intermediate flash gas is exchanged against feed gas in C-402 and is combined with the final flash gas after this gas is compressed from 3 psiu to 20 psia in J-402. About 60 percent of the flashed mases is purged from the system; the remainder of the gas is compressed to 30 psia in J-403 and flows to Section 600.

Clean synthesis gas leaves the absorber at $25^{\circ}F$ and is heated to $80^{\circ}F$ against feed gas in C-40ì. At this point the gas contains i.0 percent CO_2 , 6 grains $H_2S/100$ SCF, and 0.2 grains COS/100 SCF, and is next treated for removal of residual H_2S . The gas is contacted with finely divided iron oxide supported on wood chips, commonly called "sponge iron", which removes H_2S practically quantitatively by the classic iron oxide reaction, as follows:

⁽¹⁾ Progress Report No. 6, Contract 14-01-0001-380, January 31, 1965, P. 3.

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$$Fe_2O_3 \cdot 6H_2O + 3H_2S \longrightarrow Fe_2S_3 \cdot 6H_2O + 3H_2O$$
 (7)

Periodically, the sponge iron is revivified with air to carry out the following reaction:

$$Fe_2S_3 \cdot 6H_2O + 1 \frac{1}{2}O_2 \rightarrow Fe_2O_3 \cdot 6H_2O + 3S$$
 (8)

The sulfur is deposited in the amorphous form around the sponge iron particles and eventually encloses the sponge iron in an impervious mat, whereupon it must be discarded. Each of the five trains of equipment contains four parallel iron oxide drums, D-401 a-d, followed by an iron oxide guard chamber D-402.

Synthesis gas leaving the iron oxide drums is finally treated for COS removal by adsorption on fixed beds of activated carbon, D-493 a-c. The activated carbon drums are arranged in ten parallel trains, each train consisting of three drums which are manifolded for cyclic operation. Typically, for each train, gas flows through two vessels in series for twelve hours while the third is being regenerated with steam. During this period any COS leakage from the first drum is retained in the second. At the end of the period the first drum in line is taken off stream for regeneration, the second drum is moved into first position, and the freshly regenerated drum is placed into second position. This cycle is repeated every twelve hours.

The purified gas, containing about 1.0 percent ${\rm CO}_2$ and an average of about 0.004 grains total sulfur per 100 SCF, proceeds to Section 500.

e. Section 500 - Methane Synthesis

In this section the purified synthesis gas is catalytically converted to methane as follows:

$$CO + 3H_2 \longrightarrow CH_4 + H_2O + heat$$
 (4)

The methanation scheme selected for use here is the Hot-Gas-Recycle System developed by the Bureau of Mines. The basis of this selection was an economic comparison with the other possible methanation systems, the results of which were previously reported. (2)

⁽²⁾ Progress Report No. 11, Contract No. 14-01-0001-380, June 30, 1965, P. 3

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Gas from Section 400 at 80°F and 340 psia is preheated to 400°F in C-501. This stream is then mixed with recycle gas at 583°F and flows downward through iron catalyst reactor D-501. Each reactor contains four beds of iron catalyst in the form of lathe turnings, with internal cooling coils located between beds. As the gas flows through the reactors, heat of reaction is removed by generating steam in the coils of waste heat boilers C-502, C-503 and C-504.

Reactor effluent at 626°F is split into three streams; a hot recycle stream, a cold recycle stream, and a net reactor effluent. The cold recycle stream is cooled in exchangers C-505, C-512 and C-506 to 150°F and is separated from entrained condensate in F-503. The cold recycle is compressed by J-502, preheated to 410°F in C-505, and mixed with the hot recycle stream. Total recycle gas at a mix temperature of 573°F is compressed by J-501 and returned to the reactor inlets.

Net effluent from D-501, containing an appreciable quantity of hydrocarbons heavier than methane, flows to nickel catalyst reactor D-502, where the heavy hydrocarbons are cracked and the methanation reaction is completed. This reactor is physically separated into three sections, with each section containing an equal volume of Raney nickel catalyst. External coolers C-507, C-508 and C-509 are provided between sections to remove the heat of reaction which is used to generate steam. U-502 is also designed for recycle operation with the sotal recycle gas being fed to the top bed. Feed to the reactors is split into three streams which are fed to each of the three reactor sections.

Net product from D-502 is cooled to $193^{\circ}F$ by heat exchange with fresh feed in C-501, further cooled to $100^{\circ}F$ in C-510, and is separated from condensate in F-504. The gas is then compressed to 1015 psia in

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J-506, cooled to 100°F in C-511, and delivered to F-505, where entrained condensate is separated and purged from the system. Final product gas with a heating value of 914 ETU/SCF flows from F-505 to the gas mains at 1000 psia and 100°F at the rate of 250,000,000 SCFD.

Section 500 employs five parallel operating trains of equipment, as indicated on Figure 4.

f. Section 1100 - Offsite Facilities

Section 1100 (not shown on Figure 1) includes facilities for:

- generating steam and electric power
- (2) supplying cooling water, process water and boiler feed water
- (3) providing miscellaneous services necessary to make this a completely self-sufficient plant

Steam generation facilities consist solely of a start-up boiler capable of producing 375,000 pounds per houroof 420 psia steam at 1000°F and 125,000 pounds per hour of 420 psia, saturated steam. Once the plant is in full operation, enough steam is generated by waste heat in the process to provide all the high- and low-pressure steam needed.

Electric power is produced at 13,800 volts by turbogenerators using condensing steam turbine drives. An electric substation is provided to reduce the voltage to 4160, 440 and 110 volts.

A complete utilities summary is included as Table V.

2. <u>Economics</u>

The cost of producing 250,000,000 SCFD of pipeline gas from bituminous coal according to the process sequence just described is calculated in Tables VI and VII, assuming 90 percent stream efficiency. The procedure used is in accordance with OCR's tentative standard for cost estimating of pipeline gas plants. (3)

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

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Estimated capital investment is summarized in Table VI. Shift catalyst and activated carbon are included in fixed investment because they have very long lifetimes. Total capital investment is about \$140,000,000.

Estimated operating expenses and gas selling price are shown in Table VII. Bituminous coal is charged at \$4 per tor. Total operating expense is calculated to be 41.6c/MSCF, and gas selling price, based on a 20-year average return on equity capital of about 9.4 percent, is 50.3c/MSCF.

It should be noted here that a credit of 7.6c/MSCF is taken in the economics for the excess power produced by the waste heat (steam) available from the process. It seems entirely logical to treat the plant as an energy center, supplying an area not only with pipeline gas but with a portion of its electric power needs as well. Credit is taken for the available power at 5 mills/kwh, the cost of producing it in a conventional 160-megawatt power plant burning coalat \$4 per ton.

Design Bases and Assumptions

It is the purpose of this section to point out the important design bases and assumptions which are inherent in Sections 200 and 600 shown in Figure 4.

Calculated economics for the process are based on the premise that the gasification and ash removal steps can be carried out as shown on Figure 4, although these two operations have not as yet been demonstrated on a fully integrated basis at flowsheet conditions. It has yet to be proven, for example, that the gasifiers can operate at the proposed gasification and combustion rates (kqas. = 2.3~#C gasified/hr - #C in melt = 21.2~#C gasified/hr - CF melt; kcomb. = 1.2~#C burned/hr - #C in bed = 11.6~#C burned/hr - CF melt) at 400 psia, a 2/1 steam/carbon ratio, and with melt heights of 15 feet. In addition, a number of other assumptions had to be made in preparing this design. Briefly, the most important of these are as follows:

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- (1) Adequate heat transfer from gas to melt in combustor;
- (2) Adequate melt circulation from combustor to gasifier in order to transfer the required heat;
- (3) Separators will remove entrained melt to a point where the flue gas expander can operate and where no solids will deposit on downstream surfaces;
 - (4) Satisfactory materials of construction;
 - (5) Complete combustion of coal to CO₂ in combustor;
- (6) All oxygen in gasification coal combines with hydrogen;
- (7) All excess hydrogen in gasification coal goes to methane;
 - (8) Water-gas-shift equilibrium in gasifiers;
 - (9) Na₂ CO₃ losses at 2.4 percent per pass.

B. Projections

1. Pipeline Gas

A similar process package for pipeline gas from <u>subbituminous</u> coal will be completed. The effect of further variations in design bases and assumptions (in addition to those reported last month) will be studied.

2. Hydrogen

A conceptual design of a plant to produce 250 MM SCFD of hydrogen from bituminous coal will be started. Desired product gas characteristics were presented in the first monthly progress report under this project. Subsequent study showed however, that product gas cost can be reduced by 10-15% if a higher concentration of methane plus nitrogen (5% rather than 1%) is tolerable. Since these components will be inert for many hydrogen end uses the relaxed specification will be accepted as the objective of the present study. A complete product gas specification is presented below:

⁽⁴⁾ Progress Report No. 5, Contract 14-01-0001-380, January 31, 1965, P. 9A.

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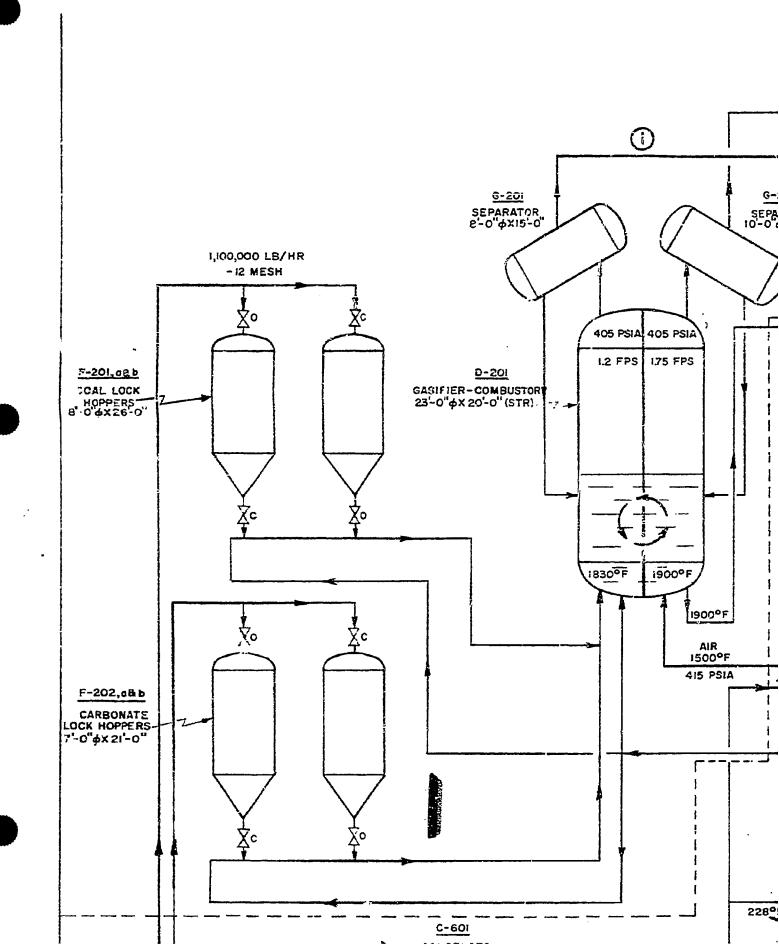
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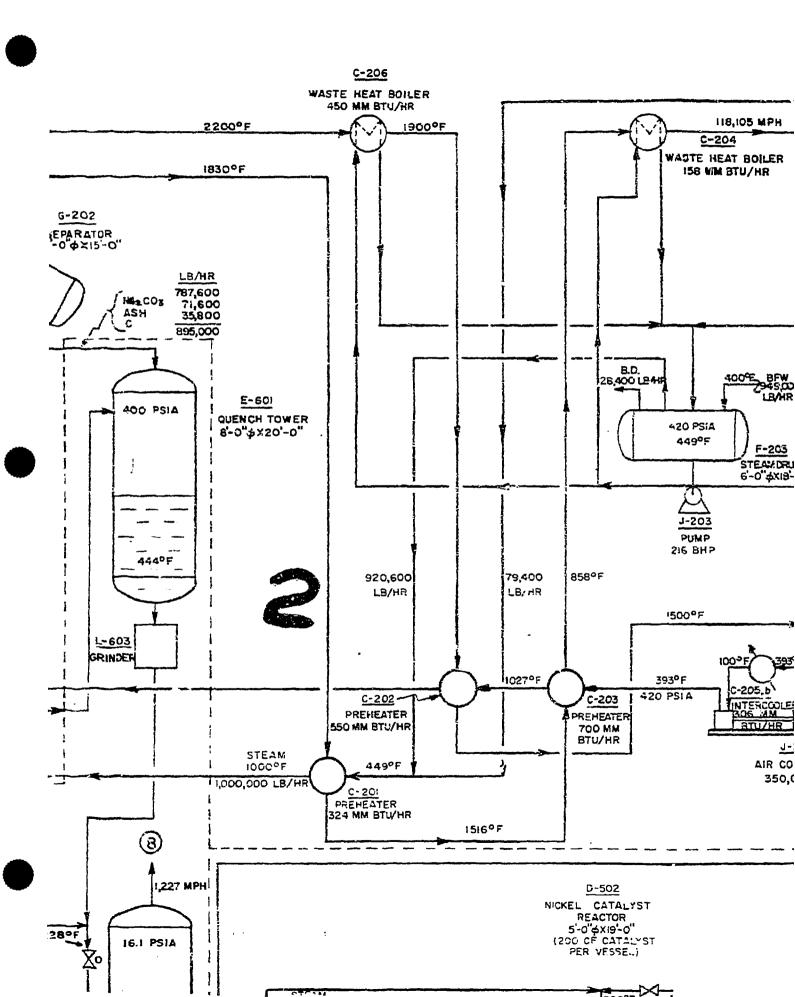
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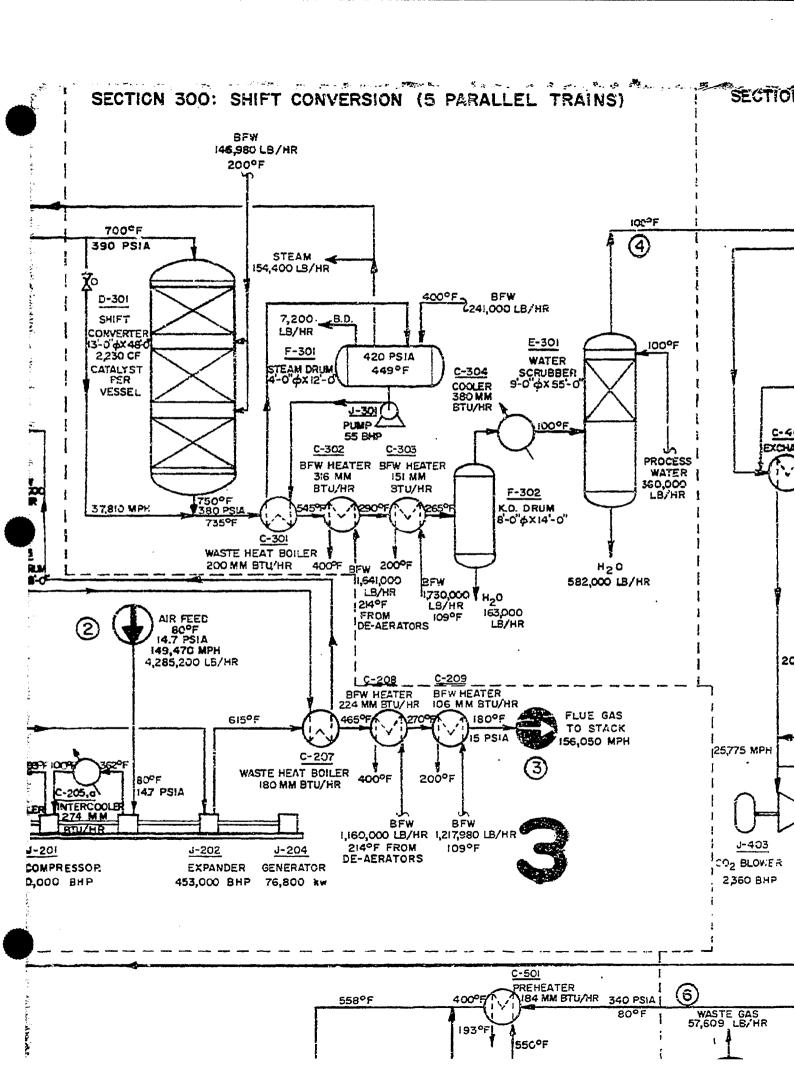
Quantity, SCFD 250,000,000 Temperature, F 100 Pressure, usig 500 Composition:

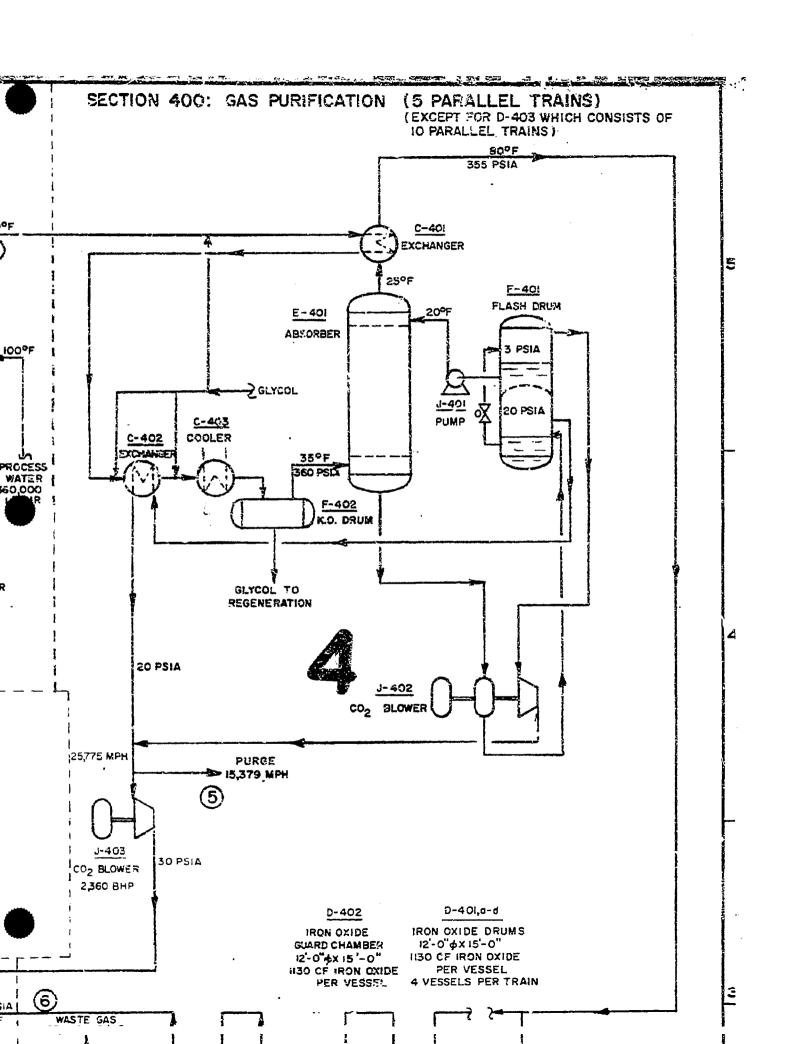
H2 $\sim 95\%$ CH4 + N2 $\sim 5\%$ CO + CO2 10 ppm max. S compounds i ppm max.

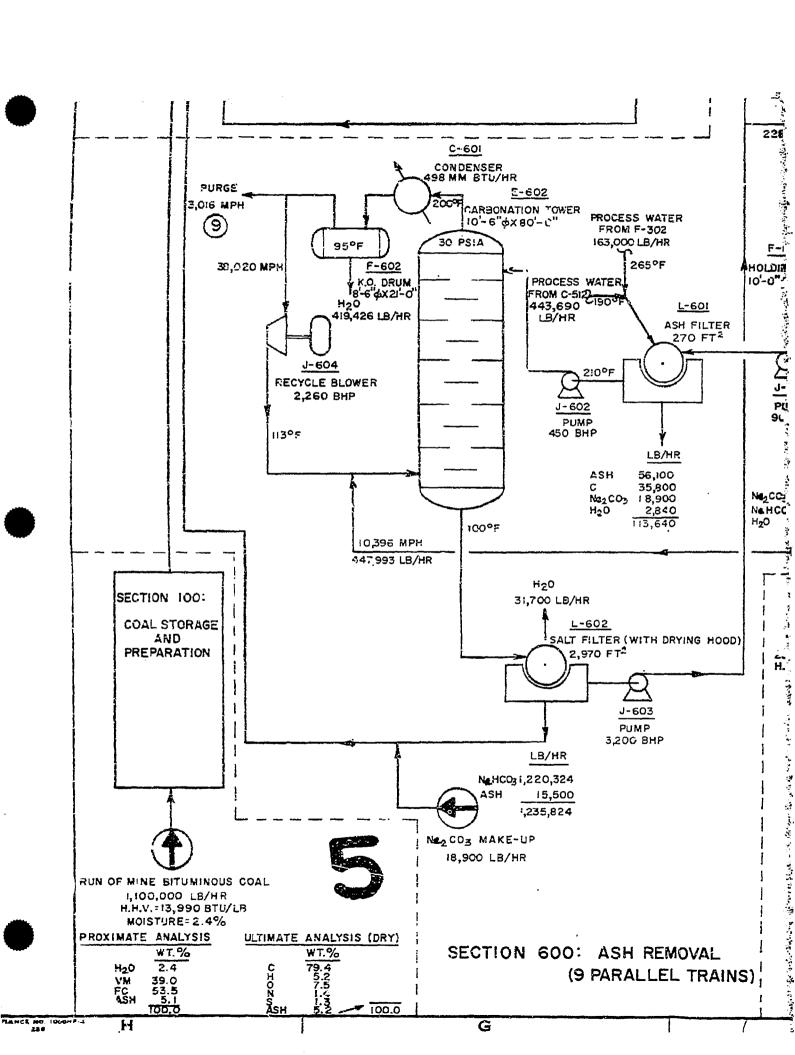
A complete process package for hydrogen production is the ultimate goal of this effort.



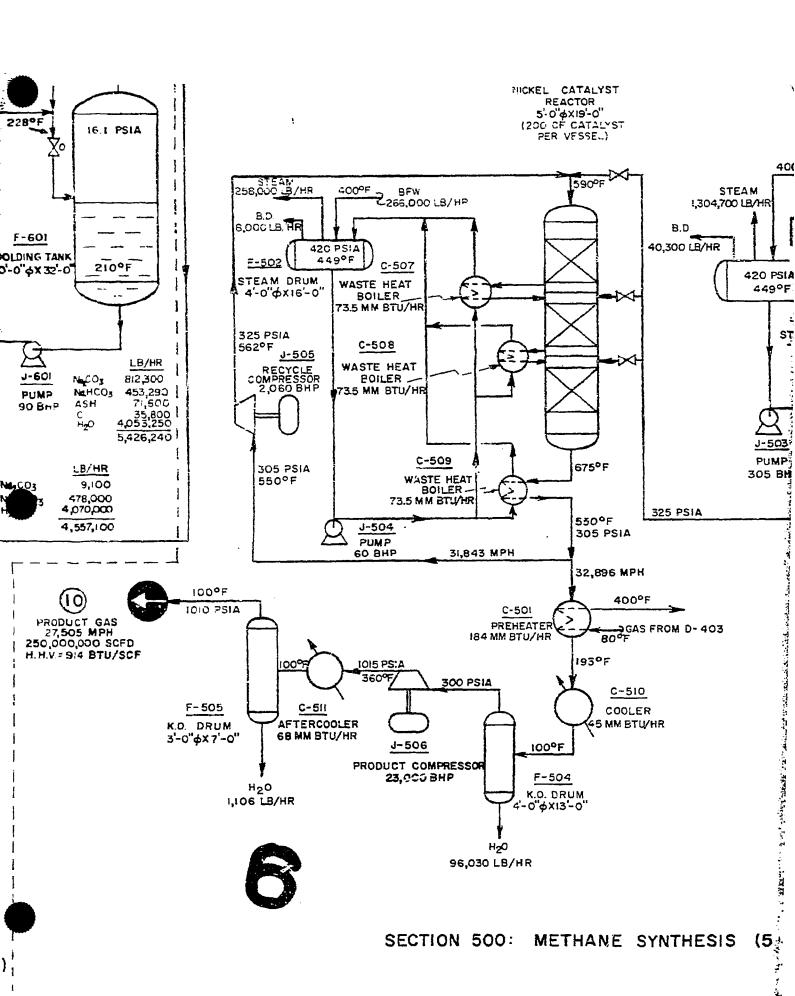






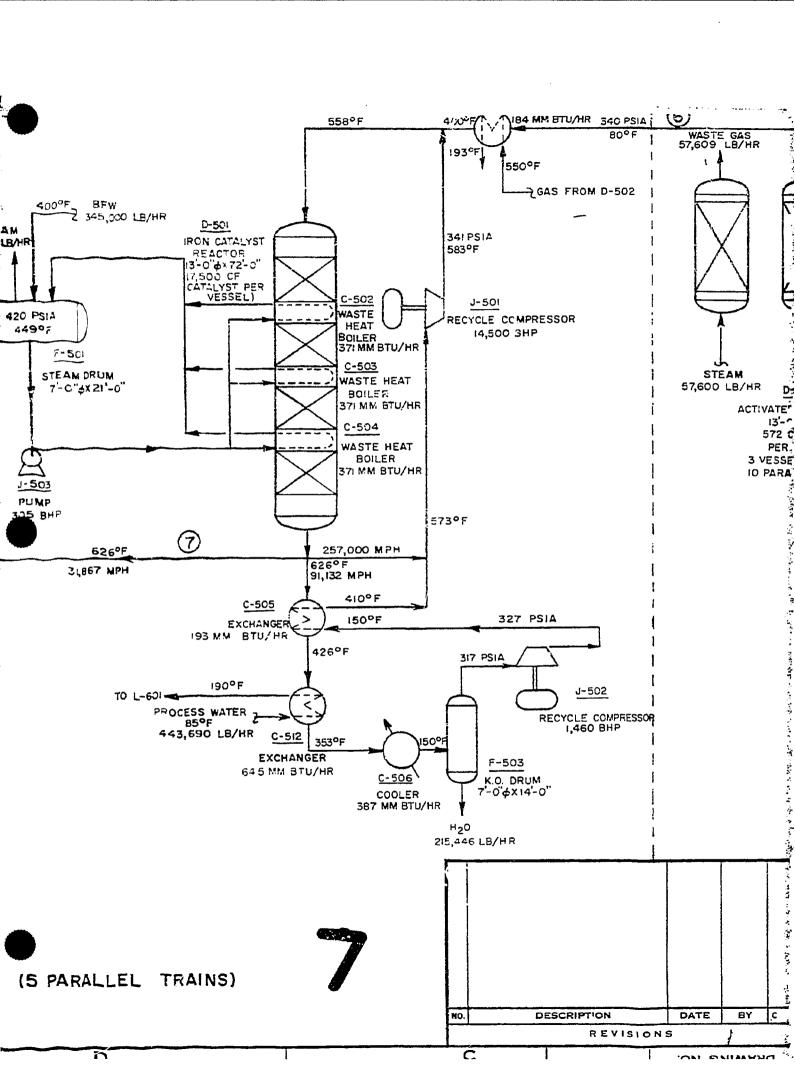


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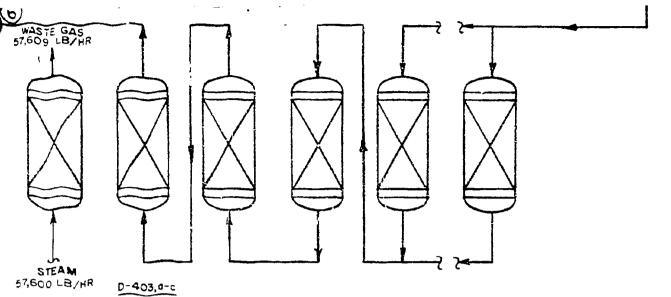


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ACTIVATED CARBON DRUMS
13-6 \$\phix:5-0"
572 CF CARBON
PER VESSEL
3 VESSELS PER TRAIN
10 PARALLEL TRAINS

NOTE: ALL QUANTITIES SHOWN ON THIS FLOWSHEET ARE TOTALS FOR ALL PARALLEL TRAINS UNLESS OTHERWISE NOTED.



THE M. W. KELLOGG COMPANY a division of FULLMAN INCORPORATED PIPELINE GAS FROM BITUMINOUS COAL 250,000,000 STANDARD CUBIC FEET PER DAY SCALE: DRAWN: OFFICE OF COAL RESEARCH CHECKED. CONTRACT NO. 14-01-0001-380 APPROVED: DATED: 4/1/66 ISSUED FOR FABRICATION L.O. 6026 6026-3-5 CHECKED ISSUED FOR DATE BY CLARE JOB NUMBER DN S AREA DRAWING NUMBER & ITEM Ā DRAWING NO.

TABLE III Process Stream Balance

250,000,000 SUFO Pipeling Gas from Blauminous Coal

Stream No.		$\overline{\Omega}$			\bigcirc			180			Q			Q	
Traperature, F		1830			80						100			70	
Pressure. Psia		405			14.7			15			_370		ļ	20	L
Flow Pale	16/00	14015/111	Hoi. /	Lb/Hr	tols/Hr.	Hol.	Lb/Hr.	Hots/lir.	Mol. 7 (0ry)	L6/IIr.	Hols./IIr.	HO[. 4	Lb/lir.	Mals/Hr.	Hol. / (Dry)
co	859,000		33.5						-:	486,000 1,120,530	17,330 25,460	16.5 24.3	4,380 647,330	156 14,714	1.0 96.7
CO2	535,000 111.500	12,160 6,370	13.3 7.6				1,108,140	25,200	17.6	111,500	6,910	6.7	1,590	99	0.7
614 ₁₄	82,044	41,072	44.9	•				116 105		108,644	54,322 355	51.9 0.3	166	83 0.6	0.5
H2 R7 H25	9,950 9,350		0.4	3,250,000	116,000	79.0	3,255,460	116,195	81.3	9,950 9,550	281	0.3	5,550	163	1,1
ros l	780	13	0.01	••••						430	7	0.00	1 1	4	υ,03
02				989,000	30,900	21.0	46,600 10,100	1,457	. 0. 1.u						
07 507 67										•		••••			
Total Dry Gas	1,607, 62 4	91,425 26,680	190,0	4,239,000 46,200		100.0	1,420,300 235,000	143,010 13,040	100.0	1,846,604 5,000	104,725 278	100.0	659,296 2,887	15,219.6 159	100,0
Total Wet Gas	2,089,624	118,165		4,285,200	145,470		,655,300	156,050		1,851,604	105,003	<u> </u>	662,168	15,378.6	
		(6)			①			®			(a)			(i)	
Stream No. Temperature, f		80			626			210			35 27			100	
- Pressura. Pala-		340	Hol. X		325	Hol.	<u> </u>	-13:	Mo1.2		, '' ·	Hol. %	 		Hol, 3
Flow Rate	Lb/Hr.	Ints/IIr.	(Dry)	Lh/Hr.	Mola/Hr.	(Dry)	Lb/IIr.	11015/117	_ (G :A)	Lb/Hr.	Mols/IIr.	(Dry)	Lb/II:	Mols/Hr	_(0tx)
co	478,650	17,068	21.6	7,160	256	0.3			••	2,970	106	3.6	700	25	0.1
ζος	35,200	800	1.0	1.3,704	393	3.6	6,460	147	100.0	113,500	2,581 67	88.3 2.3	28,600 384,000	650 23,997	2.4 81.3
CH ₁	108,840	6,804 54,182	8.6 68.4	246,200 16,362	15,344 8,180	56,1 29,9		••••		1,070	57	1,9	4.306	2,453	8,9
H2 H2 H2S	9,922	354	0,4	9,927	354	î.j		••••		. 11	0.4		9,922	354	1.3
เเรีย	••••									3,740 16B	110	8.ر ۱.0			
còs															
0, 50, c ₂										•			•		••
C ₂ *				121,58á	2,240	6.2									
Total Dry Gas	740,976 1 9 8	79,208 11	100.0	444,728 81,000	27,367 4,500	100.0	6,460 19,400	147 1,080	100.0	121,573	2,924.4 92	100.0	428,128 464	27,479 26	100.0
Total Wet Gas	741,174	72,219		525,728	31,867		25,860	1,227		123,233	3,016.4		428,592	27,505	

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TABLE IV

Section-by-Section Material Balance

250.000.000 SCFD Pipeline Gas from Bituminous Coal

Section 200 Lb/Hr. input: Coal to D-201 1,100,000 Steam to D-201 1,000,000 Air to J-201 4,285,200 N_a HCO3 recycle to F-202 1,235,824 Na_2CO_3 make-up to F-202 18,900 Total 7,639,924 Cutput: 2,089,624 Gas to Section 300 895,000 Melt to Section 600 Flue Gas From J-202 4,655,300 7,639,924 Tota1



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Section 300

Input:				Lb/Rr.
	Gas From Section 200 BFW Quench to D-30i Process Water to E-301			2,089,624 146,980
		•	Total	2,596,604
Ou:Put:				
	Gas to Section 400 Condensate from F-302 Bottoms from E-301			1,851,604 163,000 582,000
			Total	2,596,604
	-			
		Section 400		Lb/Hr.
Inout:				
	Gas from Section 300 Regeneration steam to 0-403			1,851,604
			Total	1,909,204
Output:				•
	Gas to Section 500 Purge gas Gas to Section 600 F ₂ S reacted with iron oxide Waste gas from 0-403	in D-401		741,174 662,168 447,993 260 57,609
			Totai	1,909,204



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		Section 500		
Lagueta				Lb/Hr.
Inout:	Gas from Section 400			741,174
Output:		•		
	Product gas Condensate from F-503 Condensate from F-504 Condensate from F-505			428,592 215,446 96,030 1,106
			Total	741,174
		Section 600		Lb/Hr.
Input:				
	MeIt from D-201 Process water to L-601 Gas from Section 400			895,000 606,690 <u>447,993</u>
			Total	1,949,683
Output:				·
	NaHC03 Recycle to F-202 Solids purge from L-601 Overhead from F-601 Water from L-602 Condensate from F-602 Purge gas		•	1,235,824 ~ 113,640 25,860 31,700 419,426 123.233
			Total	1,949,683

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TABLE V

<u>Utilities Summary</u>

250.000,000 SCFD Pipeline Gas from Bituminous Coal

Steam

420 psia, 449°F

A. <u>Generation</u>

Section			Normal Production Lb/Hr.
200 300 500	F-203 F-301 F-501 F-502	Steam Drum Steam Drum Steam Drum Steam Drum	920,600 233,800 1,304,700 258,000
			2,717,100

5. Consumption

Section		ltem	Normal Consumption Lb/Hr.
200	D-201	Gasifiers	1,000,000
400	J-402	Turbine	167,000
	Miscellaneo	ous Fluor Requirements	22,600
	7-403	Turbine	21,700
	Regeneratio	on steam to D-403	57,600
500	J -501	Turbine	133,100
	J-502	Turbine	13,400
	J - 505	Turbine	18,900
	J - 506	Turbine	211,000
600	J-604	Turbine	20,700
1100	N-1101	Turbogenerator	1,051,100
			2,717,100

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Electric Power

A.	. Gene	eration		Normal Generation
Section		! tem		kw_
200		J-201	Generator ·	76,800
1100		N-1101	Turbogenerator	<u>85,500</u>
				162,300
В.	Cons	sumption		
Section		ltem		Normal Consumption kw
100		L-104	Bradford Breaker	125
		L-108	Hammermills	1,230
		Miscellaneous		145
200		J-203	Pump	161
300		J-301	Pump	41
500		J - 503	Pump	228
_		J ~ 504	Pump	45
500		J-601	Pump	67
		J - 602	Pump	336
		J -60 3	Pump	2,390
	Avail	lable		<u>157.532</u>
				162,300



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Cooling Water

А	. Pro	duction		Name 1 Braduction
Section			Item	Normal Production GPM
1100		L-1101	Cooling Towers	241,970
В	. Con	sumption		Normal Consumption
Section			Item	GPM
200		C-205-a C-205-b	Intercooler Intercooler	18,200 20,400
			Total Section 200	38,600
300		C-304	Cooler	25,400
400		J-402 J-403	Surface Condenser Surface Condenser	11,400 _1.480
			Total Section 400	12,880
500		C-506 C-510 C-511 J-501 J-502 J-505 J-506	Cooler Cooler Aftercooler Surface Condenser Surface Condenser Surface Condenser Surface Condenser	25,800 3,000 -4,530 9,060 610 1,280 14,400
			Total Section 500	58,680
600		C-501 J-604	Condenser Surface Condenser	33,200 1,410
			Total Section 600	34,610
1100		N-1101	Surface Condenser	71,800



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2,947,980

	•	
	Cooling Water Consumption St	
Section	Tītle	Normal Consumption GPM
200 300 400 500 600 1 100	Gasification Shift Conversion Gas Purification Methane Synthesis Ash Removal Offsite Facilities	38,600 25,400 12,880 58,680 34,610 71,800
	Total Consumption	241,970
c.	Cooling Water Balance	GPM
	Recirculated Water Make-up Water from river water pumps	241,370 21,600
	Total Water to Cooling Towers	262,970
	Water Losses in Cooling Towers	21,000
	Total Water to process	-241,970
1	Warm Water returned to river	600
	Recirculated water	241,370
	Boiler Feed Water	~
1. <u>200°F.</u>	15 psia	
A. Section	Production	Normal Production Lb/Hr.
200 300	C-209 BFW Heater C-303 BFW Heater	1,217,980 1,730,000 2,947,980
Section	Consumption	Normal Consumption Lb/Hr.
300 1100	D-301 Quench De-aerators	146,980 2,801,000

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Normal Production

2,801,000

1!, 214°F, 15 psie

Α.	Production

Section		Item	Lb/Hr.
1100	De-aerators		2,801,000
Section	Consumption	Item	Normal Consumption Lb/Hr.
200 300	C-208 C-302	BFW Heater BFW Heater	1,160,000 1,641,002

III. 400°F, 420 psia

A. <u>Production</u>

• • •	- Todac (Toll		Normal Production
Section		ltem	Lb/Hr.
200	c-208	BFW Heater	1,160,000
300	C-302	BFW Heater	1,641,000
			2,801,000

B. Consumption

	<u> </u>		Normal Consumption
Section .		l t em	Lb/Hr.
200	F-203	Steam Drum	949,000
300	F-301	Steam Drum	241,000
500	F-501	Steam Drum	1,345,000
•	F-502	Steam Drum	266,000
			2,801,000

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Boiler Feed Water Balance

A. Losses from System

Section		İtem	Lb/Hr.
200		D-201 Gasifier Steam	1,000,000
		F-203 Blow-Down	28,400
300		D-301 Quench	146,980
1.00		F-301 Blow-Down	7,200
400		Miscellaneous Fluor Requirements	22,600
500		D-403 Regeneration	57,600
500		F-501 Blow-Down	40,300
		F-502 Slow-Down	8,000
		Total	1,311,080
	В.	Recirculation	
		Feed Water Make-up	1,311,080
		Condensate from Surface Condensers	1,636,900
		concensate from our race condensers	1,000,000
		Total Boiler Feed Water	2,947,980
		Process Water	•
	Α.	Generation	
			Norma: Generation
Section		tem	Lb/Hr.
		•	•
1100		Process water pumps	803,690
	ь	Co	
	В.	Consumption	Normal Consumption
Section		item	Lb/Hr.
3EC (1011		I ÇCIII	
300		Water to E-301	360,000
600		Wash water to L-601	443,690
•			
		Total Process Water	803,690

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TABLE VI

INVESTMENT SUMMARY

PIPELINE GAS FROM BITUMINOUS COAL

Jasis: 250,000,000 SCFD of Pipeline Gas

90% Stream Efficiency

Section		Bare Cost *
100 200 300 400 500 600	Coal Storage and Preparation Gasification Shift Conversion Gas Purification Methane Synthesis Ash Removal Offsite Facilities	\$ 4,556,300 56,408,100 3,874,400 19,909,200 10,373,100 4,662,900
	Total Bare Cost Interest During Construction and Contractors Overhead and Profit	\$ 114,275,000 20,112,000
	TOTAL FIXED INVESTMENT	\$ 134,387,000
	Working Capital	•
	30 days Coal Inventory 30 days Carbonate Inventory 30 days Catalyst Inventory Catalyst Charge Accounts Receivable at 11% of	\$ 1,580,000 211,000 73,000 294,000
	Total Operating Expense	3,730,000
	Total Working Capital TOTAL CAPITAL INVESTMENT	\$ 5,888,000 \$ 140,275,000

^{*}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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TABLE 'VII

ESTIMATED ANNUAL OPERATING COST

AND GAS SELLING PRICE

PIPELINE GAS FROM BITUMINOUS COAL

Basis: 250,000,000 SCFD of Pipeline Gas 90% Stream Efficiency

ltem	\$/Year	e/MSCF
Bituminous Coal at \$4 per ton Sodium Carbonate make-up at 1.55¢ per	\$17,410,000	21.1
pound	2,320,000	2.8
Miscellaneous Chemicals	173,000	0.2
Sponge from make-up	58,000	0.07
Methanation catalyst make-up	743,000	0.9
Direct operating labor at \$3.20 per		
man-hour	1,400,000	1.7
Power credit at 5 mills per kwh	(6,240,000)	(7.6)
Maintenance at 3% of bare cost	3,430,000	4.2
Supplies at 15% of maintenance	515,000	0.6
Supervision at 10% of operating labor	140,000	0.2
Payroll overhead at 10% of operating		
Tabor + supervision	154,000	0.2
Generall overhead at 50% of maintenance		
<pre>+ supplies + operating labor + super-</pre>		
vision	<u>2,740,000</u>	3.3
Plant Operating Expenses	22,843,000	27.7
Depreciation at 5% of fixed investment	6,720,000	8.2
Local taxes and insurance at 3% of fixed		
investment	4.030,000	4.9
Sub-total	33,593,000	40.8
Contingencies	672,000	0.8
TOTAL OPERATING EXPENSE	\$34,265,000	41.6
20-YEAR AVERAGE TOTAL REVENUE REQUIREMENT	\$41,500,000	•
AVERAGE GAS SELLING PRICE		50.3

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V. MECHANICAL DEVELOPMENT

A. Accomplishments

1. Environmental Testing of High-Temperature Materials

Oxidation Test #3 continued in progress to the 846-hour mark during this report period. At this point the furnace failed and the test was delayed while awaiting replacement parts. The results to 846 hours were as follows:

- a. Monofrax A (High-purity cast alumina) This sample has been in the reactor the full 846 hours. The average overall corrosion rate on this sample, based on 846 hours, is 0.08 in./year. The sample was still in excellent condition and was returned to the reactor to complete the 1000-hour test.
- b. Monofrax M(High-purity cast alumina with 1.1% SiO₂) This sample also has been in the reactor 846 hours. The average overall corrosion rate, based on 846 hours, is 0.27 in./year. The sample still was in fairly good condition and was returned to complete the 1000-hour test.
- c. Inconel 600 This sample was placed in the reactor at the 500-hour mark. Upon examination at the 846-hour point, the sample was found to have corroded at an average rate of 0.01 in./year based on the 346 hours of testing.
- d. Morganite Triangle R. R. (High-purity alumina) This sample was placed in the test at the 200-hour-mark.
 Upon examination at the 846-hour point the average
 corrosion rate for this sample was found to be 0.15
 in./year based on the 646 hours of test.

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Monofrax A still appears an excellent choice for the intended use. Incomel 600 also shows promise for this atmosphere. The incomel 600 has not, however, been subjected to metallographic examination, and could be subject to microscopic penetration.

2. Gasification_Rate Testing

The 4 1/4 inch reactor has been used for gasificatic rate testing with bed heights of 4" and 8". The results of these tests are reported elsewhere in this report by Process Research as part of their work on gasification rate testing.

3. Mechanical Characteristics Testing

A series of tests was conducted in the 4 1/4" - 1.D. reactor used previously for corrosion and gasification rate testing to determine bed expansion of molten sodium carbonate with various superficial velocities of steam, nitrogen, and carbon dioxide. The effect of carbon level and ash concentration on ped expansion were also explored. Expansion of the bed with continuous coal feed with 7% ash and 4% carbon level in the bed was investigated.

All the above experiments were conducted with the reactor top removed to allow observation of the bed and permit measurement of bed expansion by use of a dip stick.

Figure 5 shows the effect of superficial velocity on bed height of molten sodium carbonate using steam, nitrogen or carbon dioxide as the fluidizing gas. Figure 6 displays the same information as a percent expansion.

It is interesting to note from these curves that nitrogen appears to give a 10 to 20 percent greater bed expansion for a given superficial velocity than either ${\rm CO_2}$ or steam. Due to the obvious inaccuracies of measuring a bubbling bed height by a dip stick method, the data should be taken to indicate only a probable effect of fluidizing gas on bed expansion for a given superficial velocity. All plot points for the above curves are an average of three or more individual readings. Variations in individual readings averaged \pm 10%; however, in extreme cases at the higher superficial velocities, variations of \pm 25% were noted.

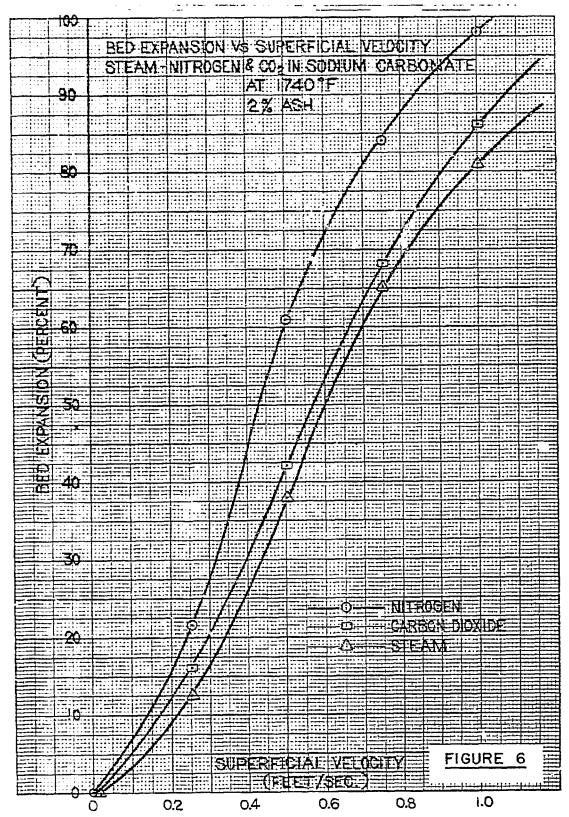
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BED EXPANSION VS. SUPERFICIAL VELOCITY STEAM-NITROGEN & 002 IN SODIUM CARBONATE AT 1740°F -2% ASH NEROGEN CARBON DIOXIDE 0/2 0/4 0/6 0/8 SUPERFICAL YELOGITY (SPV) (FEET/SEC.) FIGURE 5

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Figure 7 shows the effect with time of additions of 1, 2, 3 and a weight percent of coke in a molten sodium carbonate bed being agitated by nitrogen at 0.1 feet per second superficial velocity. The data indicates a rapid increase in bed expansion possibly resulting from liberation of volatile matter in the coke. After approximately four minutes, the bed level returned to the level produced by the agitation gas. A higher superficial velocity of 0.5 FPS was introduced after 10 to 15 minutes of each run to establish its effect with carbon level. No significant variation was noted with carbon content. It therefore appears that, within the range of up to 4 percent carbon, carbon has little effect on bed expansion.

The effect of adding 1, 2 and 4 weight percent of coke to a 7-inch high bed of molten sodium carbonate under simulated gasification conditions is shown on Figure 8. In general, a rapid expansion of the bed occurred followed by a gradual decline. The decline appears more gradual for the higher percent coke addition as would be expected. It is somewhat surprising to note that a greater maximum bed expansion was obtained with the 2 percent than with the 4 percent coke addition. Here again the difficulty of measuring a bubbling bed may explain the difference. Also, the bed may have been pre-conditioned in some manner by the previous additions of 1 and 2 percent coke; although, approximately one hour was allowed between runs to complete gasification.

Figure 9 shows the effect of ash concentration on bed expansion. Ash concentrations of 2, 5 and 8 percent were tested by addition of 4 percent coke under simulated gasification conditions (0.1 FPS superficial velocity N₂ and 0.9 FPS SPV steam). As can be seen, the ash concentration appears to have little effect on maximum bed expansion. However, it does appear to significantly increase the time for maximum expansion to develop and extend the duration of maximum expansion. It is of interest to note that the maximum bed expansion is nearly 240 percent under the conditions of this test.

Figure 10 provides the results of an experiment to determine bed expansion under conditions simulating continuous operation on coal feed at a rate of 15 lbs/hr per cubic foot of quiescent melt. The ash concentration in the carbonate bed was 7 percent at the start of the experiment but probably increased to nearly 8 percent at the conclusion as a result of the continuous coal feed. A 4 percent initial carbon level was obtained by coke addition at the start of the experiment and accounts for the early peak of the plot. Continuous coal feed was started five minutes after the initial coke addition. The plot after 30 or 40 minutes indicates that a bed expansion of about 100 percent could be expected under continuous steady-state conditions.

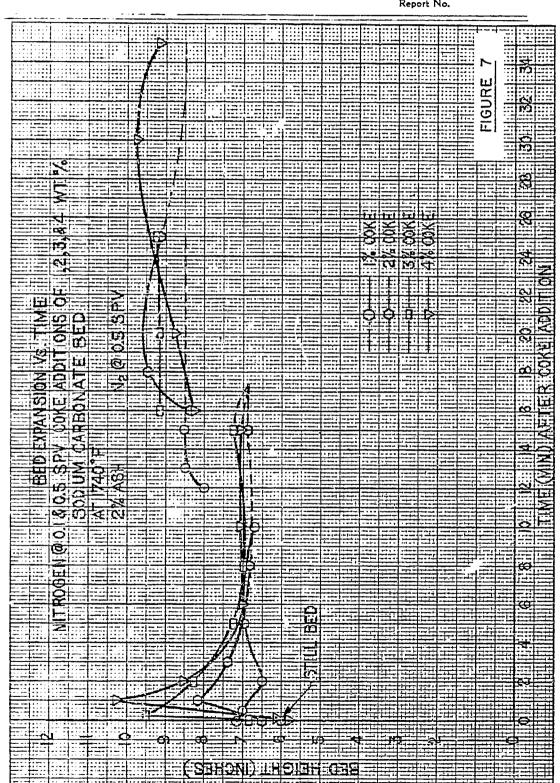
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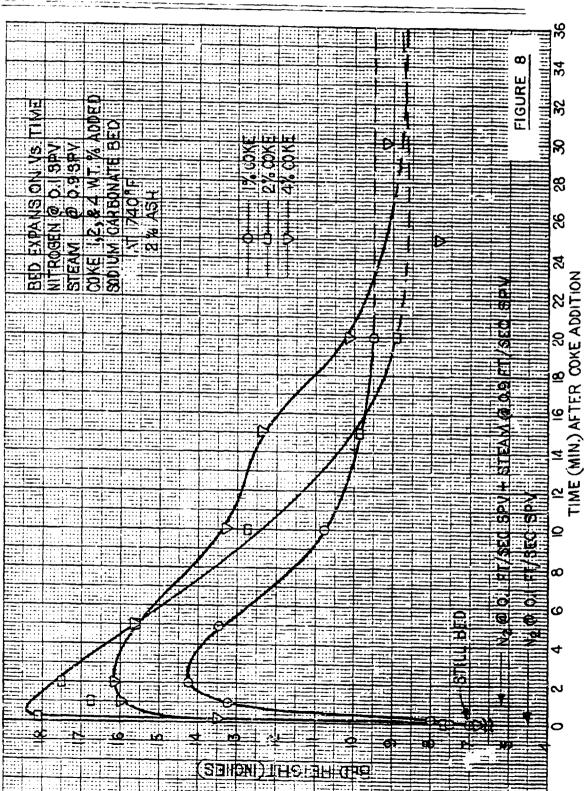
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Projections

1. Environmental Testing of High Temperature Materials

Oxidation Test #3 will be continued to 1000 hours as soon as the required furnace parts are received. Upon completion of this test, another corrosion test will be begun in the same reactor. This new test will, however, be conducted in a reducing atmosphere but without carbon feed, a gas mixture being provided to simulate the gasification conditions. The purpose of this new test will be to investigate the effect of corrosion on joints between blocks of Monofrax "A".

2. Gasification Rate Testing

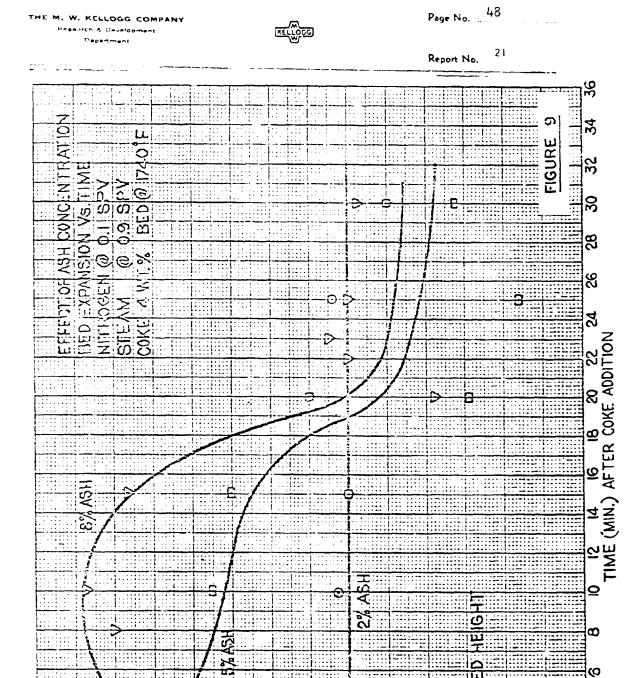
No further rate testing is proposed for the 4 1/4" reactor. Process kesearch is continuing this work in their two-inch reactor.

3. Mechanical Characteristics Testing

Work will continue on the large-diameter reactor and its supports. Delivery of the furnace is expected in July and experimentation should proceed by early August.

A suitable simulated melt material will be sought for future circulation experiments to eliminate the obvious disadvantage of working with molten sodium carbonate at temperatures in the neighborhood of 1800°F.

The 4 1/4-inch 1. D. reactor will be modified to permit experimentation with melt quenching. A model melt quench tower will be designed and constructed. Grinding requirements for the quenched melt and its dissolving rates will be determined in this model to aid in the design of future pilot plant equipment.



BED HEIGHT (FUCHES)

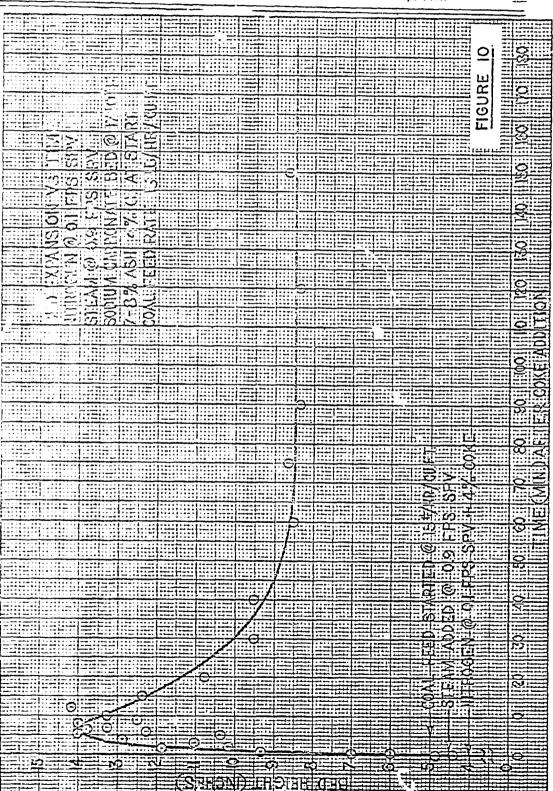
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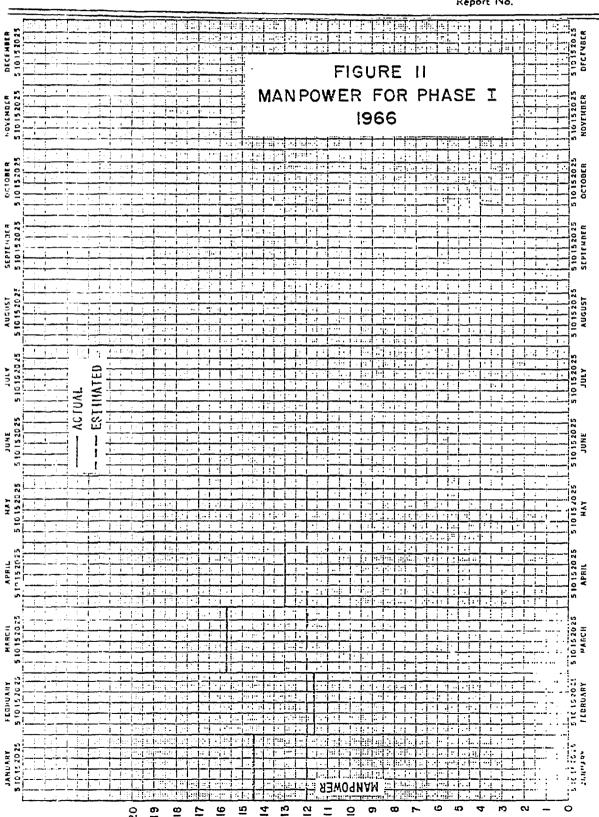
VI. MANPOWER AND COST ESTIMATES

Figure 11 shows the projected breakdown for Phase I for 1966 as well as the actual effort that was made. It can be seen that a 13.0 man-effort was made during April.

Figure 12 shows the expenditures during April. For the month \$22,765 was expanded, not including fee a \pm G & A. The total expenditures through April were \$409,194. Including fee and G & A the total expenditures were \$468,392. This is 78% of the encumber d funds.

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