

MWK-MPR-12

RESEARCH AND DEVELOPMENT DEPARTMENT



DEVELOPMENT OF KELLOGG COAL GASIFICATION PROCESS

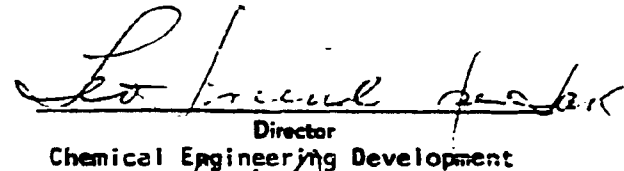
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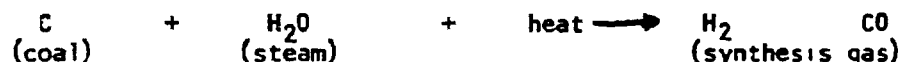
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I. INTRODUCTION

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 AND PROJECTIONS

This monthly summary is the twelfth 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 that are now being studied: process research, chemical engineering studies, and mechanical development.

The experimental program on the gasification of coal was continued. To date some 40 runs have been made exploring the various parameters. The latest runs were made to determine the effect of ash content on gasification rate. In the overall process ash and coal will be present in the melt. The presence of ash in the melt had a significant effect on gasification rate. For a melt with 10% ash there was a two-fold increase in rate over that for ash-free melt.

Up to this time experiments had been made either with anthracite or bituminous coke or coal. This month two runs were made with Renners Cove lignite. It was chosen because of its high sodium content. It was approximately twice as reactive as bituminous coal. Low-pressure batch gasification experiments are practically completed. A review of these data was made in the anticipation of the preparation of revised flowsheet designs. Until the high-pressure reactor, presently under design is commissioned, and gasification rates obtained under design pressure and temperature, they will be assumed to be equal to those obtained at the desired temperature and at atmospheric pressure. It is presently felt that this is a conservative approach.

In the proposed flowsheet coal in the melt is burned to produce the heat required in the gasification step. This month the first combustion kinetic experiments were made. Two runs were completed. The first indicated that the specific combustion rate constant for 10% O_2 in the N_2 feed was 0.37; for 4.2% O_2 , 0.11. The usual first order kinetic plot gave a specific gasification rate of 0.6. The second run using air as feed gas had a specific combustion rate of 0.32. From these initial experiments it can be seen that combustion rates may be half those for gasification. The experiments on combustion rates will be continued during the following months. The effects of the following variables will be studied: percent carbon and ash in the melt, particle size, bed height.



It was concluded earlier that the optimum flowsheet for removing ash from the melt and recovering sodium is one that included quenching and dissolving the sodium carbonate, filtering off the ash and coal, adding carbon dioxide to form sodium bicarbonate that precipitates from the solution, recovering the bicarbonate by filtration and returning it to the gasifier. Based on the experimental data that were obtained in May and June a preliminary process design and approximate cost estimate was made to determine the magnitude of costs contributed by this section of the plant. Sodium carbonate losses are somewhat higher than had been anticipated. Before the experimental data had been obtained it had been estimated that these losses would contribute from 1 to 3¢/MSCF of pipeline gas; present estimates indicate from 3 to 4¢/MSCF. These experiments further indicate that ash removal costs do not vary greatly with ash content. However, at low ash concentration the cost of coal lost may become significant if coal levels are allowed to rise. To firm up these economics additional experiments will be made. Of prime importance are the lowering of the wash water/residue ratio from about 12/1 to 3/1 and studying the effect of coal on the melt on sodium losses.

Since viscosity measurements indicated a marked effect of ash content on melt viscosity, experiments were performed in a 3-inch diameter tube to visually observe the relative effects of liquid viscosity on gas bubble size and gas-liquid contacting. Viscosities corresponding to 10 percent and 20 percent ash in the melt were simulated with glycerol-water and glycerol-Cab-o-sil. The bubble behavior and liquid circulation was so poor with 3700 cp. (20% ash) liquid compared to the 125 cp. (10% ash) liquid that it appears likely that satisfactory operations will require control of ash content of the molten salt to levels of about 10%.

Corrosion test No. 3 of the high temperature materials has been completed after subjecting the specimens to the test environment for approximately 200 hours. Specimens consisted of several cast alloys as well as refractory materials. The specimens are now being cleaned for examination. The two previous tests had indicated that stainless steels were not suitable materials of construction whereas several refractory-type materials were probably acceptable. Based on the results of test No. 3, samples will be chosen for more extensive testing over longer periods of time and at temperatures up to 1850°F.



III. PROCESS RESEARCH

A. Accomplishments

1. Gasification Kinetics

The six runs which employed gasification conditions are shown in Table I. Specific gasification rate is given only for carbon to the oxides and methane since it appears that the gasification rate calculated from reducing power suffers from hydrogen reaction with the reactor walls and is not reliable. In addition the gasification rate constant has been converted into a rate of gasification expressed as pounds of carbon gasified per hour per cubic foot of sodium carbonate when either 10% or 5% carbon is present in the melt.

Specific rate constant as defined in all previous run tabulations is a constant which includes the 2.3 factor for conversion of natural logarithm to logarithm to the base 10. To change the reported k to k' which satisfies $-\frac{dc}{dt} = k'c$, it is necessary to multiply k by 2.3. Or to obtain the rate in lbs./hr/cF at 10% carbon in the melt multiply k by 31.2 and at 5% carbon in the melt multiply k by 14.8

Run J-9815 was an attempt to compare an equivalent amount of Coke II (Island Creek #27 bituminous coal coked at 600°C) to runs using bituminous coal directly. Although a plug in the outlet line caused the run to be terminated before all the coke had gasified, the results of three conditions indicate a gasification rate of about 1.0 as compared to 0.6 for the coal, thus indicating a higher reactivity for the coke. A sample of gas which had been generated in the first five minutes of gasification was analyzed by mass spectrometry. The results, presented in the following table, show a significant amount of methane, some ethylene and only minor amounts of ethane, propane and propylene.

Analysis of 5 min. Gas Sample J-9815

CH ₄	10.7 Mole %
C ₂ H ₄	2.2
C ₂ H ₆	0.2
C ₃ H ₆	0.1
C ₃ H ₈	tr
H ₂	39.3
CO	11.9
CO ₂	17.5
N ₂	18.1



TABLE 1

GASIFICATION OF BITUMINOUS COKE AND COAL, AND LIGNITE IN MOLTEN Na₂CO₃ /1/

Run No. -J-	9815	9816	9817	9819	9821	9822
Date of Run - 1965.	6/29	6/30	7/9	7/19	7/23	7/29
Feed	Coke 11	Bit.	Coal	Renners	Cove	Lignite
% Total Carbon	86.6	81.3	81.3	-----	66.4	-----
% Volatiles	7.6	37.3	37.3	-----	48.2	-----
% Ash	5.8	3.9	3.9	-----	9.3	-----
gms. charged	20.0	20.0	20.0	20.0	20.0	17.9
mesh size	12/20	12/20	12/20	12/20	12/20	12/20
gms. Na ₂ CO ₃	414	414	414	414	414	414
% Ash in Melt /2/	-	10	10	-	-	-
Conditions						
Temperature - °F	1740	1750	1830	1840	1750	1740
% Steam in N ₂	90	90	90	90	90	90
Unit No.	2	1	1	2	2	2
min. to 0% CO	> 25	60	35	30	40	26
Total Run - min.	25	90	75	32	40	55
Results						
% C to CO + CO ₂	64.6	94.0	101.7	97.2	93.9	99.8
% C to CH ₄	0.2	4.8	3.7	4.1	2.0	2.0
% C to tar, etc.	-	6.0	6.0	-	6.0	<2
Total % C	64.8	104.8	111.4	101.3	101.9	103.8
Spec. Gasif. Rate Constant						
Basis CO, CO ₂ , CH ₄ - input	1.06	1.24	2.69	(2.3)	(2.0)	1.44
Basis CO, CO ₂ , CH ₄ - output	-	1.13	2.00	-	-	-
Rate						
lbs. c gasif./hr./CF at 10% C in bed	33	37	84	72	-	45
lbs. c gasif./hr. CF at 5% C in bed	16	18	40	34	-	21
Note	/3/			/4/	/4/	

/1/ Atm. Pres., 0.5 ft./sec. superficial gas velocity, 4-inch quiescent bed height
 /2/ Bituminous coal ash
 /3/ Terminated due to plug in outlet tube
 /4/ Inlet plug between 5-10 min. Replaced inlet and continued run.



Some of these products probably represent devolatilization of the coke without reforming. Bottom feeding through the melt instead of top feeding as well as bed height will have an influence on this composition.

The next two runs employed a carbonate melt which contained 10% bituminous coal ash in order to ascertain the effect of ash content in the melt on the gasification rate of bituminous coal. The runs were made at 1750° F and 1830° F. The gasification rates can be compared in the following tabulation with similar runs made without ash addition.

<u>Temperature - °F</u>	<u>1750</u>	<u>1830</u>
Specific Rate Constant		
With 10% Ash	1.2	2.7
Without Ash	0.6	1.3

The presence of 10% ash in the melt had a very significant effect on the rate, a two-fold increase in gasification rate was observed at the two temperatures. Literature usually implies that the alkali and iron content of ash tend to catalyze rates of gasification, but obviously here the alkali is abundantly present and it must be the iron and/or other constituents of the ash which have improved matters.

Because of the high sodium content of this material, Renners Cove lignite has been evaluated in the gasification unit. The material received from Consolidation Coal Company was screened to give 12/20 mesh material and dried at 110° C for 1.75 hours since it originally contained 32.9% moisture. Considerable difficulty was experienced with making uninterrupted runs. Upon charging the lignite, a large initial burst of gas caused pressure to build up in the inlet system and a momentary loss of steam occurred. This led to salt getting into the 1/4 inch inlet line and producing a plug. By replacing the plugged inlet tube in two runs, it was possible to continue the runs but less reliability can be given to the rate constant. In the final run J-9822 a modification of the water-steam system allowed a continuous standard type of run to be obtained. The results of the runs are compared in the following table with results from runs using bituminous coal.

	<u>Lignite</u>	<u>Bituminous Coal</u>
Specific Rate Constant at		
1740° F	1.4	0.6
1840° F	2.3/1/	1.3

/1/ Interrupted run.



Thus, this lignite was approximately twice as reactive as bituminous coal.

2. Initial Combustion Kinetics

In the first run, the usual 20 grams of Island Creek #27 bituminous coal was charged and 25 minutes of standard gasification at 1750° F was done before cutting off steam and starting combustion. At this point 0.50 moles of carbon were left in the molten salt (1.32 moles charged initially). Combustion feed gas was set at an oxygen concentration of 4.2% in nitrogen. Product gas analysis showed oxygen at about 1.1% coming through the reactor with no carbon monoxide and about 1.1% carbon dioxide for 10, 20 and 30 minute samples. After 35 minutes the oxygen concentration in the feed gas was raised to 10.5% and the run continued for 70 minutes more when the product gas showed 0% CO₂. Product gas analyses after 11, 25, 40 and 55 minutes showed 3.1, 2.8, 1.6 and 0.5% CO₂ respectively, with no carbon monoxide and 7.9 to 7.3% oxygen. A total of 81% of the carbon was accounted for at this time.

The usual first order kinetic plot of all the data gave a specific gasification rate constant of 0.6, a specific combustion rate constant (for 4.2% O₂ in N₂ feed gas) of 0.11 and an approximate specific combustion rate constant (for 10.5% O₂ in N₂ feed gas) of 0.37. This preliminary run established the technique for studying combustion and indicated that full air could be used directly since the rate of combustion appeared to be less than the gasification rate.

The second combustion runs consisted of a charge of 20 grams of 12/20 mesh bituminous coal into the molten salt with nitrogen flowing through the inlet tube. A 25 minute devolatilization period followed, then straight air was passed into the reactor at 0.5 ft./sec. superficial velocity. Product gas was analyzed every 5-10 minutes. No carbon monoxide appeared, only carbon dioxide which ran about 5% for the first 85 minutes then gradually fell off to 0.1% at 180 minutes. The data when plotted did not follow a first order kinetic expression, the rate appeared to increase with time, only the first 40% carbon consumed was linear and indicated a specific rate constant of 0.32.

A coke prepared at 950° F from the Island Creek #27 bituminous coal will be used in future combustion studies of temperature, particle size, etc.



3. Effect of Viscosity of Coke Addition to Sodium Carbonate Melt with Ash Present

An initial viscosity determination at 1700°F with 10.5% Island Creek #27 bituminous coal ash in sodium carbonate melt after two hours of equilibration using CO₂ agitation was only 125 centipoises. This value was too low for precise viscosity measurement. In order to increase the viscosity an additional amount of ash was added to bring the level to 15.8%. After 1.5 hours of mixing with CO₂ agitation the viscosity was 550 centipoises. The addition of 40/60 mesh Coke I (Island Creek # 27 coal coked at 850°C) to give 5% coke and 15% ash in the melt caused the melt to boil up and some of the melt solidified on the cool walls at the top of the vessel. In the 1.5 inch I. D. Inconel tube the viscosity became difficult to determine because the spindle would hit the wall and give an erroneous reading. The best readings indicated 1300 to 2000 centipoises.

A 2 inch I. D. Inconel tube was constructed to eliminate the wall effect on the spindle. A new melt of 15.8% ash, same as above, was equilibrated for about 3 hours at 1700°F with 0.25 ft./sec. carbon dioxide superficial velocity. The viscosity measured 630 cp. Addition of coke, same as above, to give a melt of 5% coke and 15% ash again frothed to the top of the metal tube and some solidified on the cool walls. After it settled back to normal, about 5-10 minutes after addition of coke, the viscosity appeared to be 400-500 cp. A second addition of coke to the melt, now probably 9% coke and 14.5% ash, did not produce any frothing but the viscosity in 5 minutes after addition was over 10,000 cp. It then decreased to 3000 cp in 10 minutes, about 1000 cp in 15 minutes, and 1600 cp from 45 minutes to 90 minutes.

4. Effect of Electrical Potential on Inconel in Molten Salt

Measurement of the resistance of a 1/2 inch wide Inconel strip (16 gauge) immersed 2 inches into molten sodium carbonate contained in an Inconel tube at 1800°F over an 89 hour period appeared to change from 0.2 ohm to 1.2 ohm but the readings of resistance were not very consistent. The strip was removed and the section that had been in the melt appeared to be dark and gritty in appearance from corrosion and partly as if electrochemical deposition had occurred (possible during resistance measurements). This experiment was not definitive and led to the following.



The same Inconel strip was immersed 1 inch into the molten salt at 1800°F and an AC current applied to give 22 amps allowing the change in voltage with time to be determined. The unit was blanketed with CO₂ gas. The initial voltage was 0.98 volts. After 24, 48 and 120 hours the voltages were 1.05, 1.10, and 1.22 volts respectively. The run was terminated at 120 hours. The overall voltage change was 24%. The immersed section of Inconel had a black oxidized appearance but was not pitted.

In order to ascertain the effect of 10% ash in the melt on the Inconel strip under the same current conditions, the addition of bituminous coal ash was made to the melt and agitated with CO₂. The Inconel strip was immersed to a depth of 1 inch. Upon attempting to reset current, arcing occurred which required removal of the inlet tube for CO₂. Previous experience indicated that the ash would remain mixed in the melt without CO₂ agitation. The initial voltage at 22 amps was 2.1 volts. After 15.8, 17.1, and 18.4 hours the voltages were 2.96, 3.15 and 3.55 respectively. At 21 hours the Inconel strip that had been immersed in the ash-salt melt had dissolved.



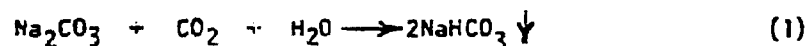
IV. CHEMICAL ENGINEERING STUDIES AND DEVELOPMENT

A. Accomplishments

1. Process and Economic Study of Ash Removal and Sodium Recovery

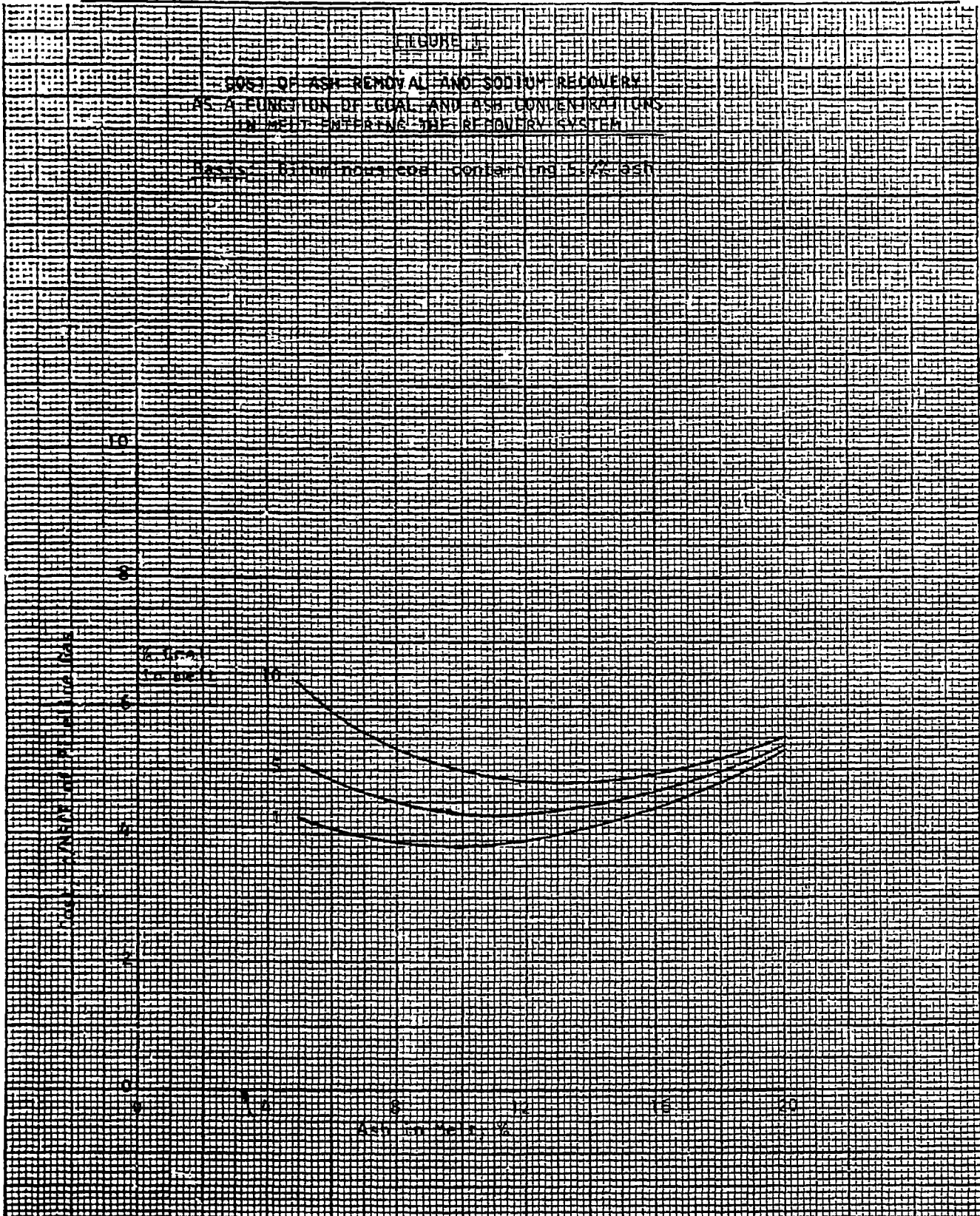
It was concluded earlier (Progress Report No. 5, p. 5) that the optimum flowsheet for ash removal and sodium recovery is one that includes the following steps:

- a. Quench a stream of melt from the gasifier with water (or an aqueous carbonate solution) to dissolve Na_2CO_3 .
- b. Filter off undissolved ash and coal particles.
- c. Contact filtrate with carbon dioxide to precipitate sodium bicarbonate according to reaction (1):



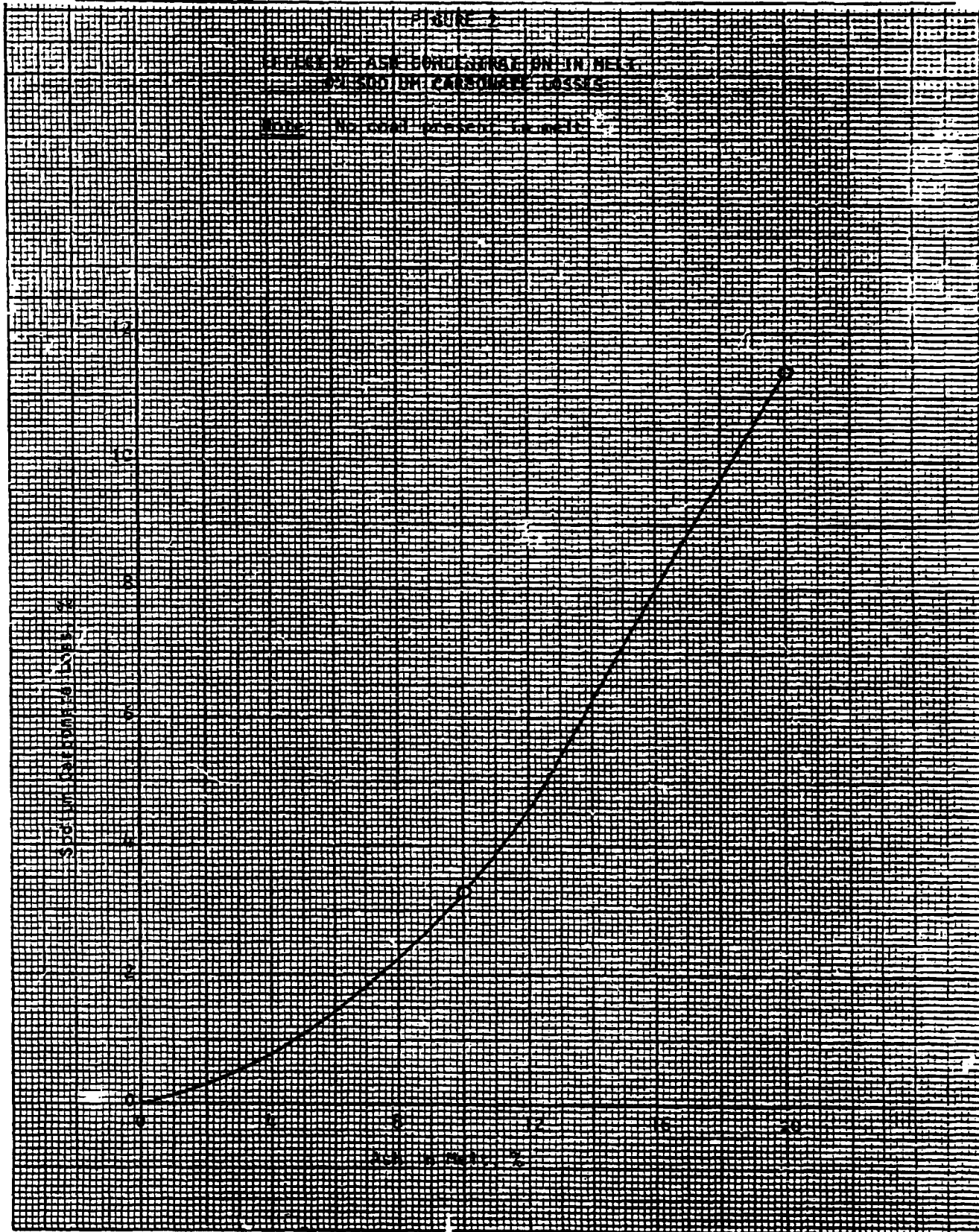
- d. Separate the solid bicarbonate by filtering or centrifuging, and return it to the gasifier.

An experimental program was then carried out to obtain data for design of such an ash removal and sodium recovery scheme. Experimental procedures and results were reported in Progress Reports No. 9, p. 8 and No. 10, p. 9. These data have been used to prepare a preliminary process design and approximate cost estimate to determine the magnitude of costs contributed by this section of the plant (ash removal and sodium recovery) to total product gas cost. The results are shown in Figure 1 for the case of 250 MM SCFD of pipeline gas produced from bituminous coal containing 5.2% ash. Further, these results are based on the relationship between sodium recovery and ash content of melt shown as Figure 2. This relationship is very preliminary at the present time, being based on only the two experimental points shown (actually averages of several experiments) and showing no effect, if there is one, of percent coal in the melt. It is not surprising, though, that sodium loss should increase as the concentration of ash in the melt entering the recovery system rises.



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The effect of two parameters is shown in Figure 1 -- the concentration of ash in the melt and the concentration of ungasified coal. Several observations are in order:

- a. Sodium carbonate losses are somewhat higher than anticipated before these data were obtained (Progress Report No. 5, p. 6), now contributing about 3-4¢/MSCF of pipeline gas instead of 1-3¢/MSCF.
- b. Ash removal costs do not vary greatly with ash content of melt between the 5 and 15% levels, especially if coal content is 5% or less. This occurs because, as ash content is reduced, sodium loss is reduced more than proportionately (Figure 2), thus tending to offset the effect of the larger investment needed.
- c. At low ash concentrations the cost of coal lost becomes significant for the higher coal levels. Even so, at 5% ash and 5% coal, carbon utilization is still greater than 90%.

Further experimental work will be recommended to firm up Figures 1 and 2, to investigate the effect of ungasified coal in the melt, and to extend the range of ash concentrations and wash water/residue ratios used.

2. Review and Analysis of Gasification Rate Data

A review of the experimental gasification data obtained to date has been made in anticipation of the preparation of revised flowsheet designs. The purpose of this review was, hopefully, to gain a better understanding of the kinetics of the catalyzed steam-carbon reaction with a view towards setting reasonable gasification rates for the conditions to be encountered in the subsequent designs.

Data obtained thus far have indicated that the gasification rate is first order with respect to carbon concentration. This is not particularly surprising since for all practical purposes carbon concentration is the only thing which varies within a given run. Such a relationship is typical of batch gasification runs, as can be seen by plotting up any of the runs of Goring, et al. (1) according to a first-order model. However, it is also known that other factors influence

(1) Goring, G. E., G. P. Curran, R. P. Tarbox, and E. Gorin, "Kinetics of Carbon Gasification by Steam," Ind. & Eng. Chem., 44, p. 1051.



the rate of carbon gasification. Partial pressures of steam and hydrogen have been found to affect gasification rate in both fixed- and fluid-bed operations^(2, 3), as have the pressures of CO and CO₂ in a catalyzed, fixed-bed, steam-carbon system.⁽⁴⁾ Indeed, that the present system does not truly obey first-order kinetics has been indicated by the fact that a decrease in steam pressure from 13.2 psia to 4.4 psia results in a lowering of gasification rate by 60%. However, any attempt to determine the exact effects of such parameters on rate must have available data taken over a wide range of conditions. These data cannot as yet be obtained because of the pressure limitations on the present bench-scale unit and the fact that it is capable of running only batch experiments. When the high pressure unit is in operation, however, a program can and will be undertaken to study the effects of these variables and determine what the kinetics of the reaction actually are.

Therefore, until more extensive experiments can be carried out, the gasification rates for the flowsheet studies (at about 400 psig operating pressure) will be assumed to be equal to those obtained at the desired temperature and at atmospheric pressure. It is presently felt that this is a conservative approach since a rather substantial steam pressure dependence has already been noted. On the other hand, the bench-scale experiments have operated at low steam conversions (10-25%), thereby providing the coal with consistently high steam partial pressures. In the commercial designs, where steam conversions must be of the order of 60-75%, the steam pressure will drop off as the gas passes upward through the bed, while the concentrations of possible "poisons" (CO, H₂, etc.) will increase, thus tending to decrease overall reaction rate. However, it is still felt that the net effect of these two factors -- higher operating pressure and higher steam conversion -- will be an increased rate at commercial conditions above those observed thus far.

3. Process Flowsheets

Revised process designs of the coal gasification plant have been started for the purpose of incorporating recent experimental data, a new method of supplying heat for the reaction (by direct combustion of coal in the melt), and perhaps slightly altered operating conditions. These designs will then be combined with necessary gas-treating and methane-synthesis facilities to yield, for the first time, process flowsheets for the completely-integrated, pipeline gas-from-coal plant. Material balances, energy balances, and cost estimates will also be prepared.

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- (2) Gadsby, J., C. N. Hinshelwood, and K. W. Sykes, Proc. Roy. Soc., London, A187, p. 129.
- (3) May, W. G., R. H. Mueller, and S. B. Sweetser, "Carbon-Steam Reaction Kinetics from Pilot Plant Data," Ind. Eng. Chem., 50, p. 1289.



B. Projections

Results of the process and economic study of ash removal and sodium recovery will be used to formulate recommendations for further experimental work in this area.

Process calculations leading to completely-integrated flowsheets for pipeline gas from bituminous coal and from sub-bituminous coal will be continued.

(4) Lewis, W. K., E. R. Gilliland, and H. Hipkin, "Carbon-Steam Reaction at Low Temperatures," Ind. Eng. Chem., 45, p. 1697.



V. MECHANICAL DEVELOPMENT

A. Accomplishments

1. Environmental Testing of High Temperature Materials

Test No. 3 of the high temperature materials has been completed. The test specimens were subjected to the test environment for approximately 200 hours and are currently being cleaned for examination.

Representatives of International Nickel Company examined the corroded sections of the Incoloy reactor and Inconel internals used in Test No. 2 and found these sections to be magnetic (both of these alloys are non-magnetic). Their preliminary opinion is that this points to sulfidation and, perhaps, carbonization. Sample coupons of these materials have been sent to INCO for analysis.

2. Coal Feeding Studies

The results of using sand as the solid material in the solids feed unit appear as Figures 3, 4 and 5. These tests were run at the following conditions:

Pressure in Transport Tube: 1, 10 & 20 psig
Velocity in Transport Tube: 30, 35, 40 & 50 fps
Solids loading, approximately: 0.2 to 1.0 lb. sand/ft.³ of air

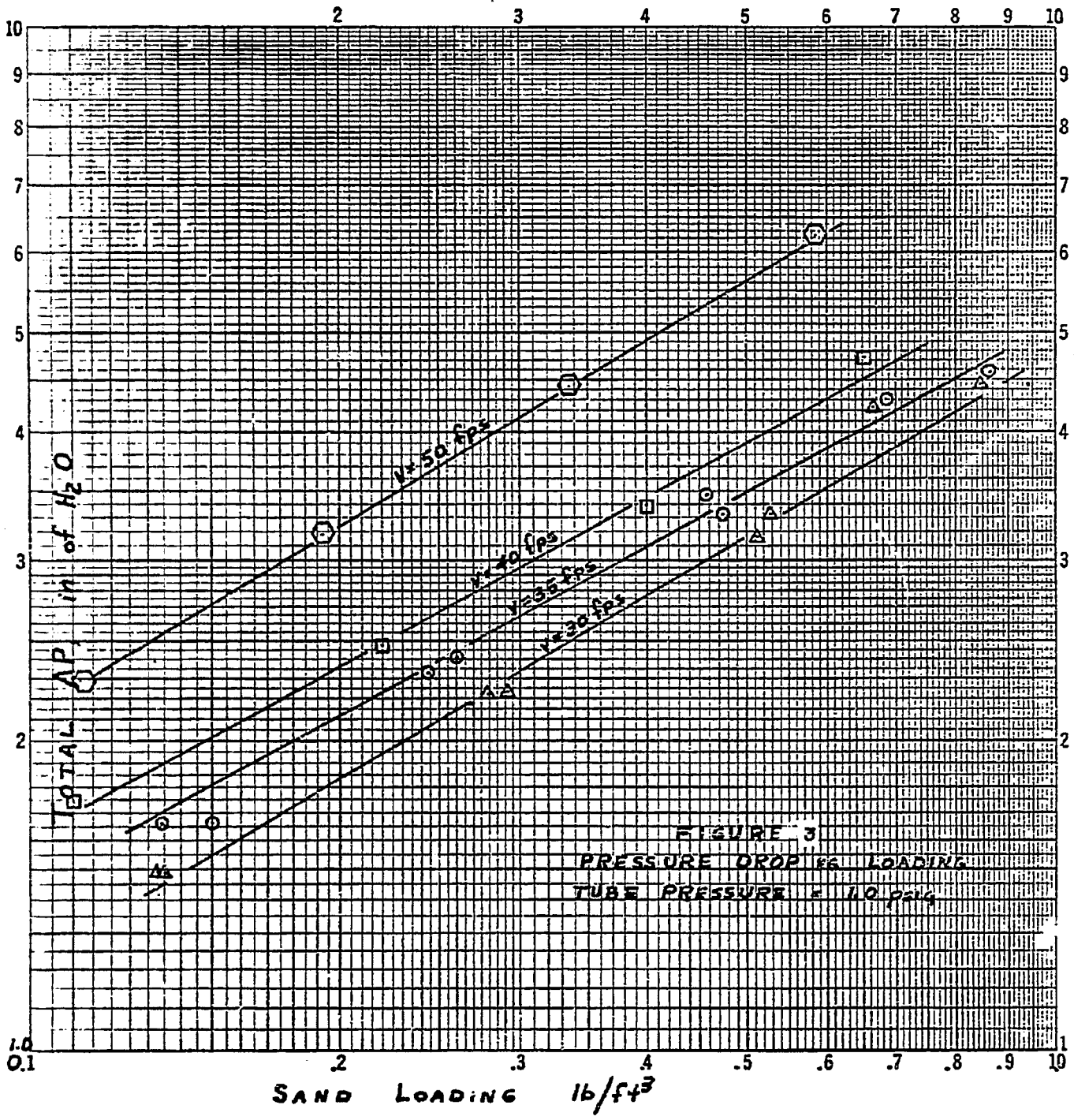
Work is continuing on fabrication of a star feeder device for use with the bench scale reactor. All material required is on hand and the machine work is well underway.

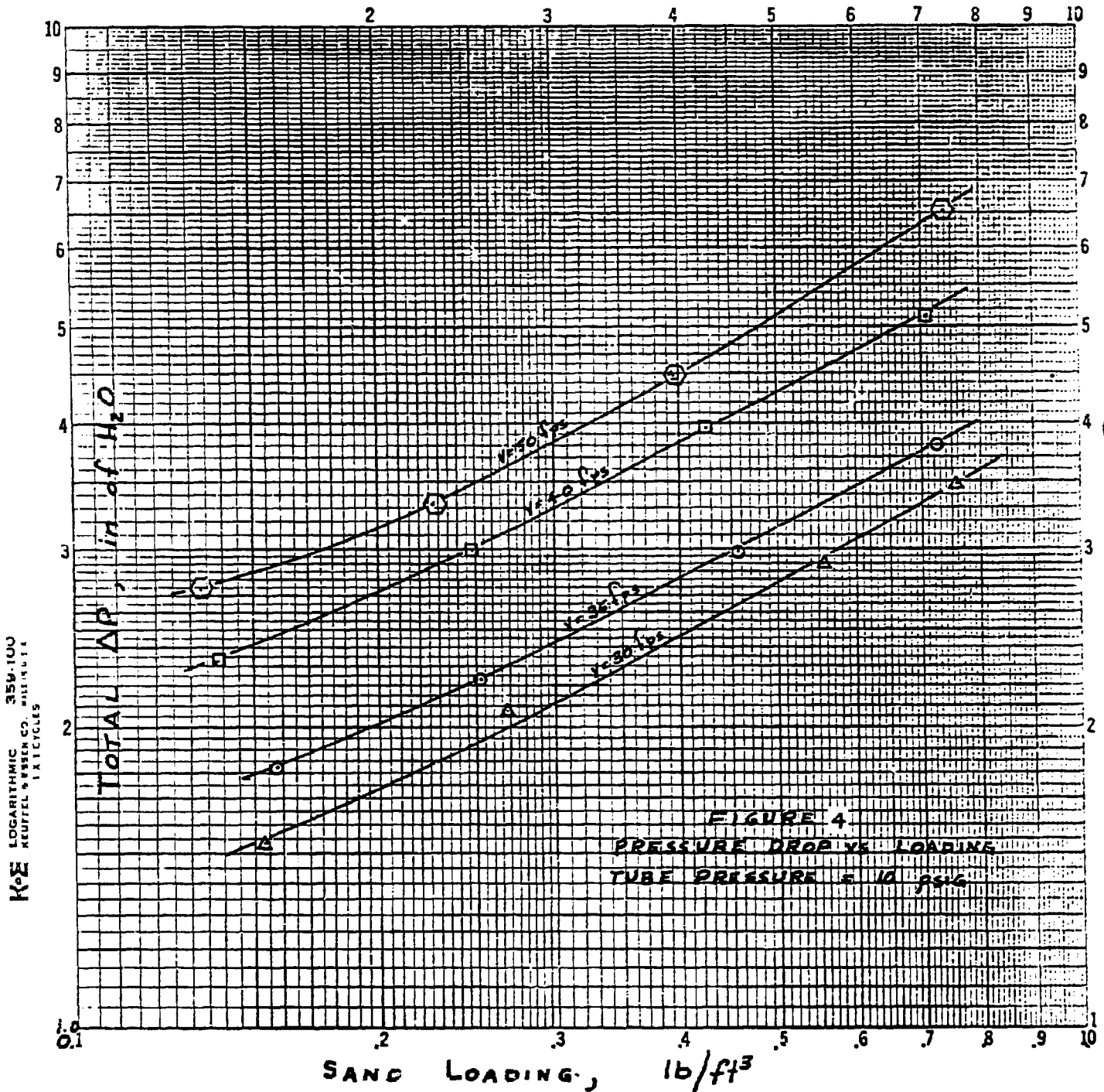
3. Effects of Physical Properties on Bubble Size

In the design of a bench scale reactor to operate at pressures up to 400 psig, it was proposed that the coal and gaseous feed streams be introduced to the melt at a level one inch above the bottom. The Inconel inlet tube, having an outside diameter of 7/8-inch and an inside diameter of 1/4-inch, was located at the axis of the 3-inch reactor to form an annulus for the melt and the effluent gases.

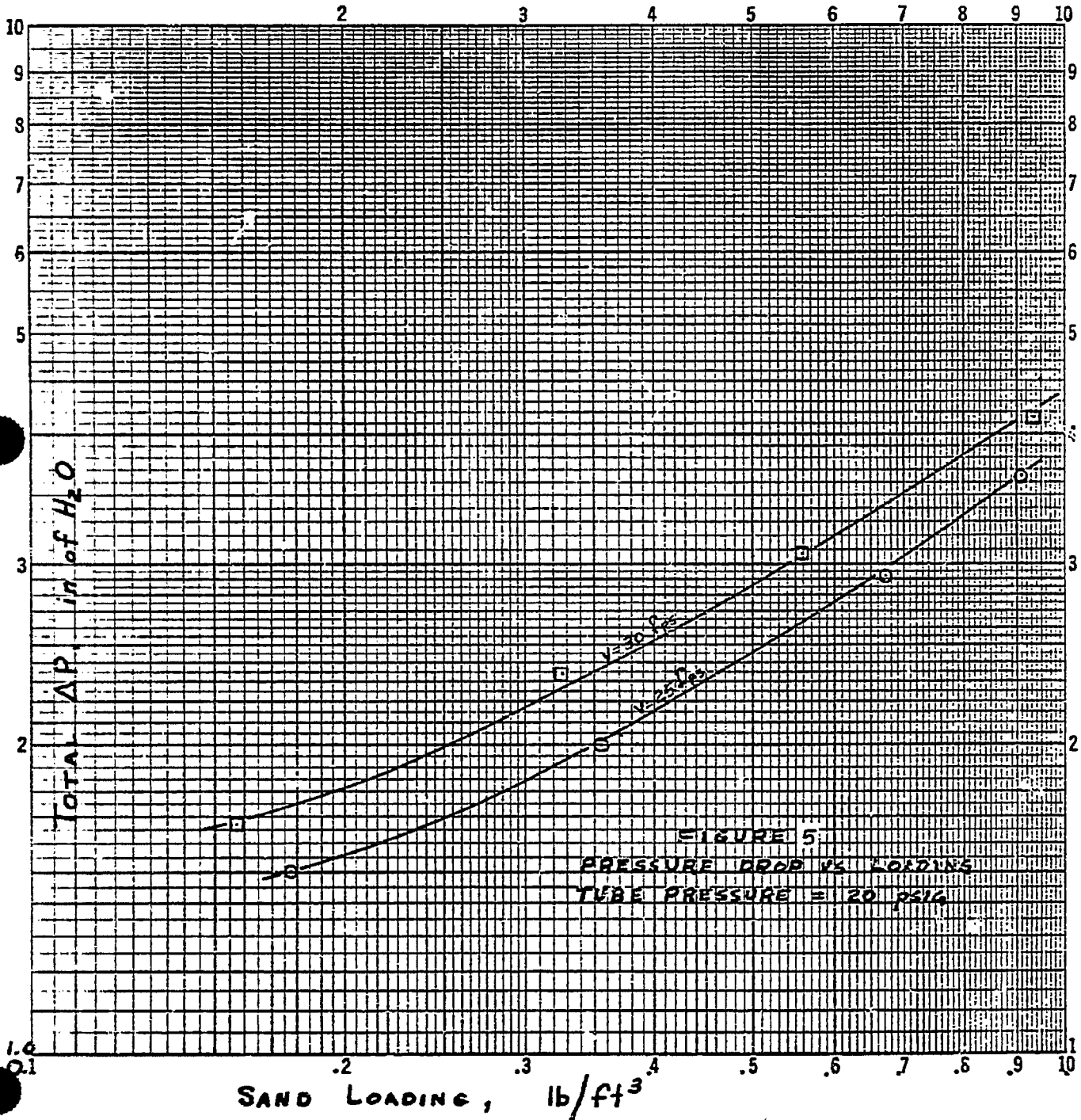


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 1.1 CYCLES



Since viscosity measurements indicated a marked effect of ash content on melt viscosity, experiments were performed to permit visual observation of the relative effects of inlet tube location and of liquid viscosity on gas bubble size and gas-liquid contacting.

A 3-inch diameter by 8 feet high glass tube was set up in the laboratory. Provisions were made for introducing known quantities of air either through a sparger in the bottom, or by means of 1/2-inch schedule 40 pipe located within the glass tube. A pipe cap containing a 1/4-inch hole was screwed to the lower end of the pipe and admitted the air in a downward direction. The performance was tested with the pipe located axially and also when held along the inside wall of the glass tube. Two different liquids were used to simulate the molten salt. A liquid having a viscosity of 125 centipoises (equivalent in viscosity to molten salt containing 10 wt. % of bituminous ash) was prepared by mixing about 88 wt. % glycerol and 12 wt. % water. Another liquid having a viscosity of about 3700 centipoises (equivalent to 20 wt. % ash in melt) was prepared by adding "Cab-o-sil" to glycerol. Small pieces of cork were added to simulate coal particles and permit observation of liquid flow patterns.

Visual observations and conclusions can be summarized as follows:

1. Method of introducing air -- The two-hole sparger containing 1/8-inch holes was the best inlet arrangement. Bubbles were generally smaller, and maintained individuality through a greater length of travel upward. Sausage-shaped bubbles about 4-8 inches long formed about 8-12 inches above the sparger, and grew in length to about one foot as they passed up the tube. Bubble behavior was similar with air introduced through the 1/2-inch pipe either axially located or along the glass tube wall. Agglomeration of air bubbles occurred closer to the air inlet with either arrangement than with the sparger. Large bubbles were shaped like elongated doughnuts with the centrally located air inlet.

Although the sparger appeared to produce better bubble patterns, it was decided to use the centrally located tube because of better prospects for feeding coal continuously with the process steam.



2. Viscosity of liquid -- With any mechanical arrangement of air inlet, velocity of air through the 3-inch tube, and at liquid heights of 6 inches to 3 feet, bubble behavior and liquid circulation was extremely poor with the 3700 cp liquid as compared with the 125 cp liquid. Based on the relative behavior it was decided to make experiments with the high pressure unit with molten salt containing a maximum of 10 wt. % ash.

B. Projections

1. Environmental Testing of High Temperature Materials

The test samples from the completed Test No. 3 will be examined. Based on the results of Test No. 3, samples will be chosen for additional tests.

2. Coal Feeding Studies

Work will continue on the fabrication of the star feeding device for the bench scale reactor. This feeder, when completed, will be tested for operation prior to installation in the bench scale coal feeding system.