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#### • EARCH AND DEVELOPMENT DEPARTMENT -

DEVELOPMENT OF KELLOGG COAL GASIFICATION PROCESS

Contract No. 14-01-0001-380

February 28, 1965

Progress Report No. 19

APPROVED:

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Project Manager

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DIRECTOR

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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 atla, on a commercial scale, to convert coal into pipeline gas at a cost of 5Gc/MSCF or hydrogen at 25c/MSCF. Five raw materials are to be studied -- an anthracite, a high-volatile bituminous coal, a subbituminous 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 take 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:

С	+ H <sub>2</sub> 0	+ heat> Hg		
(coal)	(steam)	(s)	nthesis	gas)

The metessary 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:

- Banch-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.
- Mechanical development -- to find acceptable materials of construction and develop techniques for handling the molten salt and powdered coal.



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Phase I will be concluded by the design of a pilot plant to gasify 24 cons 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.

The last monthly report, Progress Report No. 18, dealt exclusively with an accessment of the technical and economic feasibility of the Kellogg Coal Gasification Process; consequently, no experimental results were reported. Data presented in this report are, therefore, those obtained during January and February.



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### II. SUMMARY

This progress report is the nineteenth 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 restarch, chemical engineering studies, and mechanical development.

The effect of reactor pressures up to 60 psia on the rate of gasification of pituminous coal and coke was investigated in the 2-inch ID Inconel reactor. This was done in the presence or absence of ash and for various levels of the reactowing variables: superficial linear velocity, reaction temperature, and interacted sait depth.

The twenty experiments currently being reported, along with seventeen reviously reported, showed the following results over the ranges studied:

1. Higher pressures yield substantially higher specific rate constants at least up to about 4 atmospheres. The magnitude of the increase is about 60% to 80% in going from 1 to 4 atmospheres.

2. Increase in superficial velocity leads to an increase in the specific casification rate constant.

3. In the presence of ash, the specific gasification rate constant we cases.

4. As the depth of the unaerated salt is increased from 4 inches to 8 irres. however, the specific rate constant decreases by some 20%.

The last three results are believed to represent differences in the efficiency pimixing steam, carbon and melt in the reactor. Further experiments are planned in an attempt to illuminate these effects.

It is encouraging, however, that an overall gasification rate of 39 pounds posified per hour per cubic foot of salt can be projected from the present data for operation — at 1840°F, 4 atmospheres, 8-10% ash, 4 inches of salt depth, and 4% coke in the melt.

The experimental results at 1840°F are less consistent than at 1740°F because the reaction occurs so rapidly that precision is reduced. Nevertheless, it espears that the specific rate constant is increased by a factor of about 1.8 upper to the 100°F rise in temperature. The operating conditions and



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Tate constant obtained by extrapolation of these experimental data are compared below with the values used in the most recent commercial gasifier design:

Parameter	Flowsheet Design	Experimental Results
Feed material Gasifier temperature, °F Avg. steam partial pressure, psia Ash concentration in melt, % Carbon concentration in melt, % Superficial gas velocity, ft/sec Eed depth	Bituminous coal 1830 250 8 4 1.2 15 feet	Bituminous coal 1840 40-50 8 4 (initial) 1.0 4 inches
Specific rate constant, k	2.3	3.3 (extrap.)

This summary shows that the major difference between the design case and experimental results to date is in depth of bed employed. The importance of continued experimental effort to elucidate the observed effect of bed depth is thus re-emphasized.

Data on salt carryover in gasifier effluent have been collected in recent experiments, but have not yet been correlated to present a clear picture. It is hoped that further data will aid in clarification.

Work continued toward the objective of assembling base-case "process packages" for the conversion of bituminous coal and subbituminous coal into 250 MM SCFD of pipeline gas. Each package will include process flowsheet, material balance, utilities summary, capital cost estimate, and calculated pipeline gas production cost. These base-case designs will then be used to determine the effect of new experimental findings or assumptions on process economics.

Process flowsheets for proposed pilot-plant and pre-pilot units were prepared to assist in deliberations concerned with the scale of operation for the next stage of the development program.

Testing of materials of construction in the very corrosive reactor environment is continuing. Corrosion test #8 was run for 260 hours at 1840°7 under gasification conditions. Several high-purity aluminas, several zirconias, and Haynes Alloy 150 were tested. Only the aluminas



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produced by General Electric and Mc Danel passed the test satisfactorily. Monofrax  $\underline{A}$  was not tested.

Oxidation test #2 was run for 135 hours at 1840°F in a simulated combustion atmosphere. Samples of high-purity alumina by Norton, McDanel, and Harbison-Carborundum were tested. Only the Harbison-Carborundum material (Monofrax A), the only high-purity alumina that is cast in the molten state, passed the test. Its measured corrosion rate was 2 very satisfactory 0.046 inches per year.

Oxidation test #3 at 1840°F in a simulated combustion atmosphere is now in progress. Specimens being tested include four high-purity aluminas, an industrial sapphire, and an alumina-zirconia-silica mixture. The samples were removed for inspection after 85 and 200 hours. Monofrax <u>A</u> is in excellent condition, with a maximum corrosion rate of 0.05 inches per year. Monofrax <u>A</u> continues to be the best material tested for the intended service.

The 4 1/4-inch ID corrosion test reactor has been modified to run gasification rate tests at atmospheric pressure. This larger reactor will tend to minimize wall effects and should permit investigation of the effects of bed height and top vs. bottom feeding of coal. These results will be compared with those obtained in the 2-inch diameter unit.



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#### III. PROCESS RESEARCH

#### A. <u>Accomplishments</u>

Data for all the gasification runs performed since Report No. 17 are presented in Table I. The main object of these runs has been to elucidate the effect of steam pressure up to 60 psia on the kinetics of gasification with the hope that extrapolation to commercial design pressure of 400 psia can be made.

The initial attempt under pressure was made to obtain four runs at 1840°F and 0.5 ft/sec superficial gas velocity at approximately 1, 2, 3 and 4 atmospheres steam pressure. Two of these runs were below par in weight balance, apparently due to some coke sticking to the metal of the inlet coke hopper. Additional electrical heating was installed to eliminate this problem. Two runs were also made at 1 ft/sec velocity to take advantage of the higher reaction rates previously observed (Report No. 16). Infortunately, the runs at 1840°F took place so rapidly that it was felt accuracy was lowered and variability was enhanced. Thus, the runs were duplicated at 1740°F to obtain a more reliable index of the effect of steam pressure on the gasification kinetics. These runs will be discussed first.

The 1740°F runs in Table 1, as well as all the earlier runs using bituminous coal and coke il (prepared by heating bituminous coal to 600°C) at this temperature, have been used to obtain Figure 1, where the effect of steam pressure on the specific gasification rate constant (output basis) has been plotted. Notably the results line up extremely well and give excellent correlations for three sets of conditions.

First of all, normal data for a 4-inch quiescent bed depth and 0.5 ft/sec superficial gas velocity have been correlated from 4 to 60 psia of steam. Next come data from runs with a 4-inch quiescent bed using either no ash in melt at 1 ft/sec velocity or 8 to 10% ash in melt with 0.5 ft/sec velocity. The last correlation is for an 8-inch bed depth with 0.5 ft/sec velocity and no ash present. The correlation with ash present (ash is not usually present in experimental runs because of corrosion and

TABLE	I
	Ξ.

# GASIFICATION OF BITTRIINOUS CORE IN HOLTEN NA2CO3 (1)

Rua NoJ- Date, 1966	9924 173	9925 175	99 <b>2</b> 6 177	9927 1/11	9928 1713	992 <b>9</b> 1/17	9930 1/19	9931 1/21	9932 1725	9933 1/27	9934 271	9915 273	9936 277	9917 2/9	9938 2711	993) 2/15	9940 2718	9077 2/21	9978 2/23	9979 2725	
Feed ? <b>1</b> 9 <b>tal</b> Carbon ? Volatiles ? Ash	******				- 93.2 - 0.6																
gas, charged	18.5				19.0									38	38	J8	19	19	19	19	2
mesh size NapCOJ-gma. fedht inches Ash - gma. A in melt 7. C in melt initially	-7				- 414								>	828 8	828 R	729 8 66 7.9 4.2	364.5 4 33 7.9 4.2	161 4 7,9 6,2	161 4 7.9 4.2	364.5 4 33 7.9 4.2	
Conditions Temp *F	1845	1850	1850	1850	1.755	1740	1740	1740	1740	1740	1840	11/0	1.04.0	13/0							
Pressure - psta Stean Pres.,psta	60.7 55.8	31.2 28.9	28.9 26.8	14.7 13.5	14.7 13.5	45.5	61.5 55 <b>.8</b>	30.2 27.5	29.7 27.6	14.7	14.7	1740 14.7 13.5	1840 44.0 39.8	1740 45-7 41.3	61.7 56.6	45 41	44.6	60.5	45.5	60.6 55.7	
Gas in Sleam Ft/sec, Stm. & Gas Minutes to 0% CO Total Run - minutes ce N20 in/hr. ce N2 in/hr.	0.52 25 35 768 1692	1.04 25 35 768 1692	0.51 45 55 384 846	1.05 35 45 384 846	- N2	0.50 45 55 576 1269	0.51 35 45 768 1692	1.01 35 45 768 1692	0.50 65 75 384 846	1.04 55 65 384 846	0,56 60 65 192 423	0,54 25 102 423	0.53 25 35 576 1 <b>2</b> 69	0.51 55 65 588 1269	0.48 55 65 768 1692	0.5 	0,50 45 45 576 1269	0,52 45 45 797 1552	0.51 55 55 587 1206	0.50 35 45 787 1/441	
Results Total 7 C to Goxides Gault, Rate Const	95. <b>)</b>	. 80. 1	103	89.4	99.4	104. L	103,2	104.6	115.7	99.2	LDO. L	68.0	97.6	102.1	105.7		98.L	89.6	89.9	101.6	
Banik Tuput Banis Dubput Rate-1bs, C/hr/CF	1.80 1.93	1.64 1.96	1.76	1.44 1.64	0.92 0.92	1.17 1.13	1.22 1.20	1.60 1.64	0.98 0.87	1.03	1.26 1.26	1.22 _	2.31 2.40	0.88 0.86	1.06 0.98		1.76 1.78	1.24 1.48	1.09	1.84 1.82	10 V 4 4
(40%C in bed (44%C in bed Salt Carryover - gas, Notes	60 23 6.5 (2)	61 23 9 (2)	53 20 25	51 19 12,2	29 11 22	)5 13 20.5	)7 14 8,9	51 19 8.8 (3)	27 10 21.4 (3)	)2 12 16,6 (3)	39 15 23,8	38 14 (4)	75 28 7.7	27 10 14.0	31 12 0.5	 (5)	56 21 10.9	46 17 6,5 (6)	41 16 6.4 (6)	57 72 6.2	90 No. 19

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Notes:

(1) Island Creak #27 bitiminous coal coked at 950°C to give coke 1V; 2-inch ID Inconel reactor; steam prehented to 700-750°F.

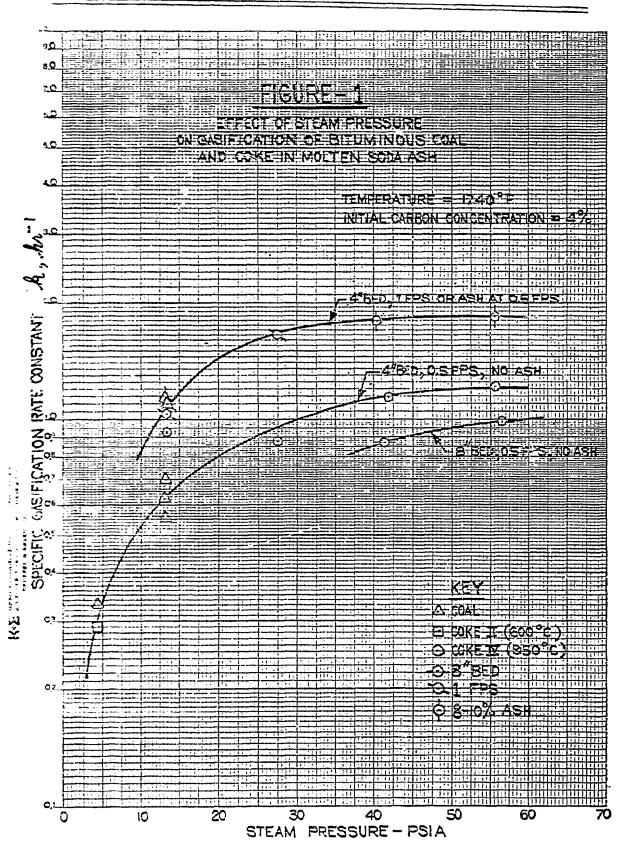
(2) Some of coke charge stuck in valve and hopper inlet system.
(3) Distribution of salt carryover taken, see body of report.
(4) Exil plugged, run terminated.
(5) Unit plugged from frothing over of molten salt when coke charged.
(6) Reused molten salt from previous run, small makeup of Na2CO3ndded.

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reactor cleanup difficulties) represents commercial operation more closely than the others do. In all runs the contentration of carbon was 4% at the start of the run.

The results shown in Figure 1 at 1 to 4 atmospheres steam pressure are reproduced in the following table.

Gasification Rate Constant - k

Steam Pressure	-0.	SFPS-	4'Bed, 1FPS or
atm	<u>4" Bed</u>	<u>8" Bed</u>	8-10% Ash at 0.5775
1 -	0.67	-	1.15
2	0 <b>.9</b> 7	-	1.67
3	1.15	0.89	1.81
4	1.21	0.99	1.83

The kinetic constant is improved by a factor of 1.6 - 1.8 by going from atmospheric pressure to four atmospheres. It does appear that pressures above 4 atmospheres will have very little effect upon the kinetics. However, one verification point at 10 atmospheres pressure appears experimentally possible in a new Inconel reactor and has been placed on the agenda. At a carbon concentration of 4%, the 1.8k represents about 22 lbs. of carbon gasified per hour per cubic foot of melt, a very respectable figure at only 1740°F.

One point on the curve, coke IV at one atmosphere and normal conditions (0.92k), appears to be definitely out of line and may have been a result of some incompletely washed-out ash left in the reactor from a previous run. Five other points which averaged 0.63k indicate the above point is probably in error. Two other runs, 9977 and 9978, were made with melt from the previous run and gave rate constants below the top curve. Analysis of the data indicated uptake of carbon dioxide by the meit in the initial period decreased the value of the rate constant. This effect was caused by loss of carbon dioxide from the carbonate of the melt during heating and cooling with a nitrogen sweep to keep the inlet clear of salt. Uptake of carbon dioxide then occurred as it was generated early in the gasification period.

It is interesting to note that the top curve in Figure 1 shows that an increase from 0.5 to 1 ft/sec velocity not only equals the effect of adding ash to the melt at 0.5 ft/sec, but gives an increase in rate constant by a factor of 1.5 - 1.8. This increase in the presence of plenty of excess steam lends strong support to the proposition that



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improved mixing of the coke and steam is the underlying reason for the improvement. The 20% drop in rate constant shown for the 8-inch bed from that for the 4-inch bed at 0.5 ft/sec is believed to be due to the greater difficulty of mixing in the higher bed at the same 2-inch diameter. An attempt to employ an 8-inch bed with ash present to improve mixing failed because the salt frozhed up upon charging the coke and plugged the cutlet. This work has led to the conclusion that higher bed heights must be investigated in larger-diameter reactors before proceeding to construction of a pilot plant.

All gasification results at 1840°F have been plotted in Figure 2 against steam pressure. It is immediately apparent that the results are not as consistent as those at 1740°F (Figure 1). Nowever, the results are in general agreement. The slope of the curve at higher pressures is much less certain than at the lower temperature. This of course reflects the difficulties of kinetic analysis at high gasification rates. If one assigns the factor of 1.5 as obtained from Figure 1 at 60 psia for the 4-inch bed with ash present over the non-ash result, then at 1840°F the rate constant should be about 3.3 at 60 psia with ash present. This corresponds to a very sizeable gasification rate of 39 lbs./hr./cubic foot of bed at 4% carbon concentration.

Additional data have been collected on the salt carryover problem. These data have not as yet been correlated to present a clear picture. It is hoped this can be done for the next progress report.

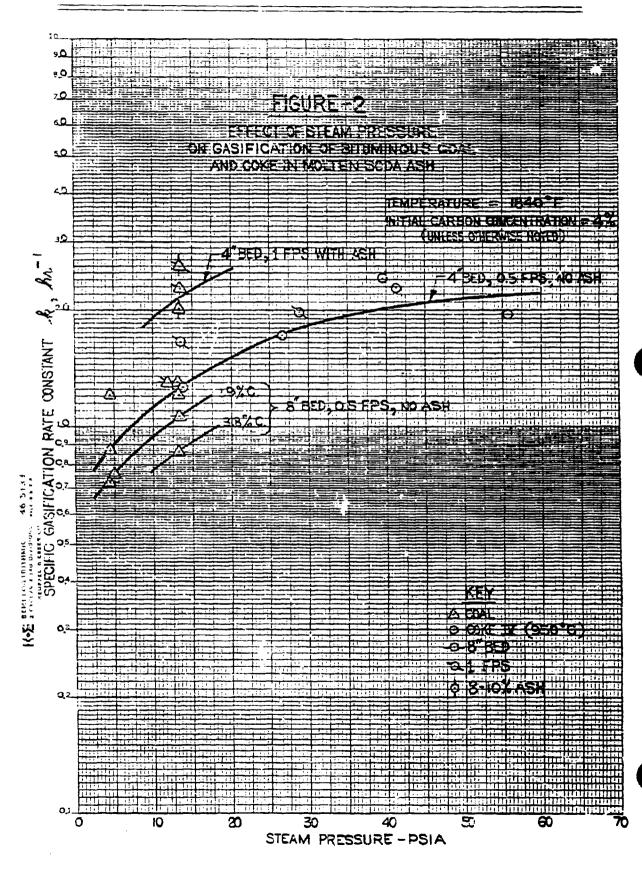
### 5. <u>Projections</u>

The Process Research Group will cooperate with the Mechanical Development Group to obtain data in a four-inch diameter reactor at two levels of bed height. It is hoped these data will furnish some insight into the effect of reactor diameter, bed height and point of coal entry to be used in designing larger equipment. THE M. Y. KELLOGG COMPANY Refresh & Development Department

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### LV. CHEMICAL ENGINEERING STUDIES AND DEVELOPMENT

### A. Accomplishments

The immediate objective of this effort is to prepare a base-case "process package" for the production of 250 MM SCFD of pipeline gas from (1) bituminous coal and (2) subbituminous coal. These packages will be based on recent experimental data, plus assumptions where necessary, and will consist of:

- 1. Process flowsheet
- 2. Material balance
- 3. Utilities summary
- 4. Capital cost estimate
- 5. Computation of gas production cost and selling price.

Working from these base-case designs, the effect of new experimental findings or different design assumptions can be evaluated officiently.

The process flowsheet for the gasification and ash removal sections of the pipeline-gas-from-bituminous-coal plant was completed. Instrumentation and control of the major process variables associated with gasification, combustion, and ash handling during normal operation were studied. Equipment was sized for these sections, as well as for the rest of the process facilities -- shifting, gas purification, and methanation.

Naterial and utilities balances were revised for a commercial plant producing pipeline gas from subbitumineus coal. New equipment specifications for process plant and offsite facilities were prepared for cost estimating purposes.

Two other flow diagrams were prepared for the gasification and ash removal sections of the process:

- 1. A proposed pilot plant sized to meet the contract requirement "of a minimum capacity of one ton of coal per hour".
- 2. A possible pre-pilot unit (no. 2) which could serve as an alternative to the 5 1/2"-ID, electrically-heated reactor (pre-pilot unit no. 1) as the next experimental step in the development program. Size of the gasifier for this unit was set, somewhat arbitrarily, at 12" ID, resulting in a nominal coal rate of about 1/4 ton per hour.

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These flowshects were prepared for two purposes:

- To assist in identifying the problems that must be solved and the uncertainties that must be resolved in the development of a successful commercial process, and in reviewing at what stage of the program these problems should be attacked.
- 2. To assist in the preparation of a preliminary mechanical design and cost estimate for pre-pilot unit no. 2 so that the merits of units no. 1 and 2 may be compared.

Equipment vendors and design firms were consulted in connection with the facilities needed for receiving and storing coal, and preparing it for gasification. Several systems were recommended. Valuable assistance was obtained in estimating capital costs for these sections of the plant.

### B. <u>Projections</u>

### 1. Bituminous Coal

A process flowsheet will be prepared for the entire plant, from coal preparation to product gas. Utilities consumption will be estimated and the size of offsite facilities will be determined. The capital cost estimate will be started.

2. Subbituminous Coal

The economic effect of quenching the hot effluent gas from gasifier and combustor to remove coal tars and entrained melt -if this should prove necessary -- will be investigated. The effect of varying the gasifier operating temperature will also be studied.



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### V. MECHANICAL DEVELOPMENT

## A. Accomplishments

#### 1. Environmental Testing of High-Temperature Materials

Corrosion test #8 was completed during January. This test, conducted in the reducing, or process, atmosphere, lasted approximately 260 hours and included the following samples:

a.	Norton 5995	High-purity	aiumina	•
ь.	Haynes 150	28 Cr, 50 C	o, 20 Fe	
с.	Lucalox	High purity	elumina	(General Electric)
d.	Zirconia Y-1027	Yttria-stab:	ilized z:	irconia
e.	Zirconia Y-1235	11	11	**
f.	Zirconia Y-1484	FT	T1	18
8.	Zirconia Y-1485	11	11	f 1
h.	McDanel AP-35	High-purity	alumina	liner for the
		test reactor	Ξ.	

The results of this test are as follows:

- a. Norton 5995: All three samples completely vanished.
- b. Haynes 150: All three samples recovered, but all severely corroded. The sample in the vapor space was in the best condition, but wasn't deemed satisfactory for this service.
- c. Lucalox: All samples suffered mechanical damage and were found to be in fragments after the test. The corrosion attack, however, was very slight, 0.052 in/yr (average).
- d. Zirconia Y-1027: There were only two samples of this material in this test. The one in the vapor space was still intact, but appeared badly corroded and crumbled easily in the fingers. Calculated corrosion rate was 0.254 in/yr. The second specimen (from the liquid-vapor interface) failed so completely that only fragments remained.

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- e. Zirconia Y-1235: No samples recovered.
- f. Zirconia Y-1484: The samples from the vapor and the liquidvapor interface were recovered. The one in the melt vanished. Both samples that were recovered had increased slightly in size, indicating a phase transformation which was not controlled by the yttria stabilizer.
- g. Zirconia Y-1465: Only the sample from the vapor space was recovered. The comments from "f" above apply.
- h. McDanel AF-35 Liner: The liner appeared in good condition except for mechanical damage from thermal shock (the tube cracked again). It is not possible to determine a corrosion rate for the liner, but the effect of corrosion seems to have been very slight.

A second combustion atmosphere test (Oxidation test #2) was also completed during January. This test ran for approximately 135 hours and included samples of:

- a. Norton 5995 High-purity alumina
- 5. Monofrax A
- c. McDanel AP-35 Central tube and liner of high-purity alumina.

The test results were:

- a. Norton 5995 completely vanished.
- b. Monofrax a in very good condition with a corrosion rate of 0.046 in/yr.
- c. McDanel AP-35. The central tube again thinned out to failure, and the liner cracked due to thermal shock.

Oxidation test #3 has been in progress during the remainder of this report period. The testing was interrupted at 85 and 200 hours to obtain intermediate corrosion rates. Testing included the following samples:

- a. Monofrax A (High-purity cast alumina).
- b. Monofrax M (High-purity cast alumina w/1.1% 5102) 0-200 hours.
- c. Norton 6040 (Bonded high-purity alumina bubbles) 0-85 nours.
- d. Corhart ZAK 501 (49% Al203 34% Zr02 15% Si02) 0-200 hours.
- e. Industrial Sapphire replaced Norton 6040 at 85 hours.
- f. Morganite Triangle RR (high-purity alumina) replaced Corhart



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### ZAK 501 after 200 hours.

The results of this test after 85 hours were as follows:

- a. Monofrax A: Sample recovered on the speciman rack in excellent condition. A maximum corrosion rate of 0.01 inches per year was obtained.
- b. Monofrax M: Recovered on the specimen rack in good condition. One corner was broken off by mechanical shock during recovery; however, this did not reflect a shortcoming of the material. A maximum corrosion rate of 0.25 inches per year was obtained.
- c. Norton 6040: The specimen was not recovered on the rack. However, a badly deformed mass of what appeared to be this material was found in the melt remains. Unexposed, this material lacked mechanical strength and was quite porous, which probably led to its early failure.
- d. Corhart ZAK 501: Recovered on the specimen rack in good condition. Some rounding of the edges was noted. A maximum corrosion rate of 0.28 inches per year was obtained.

The results of this test after 200 hours were as follows:

- a. Monofrax A: Recovered on the specimen rack in excellent condition. A maximum corrosion rate of 0.05 inches per year was obtained (measured from time zero).
- b. Monofrax M: Recovered on the specimen rack in good condition. A maximum corrosion rate of 0.25 inches per year was obtained.
- d. Corhart ZAK 501: Specimen not recovered at this time; however, a possibility exists that it remains in the melt, which was not removed for the 200-hour inspection.
- e. Industrial Sapphire: Recovered on the specimen rack in good condition, although cracked by either thermal shock or differential expansion of the specimen rack on withdrawal. A maximum corrosion rate of 0.095 inches per year was obtained based on the 115 hours of exposure time.

Monefrax A continues to be the best material tested for the intended service.

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### 2. Gasification Tests

The 4-1/4-inch I.D. corrosion test reactor used for the reducing-atmosphere tests has been modified to run gasification rate tests at atmospheric pressure. This larger-diameter reactor will tend to minimize wall effects, and will also allow the effects of bed height and top vs. bottom coal feeding to be explored in relation to results obtained in the 2-inch diameter unit. An exploratory run with a 6-inch bed height has been completed. The results obtained in these experiments will be reported by the Process Research Group as part of their overall gasification rate testing.

### 3. Coal Feeding Studies

Design of the pre-pilot plant coal feeder has been completed and is shown in Figure 3.

# B. <u>Projections</u>

### 1. Environmental Testing of High-Temperature Materials

Testing of the materials currently in Oxidation Test #3 will continue.

Additional gasification rate tests will be conducted at atmospheric pressure with two bed heights (one approximately twice the other) and with top and bottom coal feeding at one bed height.

