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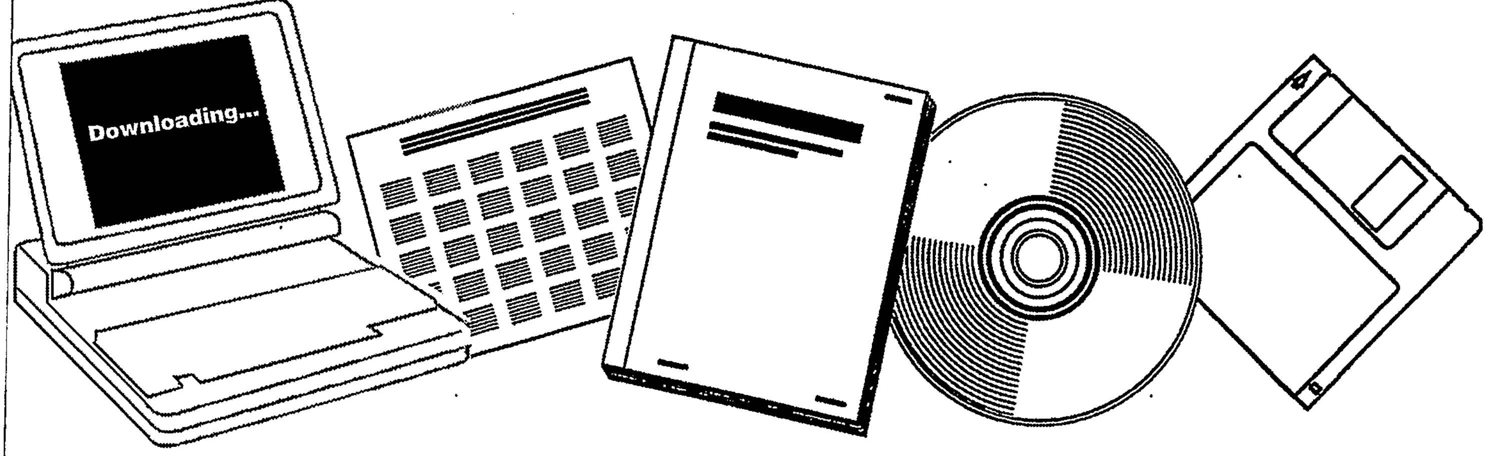
DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS. PROGESS REPORTS, JANUARY--DECEMBER 1967

INSTITUTE OF GAS TECHNOLOGY, CHICAGO,
ILL

1967



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FE--381-T-4

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Reports for the
Period January - December 1967

Institute of Gas Technology
IIT Center
Chicago, Illinois 60616

Prepared for

Office of Coal Research
U. S. Department of the Interior
and
American Gas Association
(Third Quarter Report)
OCR Contract No. 14-01-0001-381*

*This contract evolved into OCR Contract No. 14-32-0001-1221 in 1972

FE--381-T-4

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IGT-MPR--1/67

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report — January 1967
to
Office of Coal Research
Contract No. 14-01-0001-381

Summary

1. Six hydrogasification tests were conducted in January.
2. The test series on West Virginia No. 5 Block seam coal was completed.
3. Gasification of Pocahontas coal, using the normal procedure with which other coals have been tested, tends to be difficult. Problems encountered are probably due to pretreatment procedure.
4. Four coal pretreatment runs were made with Illinois No. 6 coal. These runs complete the pretreatment work for this specific coal.

Hydrogasification

Six hydrogasification tests were conducted this month in the balanced-pressure pilot unit. One test (Run HT-149) was made with a lightly pretreated West Virginia No. 5 Block seam bituminous coal. This test concluded the basic test series for evaluating the fluid-bed hydrogasification performance of this coal in the pilot plant. Two tests (Runs HT-150 and HT-151) were conducted with a lightly pretreated Pocahontas No. 4 seam low-volatile-content bituminous coal from West Virginia. In the other three tests (Runs HT-152, HT-153, and HT-154), the feed was a lightly pretreated Pittsburgh seam bituminous coal from the Ireland mine. The test with the West Virginia coal was successful. In both tests with Pocahontas coal, the coal agglomerated in the reactor tube, halting further operation. A partially successful operation was obtained with Ireland mine coal in Run HT-154. In the two other tests with this coal, excessive reactor pressure rises, caused by a partially plugged product gas filter, resulted in plugging of the top of the coal feed tube.

Run HT-149 is the fourth test of the series conducted with pretreated West Virginia No. 5 Block coal. Fed at a nominal rate of 65 lb/hr, the coal was reacted in a 3-1/2-foot-deep fluidized bed with 530 SCF/hr of hydrogen and 25 lb/hr of steam. At these flow conditions the hydrogen/coal ratio was 20% of the stoichiometric ratio, and the steam concentration in the hydrogen-steam feed gas was 50 mole percent. Reactor pressure was 1000 psig. The coal bed temperature was controlled to a nominal 1700°F. This very successful test lasted 4-1/2 hours, 1-1/2 hours at steady state. The run was terminated when the coal feed supply was used up.

Run HT-150, the first test with Pocahontas coal, was conducted at 1000 psig with a 3-1/2-ft fluidized coal bed controlled to a nominal temperature of 1700°F. The coal was fed at a nominal 46 lb/hr and reacted with 530 SCF/hr of hydrogen (25% of the stoichiometric hydrogen/coal ratio) and 25 lb/hr of steam (50 mole percent of the hydrogen-steam feed gas). About 45 lb of coal was fed for 1 hour before the run had to be terminated. The coal had agglomerated in the 4-inch-diameter reactor tube, and could not be discharged. Agglomeration of the coal was noted throughout the reactor tube.

Run HT-151 was conducted at conditions similar to those of Run HT-150. The start-up procedures, however, were modified to eliminate or substantially reduce the agglomerating tendencies of the coal. From the experience of Run HT-150, it appeared that the reactivity of the Pocahontas coal was lower than that of the medium-volatile-content bituminous coals hydrogasified in previous tests. To keep the rate of reaction high, the hydrogen/coal ratio at the start was at least 50% of stoichiometric. Also, the temperature at the top of the reactor was about 100°F higher than the usual 1300°F. This run was terminated after 1-1/2 hours when coal agglomerated in the reactor. About 34 lb of coal was fed during this period. Considering the quantity of coal and the size of the agglomerates formed, agglomeration in this test was not as serious as in Run HT-150. The pretreated coal feed of Runs HT-150 and HT-151 was found to be free-flowing when subjected to the standard laboratory agglomeration tests.

The three runs made with the Ireland mine bituminous coal were attempts to reproduce the results of a previously conducted test with this coal at 1500 psig. By reproducing these results, the effects of operating at 1500 psig would be verified. Operating conditions for these fluid-bed tests were similar to those of the previous test at 1500 psig (Run HT-129, August 1966 Progress Report). Two of these tests, Runs HT-152 and HT-153, had to be terminated before steady-state operation could be established because of plugging of the coal feed tube. In Run HT-152, which lasted 1-1/2 hours, a relatively large leak at the coal feed hopper closure contributed to the plugging by disturbing the smooth flow of coal from the hopper. In both tests the outflow of product gases was restricted by a partially plugged metal filter. This resulted in a progressive rise of the reactor pressure of 75 psig in Run HT-152, and 135 psig in Run HT-153. We believe that this pressure rise contributed partially to the plugging of the coal feeder in Run HT-153.

A new product gas filter was installed for Run HT-154, which was effective in keeping the reactor pressure from rising. But this test was only partially successful as the steady-state portion was interrupted by temporary plugs in the coal feed tube. The stirring rod in the coal feed tube, used to promote the flow of coal, had snapped at the shaft sometime during the test. This caused the coal, being fed at 65 lb/hr, to choke in the tube. The test lasted 3-1/2 hours, about 3/4 hour at steady state.

Coal Pretreatment

Four pretreatment tests were run in the fluidized-bed pretreater to study minimum pretreatment conditions and to supply the hydrogasification unit with nonagglomerating feed char.

Table 1. NOMINAL CONDITIONS FOR PRETREATMENT TESTS

Run No.	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Avg Bed Temp, °F
FP-102D	0.28	1296	21.0	775
FP-103	0.26	1288	21.0	813
FP-104	0.24	1252	21.0	794
FP-105	0.25	1222	21.0	800

Bituminous coal, crushed and screened to −16+80 mesh, was used as feed for all four runs. The coal was from the Illinois No. 6 coal bed, supplied by Freeman Coal Mining Company's Crown mine. These runs were made to determine an optimum bed temperature with a minimum coal residence time.

Runs FP-102D and FP-103 were made with predried coal; Runs FP-104 and FP-105 used wet coal (12-13% as received). The wet coal reduced the capacity of the unit somewhat because of the amount of heat needed to flash off this moisture.

Run FP-102D ran for 5-1/4 hours; it was terminated when the feed supply ran out. The product char was found to be free-flowing with 25% volatile matter. Run FP-103 was terminated after 1-1/2 hours when the outlet to the reactor became plugged with tars and fine solids. The plug developed because the top reactor furnace failed at the beginning of the run. One hour of steady-state data were obtained. The char was found to be free-flowing with 24% volatile matter. Run FP-105 ran for over 7 hours; it was stopped when the supply of feed was depleted. The char was found to be free-flowing with 23% volatile matter.

The steam muffler was received and installed to heat the scrubber water, starting with Run FP-103. The muffler has effectively eliminated the steam hammering in the system. The scrubber system did not show any signs of plugging after the muffler was installed. This series of runs is the last to be made on the Illinois No. 6 coal. This coal also appears to be similar to the other high-volatile-content bituminous coals: It can be pretreated at high capacity at 800°F with a 15-20 minute residence time. The volatile matter in the free-flowing Illinois No. 6 char is in the 25 to 26% range for minimum pretreatment.

Methanation

Methanation kinetics studies and life tests are being run now with the following feed gas composition, which simulates the feed gas expected in the pilot plant methanator:

	mole %
CO	4.0
H_2	17.0
CH_4	71.5
CO_2	0.5
C_2H_6	3.5
N_2	3.5
Total	100.0

Stirred reactor runs with Girdler G-65 catalyst and this feed have covered temperatures of 650°-950°F and product gas CO compositions of 0.5 to 2.4 mole percent. The rate of reaction of CO_2 is too low to measure accurately. Ethane conversion to methane increases with temperature, but not as rapidly as with Harshaw nickel-on-kieselguhr catalyst.

Another life study using crushed Girdler G-65 catalyst was begun. The space velocity is about 3000 SCF/cu ft-hr which simulates the space velocity expected for the pilot plant.

We are now starting an investigation on the use of hydrodesulfurizing catalysts for conversion of organic sulfur compounds to H_2S in the gas cleanup system. One of the requirements of such a catalyst is that it be a poor methanation catalyst to prevent temperature rise in the reactor.

Coal Characterization

Calorimetry

Determination of the heat of reaction of the pretreatment of coal (Ireland mine) was completed for 700° and 800°F. Experimental data cover a range of pretreatment from 9 to 18%. The heats of reaction of hydrogen with North Dakota lignite and with Ireland mine coal is being measured at 1500°F.

Engineering Economics Studies

The study on the gasification of char with electric heat for hydrogen production is nearly complete. The total fixed investment where power is generated in the plant using rotating machinery is $185,410,000, whereas the total fixed investment with purchased power is $103,270,000. The state-of-the-art design (hydrogen by steam-oxygen) has been modified to base the pipeline gas section on pilot plant data so it is on a common basis with the cases in this study. The total fixed investment for this revised state-of-the-art design is $142,600,000, compared to $141,000,000 for the previous state-of-the-art design.

During the month no inventions were made in the course of this work.

Signed
Frank Schora, Assistant Director

PROGRAM FOR DEVELOPMENT OF I

OCR: Contract No. 14-01-000

1964					1965											
Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec

1st Year

PHASE I

a. Hydrogasification

1 Establish rate-controlling areas of operation
E-13
T-36
DS-$15,800
ODC- $5,500

2 Study effect of degree of pretreatment
E- 6
T-18
DS-$7,500
ODC-$2,500

3 Study effects of variables to establish kinetics of fixed-bed system
E-13
T-36
DS-$15,800
ODC- $4,000

4 Study fluidization and establish rates for fluid-bed operation
E-104
T-27
DS-$12,580
ODC- $3,000

5 Study H2O-O2-C System
E-3
T-9
DS-$3800
ODC-$1800

6 Study particle size effect for both fixed and fluid-bed operation
E- 9
T-27
DS-$11,300
ODC- $2,800

b. Coal Pretreatment

1 Modify coal pretreatment unit
E- 3
T-11
DS-$4,250
ODC-$6,000

2 Study effects of pretreatment variables on char properties
E- 45
T-224
DS-$30,400
ODC- $4,000

Establish

Produce material required for hydrogasification runs

c. Methanation

1 Review state of the art, and design lab test unit
E-7
T-0
DS-$3,800
ODC-0

2 Build and shake down lab unit
E-3
T-9
DS-$4,250
ODC-$3,500

3 Lab scale catalyst evaluation
E- 5
T-12
DS-$6,850
ODC-$1,250

4 Study effects of gas composition
E- 5 DS-$6,150
T-10 ODC-$1,900

5 Design and construct small pilot unit
E- 5 DS-$8,500
T-18 ODC-$13,500

d. Coal Characterization

1 Develop techniques and study petrographic characteristics of coal, char, and spent hydrogasified residue. Determine heats of reaction of coal and chars
E-13
T- 8
DS-$8,900
ODC- $20,500

Establish te estimation

PHASE II

a. Analysis of Experimental Data

1 Analyze and correlate data from hydrogasification, coal pretreatment, and methanation work, and recommend conditions for additional tests. Develop kinetic equations to use in reactor design
E-32
T- 0
DS-$13,800
ODC- 0

b. Process Concept Development

1 Develop simple math model of process based on present data
E-5
T-0
DS-$2,300
ODC-0

2 Improve and expand model when additional data becomes available and determine areas where data are needed. Modify process concept as necessary and keep process flowsheets upd
E-20
T- 0
DS-$9,200
ODC-$1,500

c. Reactor Design Studies

d. Economic Evaluation

1 Cost data review and extension
E-7
T-0
DS-$3,200
ODC-0

2 Develop simple flow sheet
E-2 DS-$900
T-0 ODC-0

3 Detailed flow sheet, energy and material balances
E-3
T-0
DS-$1,400
ODC-0

4 Process design of various sections
E-4
T-0
DS-$1,800
ODC-0

5 Cost estimate of equipment, plant and operating expenses
E-3
T-0
DS-$1,400
ODC-0

6 Report Preparation
E-2
T-0
DS-$900
ODC-0

7 Revision and updating of flowsheet
E-6
T-0
DS-$2,600
ODC-0

PHASE III

a. Preliminary Pilot Plant Design

TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)

	TOTAL	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	
Planned	855,400	23,500	23,500	23,500	23,800	22,400	24,100	24,300	24,700	23,700	23,300	23,800	30,300	29,300	27,800	27,400	28,100	28,100	2
Actual		10,000	15,000	23,300	14,500	23,000	21,100	19,800	28,900	23,400	30,200	24,500	22,900	25,500	21,900	26,800	27,800	22,300	2
Cumulative		10,000	25,000	48,300	62,800	85,800	06,900	26,700	155,600	179,000	209,200	233,700	256,600	282,100	304,000	330,800	358,700	381,000	4

Manpower shown in total man months: E = Engineers
T = Technicians

Costs shown are costs to OCR or

OF IGT HYDROGASIFICATION PROCESS

14-01-0001-381 A.G.A. PB-23a

1966 — 1967

Dec Jan Feb Mar Apr May Jun Jul | Aug Sep Oct Nov Dec Jan Feb Mar Apr May Jun Jul

2nd Year | 3rd Year

le size
ath lized
operatior
12,300
12,000

7
Study hydrogasification of various coals, lignite, under most suitable conditions
E = 214
T = 63
DS = $27,200
ODC = $7,000

8
Perform any support work necessary for phase III
Double check data on which pilot plant is being designed

3
Establish pretreatment properties of various coals
E = 5½
T = 27½
DS = $12,600
OCD = $4,500

fication runs

4
Produce material needed for any hydrogasification runs

mposition
$6,150
$1,000

6
Run small pilot unit and develop kinetic equations for design
E = 10
T = 19
DS = $11,500
ODC = $5,250

7
Perform any support work necessary for phase III

2
Establish tests and develop correlations to enable estimation of worth of coal for hydrogasification
E = 14
T = 11
DS = $10,200
ODC = $500

Laboratory and Pilot Plant Phase I-a8, b4, c7 II-a2 | Engineers 24 | Technicians 105 | Salaries $40,400 | Other Direct Costs 11,400

ork, and
design

2
Perform analysis of past data and new data in any areas necessary for phase III work

ermine areas where additional
ess flowsheets updated
3

1
Make preliminary reactor design based on process concept and hydrogasification results
E = 6
T = 0
DS = $2,800
ODC = 0

2
Work with subcontractor on design of reactors for hydrogasification and methanation
E = 4
T = 0
DS = $1,800
ODC = 0

f flowsheet

8
Revision and refinement of capital and operating costs
E = 6
T = 0
DS = $2,800
ODC = 0

9
Work with subcontractor on capital and operating costs for pilot plant
E = 9
T = 0
DS = $4,100
ODC = 0

1
Develop pilot plant flowsheet with heat and material balances
E = 14
T = 0
DS = $7,700
ODC = 0

Select architect-engineer subcontractor

2
With assistance of subcontractor work on phase III as outlined in Contract No. 14-01-0001-381.*
E = 34
T = 0
DS = $16,300
ODC = 0

2

* $85,500 available for architect-engineer subcontractor and engineering consultants.

AND FEE)

00	28,100	23,300	23,300	23,300	23,300	23,300	23,300	23,300	15,800	15,800	15,800	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700
00	22,300	23,400	22,300	25,000	32,600	25,000	26,600	27,200	22,900	25,700	19,200	21,800	21,400	22,900						
700	381,000	404,400	426,700	451,700	484,300	509,300	535,900	563,100	586,000	609,700	628,900	650,700	672,100	695,000						

re costs to OCR or one-half of total costs. DS = Direct Salaries, $
ODC = Other Direct Costs, $

IGT-MPR--2/67

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report — February 1967
to
Office of Coal Research
Contract No. 14-01-0001-381

Summary

1. Five hydrogasification tests were conducted in February. Three tests were made with Illinois No. 6 and two with Indiana No. 6 coal.
2. Five pretreatment tests were attempted, one of which was terminated early because of equipment problems. The coal being studied was Indiana No. 6 from the Minnehaha mine of Republic Coal & Coke Company.
3. A hydrodesulfurization catalyst is being evaluated for probable use in the forthcoming pilot plant.
4. A brief study is now being undertaken to examine the several alternatives for producing hydrogen using purchased electric power or a fuel cell as the power source.

Hydrogasification

Five hydrogasification tests were conducted this month in the balanced-pressure pilot unit. Three of these tests (Runs HT-155, HT-156, and HT-157) were made with a lightly pretreated Illinois No. 6 high-volatile-content bituminous coal. All three of these fluid-bed tests were successful. In the other two tests conducted this month (Runs HT-158 and HT-159) the feed coal was a lightly pretreated Indiana high-volatile-content bituminous coal from the Minnehaha mine's No. 6 vein. The first test with the Indiana coal was successful, but in the second test the coal caked at the end of the coal injection tube.

Run HT-155 was the first test of the series to evaluate the fluid-bed hydrogasification of Illinois No. 6 coal in the pilot unit at a pressure of 1000 psig and a coal bed temperature of 1700°F. At a nominal feed rate of 56 lb/hr, the coal was reacted in a 3-1/2-ft fluidized bed with 530 SCF/hr of hydrogen and 25 lb/hr of steam. At these flow conditions, the hydrogen/coal ratio was 25% of the stoichiometric ratio, and the steam concentration in the feed gas was 50 mole percent. The duration of the test was 5-1/4 hours, with 3 hours at steady-state conditions. About 34% of the carbon in the coal was gasified in producing a gas of 549 Btu/SCF (nitrogen-free basis).

In Run HT-156, the Illinois coal was reacted at the same pressure, temperature, and coal bed height as in Run HT-155. However, the nominal coal feed rate was 79 lb/hr, the hydrogen rate was 743 SCF/hr, and the steam rate was 15 lb/hr. At these flow conditions, the hydrogen/coal ratio was 25% of the stoichiometric ratio, and the steam concentration in the feed gas was 30 mole percent. Three hours of steady-state operation were obtained in this test that lasted 5-1/2 hours. A product gas with a heating value of 595 Btu/SCF (nitrogen-free basis) was produced.

In the third test of the series with Illinois bituminous coal (Run HT-157) the pressure, temperature, and coal bed height were similar to those of Run HT-155. With a coal feed rate of 70 lb/hr and a hydrogen rate of 530 SCF/hr, a hydrogen/coal ratio of

20% of stoichiometric was maintained. The steam rate was 2' lb/hr for a concentration of 50 mole percent in the feed gas This test lasted 5 hours. During the 3 hours of steady-state operation a product gas of 549 Btu/SCF was produced.

The operations with Illinois bituminous coal were followed by a successful test with a lightly pretreated Indiana bituminous coal (Run HT-158). The coal was reacted in a 3-1/2-ft fluidized bed at a pressure of 1000 psig and a nominal coal bed temperature of 1700°F. It was fed at a nominal 54 lb/hr for reaction with 530 SCF/hr of hydrogen and 25 lb/hr of steam. Total test time was 5-1/2 hours, with 3-1/4 hours at steady state. A product gas of 554 Btu/SCF was produced.

Again, the lightly pretreated Indiana coal was used as feed in Run HT-159. This material showed a slight tendency to agglomerate when subjected to the standard laboratory agglomeration test. However, as this tendency was only marginal, and the analysis of the pretreated coal indicated sufficient pretreatment, the coal was used as feed in the hydrogasification pilot unit. It was fed successfully for 2-1/2 hours before it caked at the end of the 1-inch-ID coal injection tube and jammed the feed screw.

Additional coal had agglomerated on the walls of the reactor tube about 5-1/2 ft below the top of the reactor. Contributing to the plug in the coal feed tube were reactor pressure variations resulting from a partially plugged product gas bayonet filter. Although the nominal coal feed rate for this test was 76 lb/hr, the actual rate at the time of the plug was 95 lb/hr. The hydrogen rate was 743 SCF/hr, and the steam rate was 15 lb/hr. Reactor pressure, coal bed temperature, and coal bed height were similar to those of Run HT-158.

Coal Pretreatment

Five pretreatment tests were run in the fluidized-bed pretreater to study minimum pretreatment conditions and to supply the hydrogasification unit with nonagglomerating feed char.

Table 1. NOMINAL CONDITIONS FOR THE PRETREATMENT TESTS

Run No.	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Avg Bed Temp, °F
FP-106	0.27	1216	21.0	797
FP-107	0.29	1219	21.0	777
FP-108	0.15	1419	21.0	814
FP-109	0.15	1418	21.0	824
FP-110*	0.15	1425	21.0	820

* Intended run conditions, but not attained.

Bituminous coal, crushed and screened to –16+80 mesh, was used as feed for all five runs. The coal used was from the Indiana No. 6 vein, supplied by Republic Coal & Coke Company's Minnehaha mine. The runs were made to determine an optimum bed temperature with a minimum coal residence time. Runs FP-106, FP-107, and FP-108 were made with undried coal. Runs FP-109 and FP-110 used coal that was predried in batch driers just enough to prevent bridging in the feed hopper.

Run FP-106 ran for 5-3/4 hours, terminating when the feed supply ran out. The product char was found to be free-flowing with 23.5% volatile matter. Run FP-107 was terminated after 5-3/4 hours when the feed was depleted. The product char was found to be very lightly caked with 24.5% volatile matter. The coal feed for Run FP-108 was so wet from surface moisture that bridging caused the shutdown of this run after 3 hours. The char was found to be very lightly caked with 23.5% volatile matter. Run FP-109 was a repeat of FP-108 with the coal dried. The run was terminated after 5 hours when the feed supply was used up. The char was found to be free-flowing with 23% volatile matter. Run FP-110 was terminated before steady state was reached because of a mechanical failure at the air compressor and air-regulating system. The last three runs were attempts to again increase capacity of the unit. For the Indiana coal, a residence time of 9-10 minutes at 800°-825°F appears sufficient to pretreat the coal and make it nonagglomerating. An increased air feed rate to a superficial velocity of 1.75 ft/sec reduces the fluidized-bed density enough to cut down the coal residence time and produce enough agitation to prevent agglomeration in the bed at 825°F. The Indiana coal appears to be free-flowing only when the volatile matter has been reduced to 23.5%. This is slightly lower than the volatile level needed to render previous high-volatile-content coals nonagglomerating.

High-Pressure Methanation

We have made several runs with Harshaw Co-Mo-0601 (cobalt molybdate) hydrodesulfurization catalyst to check its methanating ability. At space velocities of about 3200 hr^{-1} and temperatures from 600° to 800°F the methanation rate is low. The possibility of using this catalyst in a hydrodesulfurization step prior to the methanator in the pilot plant depends on its ability to convert organic sulfur compounds such as COS and CS_2 to H_2S without having simultaneous methanation cause heat removal problems. The hydrodesulfurizing ability of this catalyst at run conditions will be checked in the life test unit after the current test is completed.

The life study with Girdler G-65 catalyst, mentioned last month, was terminated after 168 hours on-stream. Carbon deposition (due to high temperatures) and sulfur poisoning were suspected causes of the deactivation. We are now testing Harshaw Ni-0104T catalyst in the life study system.

We have received another nickel-on-kieselguhr catalyst, Harshaw Ni-0116T. Its crush strength is about three times as great as that of Harshaw Ni-0104. We are comparing the activities of these two catalysts in the stirred reactor.

Coal Characterization

Petrography

Feed and residue samples from the fluid-bed hydrogasification of Ohio No. 6 and West Virginia No. 5 Block coals and North Dakota lignite are being mounted and polished for petrographic examination. In addition, the agglomeration of the Pocahontas coal in attempted hydrogasification runs is being investigated.

The surface area of Ireland mine coal at different stages of fluid-bed hydrogasification was determined.

Calorimetry

Determination of the heat of reaction of hydrogen and North Dakota lignite was completed at 1500°F and 1000 psia. The heat of reactions of hydrogen and Ireland mine coal and chars are being measured at 1500°F and 1000 psia.

Because of the process economics sensitivity to the cost of hydrogen, we are now making a brief study of possible methods of producing hydrogen. This study is based on evaluation of several alternative routes for the production of hydrogen, using electric power produced both by rotating machinery and fuel cells.

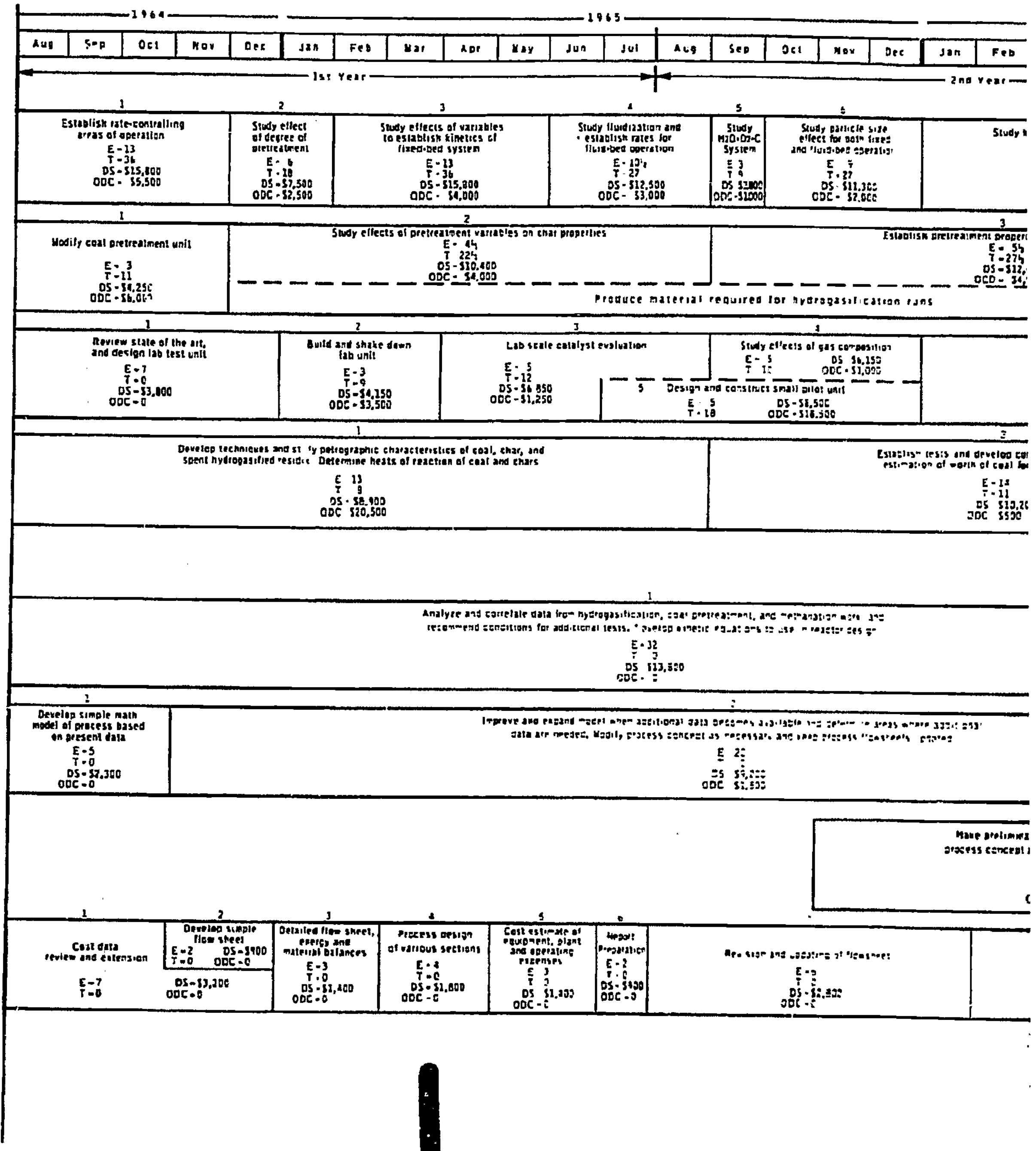
No new inventions were made this month in the course of this work.

Approved____________________ Signed____________________

Jack Huebler, Associate Director Frank Schora, Assistant Director

PROGRAM FOR DEVELOPMENT OF IGT

OCR: Contract No. 14-01-0001-381



PHASE I

a. Hydrogasification

1. Establish rate-controlling areas of operation: E - 13, T - 36, DS - $15,800, ODC - $5,500
2. Study effect of degree of pretreatment: E - 6, T - 18, DS - $7,500, ODC - $2,500
3. Study effects of variables to establish kinetics of fixed-bed system: E - 13, T - 36, DS - $15,800, ODC - $4,000
4. Study fluidization and establish rates for fluid-bed operation: E - 10½, T - 27, DS - $12,500, ODC - $3,000
5. Study H2O-O2-C System: E 3, T 9, DS $2800, ODC - $1000
6. Study particle size effect for both fixed and fluid-bed operation: E 9, T - 27, DS - $11,300, ODC - $2,000

Study h

b. Coal Pretreatment

1. Modify coal pretreatment unit: E - 3, T - 11, DS - $4,250, ODC - $6,000
2. Study effects of pretreatment variables on char properties: E - 44, T 224, DS - $10,400, ODC - $4,000
3. Establish pretreatment proper: E - 54, T - 274, DS - $12, ODC - $4,

Produce material required for hydrogasification runs

c. Methanation

1. Review state of the art, and design lab test unit: E - 7, T - 0, DS - $3,800, ODC - 0
2. Build and shake down lab unit: E - 3, T - 9, DS - $4,150, ODC - $3,500
3. Lab scale catalyst evaluation: E - 5, T - 12, DS - $6 850, ODC - $1,250
4. Study effects of gas composition: E - 5, T 12, DS $6,150, ODC - $1,000
5. Design and construct small pilot unit: E - 5, T - 18, DS - $8,500, ODC - $18,500

d. Coal Characterization

1. Develop techniques and study petrographic characteristics of coal, char, and spent hydrogasified residue. Determine heats of reaction of coal and chars: E 13, T 9, DS - $8,900, ODC $20,500
2. Establish tests and develop co estimation of worth of coal fo: E - 14, T - 11, DS $10,2, ODC $500

PHASE II

a. Analysis of Experimental Data

1. Analyze and correlate data from hydrogasification, coal pretreatment, and methanation work and recommend conditions for additional tests. Develop kinetic equations to use in reactor design: E - 32, T 0, DS $13,500, ODC - 0

b. Process Concept Development

1. Develop simple math model of process based on present data: E - 5, T - 0, DS - $7,300, ODC - 0
2. Improve and expand model when additional data becomes available and determine areas where additional data are needed. Modify process concept as necessary and keep process flowsheets updated: E 20, DS $9,000, ODC $1,500

c. Reactor Design Studies

Make prelimina process concept

d. Economic Evaluation

1. Cost data review and extension: E - 7, T - 0
2. Develop simple flow sheet: E - 2, T - 0, DS - $900, ODC - 0

DS - $3,200, ODC - 0

3. Detailed flow sheet, energy and material balances: E - 3, T - 0, DS - $1,400, ODC - 0
4. Process design of various sections: E - 4, T - 0, DS - $1,800, ODC - 0
5. Cost estimate of equipment, plant and operating expenses: E 3, T 0, DS $1,300, ODC - 0
6. Report Preparation: E - 2, T - 0, DS - $900, ODC - 0

Revision and updating of flowsheet: E - 5, T - 0, DS - $2,800, ODC - 0

PHASE III

a. Preliminary Pilot Plant Design

TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)

	TOTAL	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb
Planned	855,400	23,500	23,500	23,500	23,800	22,400	24,100	24,300	24,700	23,700	23,300	23,800	30,300	29,300	27,800	27,400	28,100	28 100	23,300	23,300
Actual		10,000	15,000	23,300	14,500	23,000	21,100	19,600	28,900	23,400	32,200	24,500	22,500	25,500	21,900	26,900	27,800	22,300	23,400	22,300
Cumulative		10,000	25,000	48,300	62,800	85,800	106,900	126,700	155,600	179,000	209,200	233,700	256,600	282,100	304,000	330,900	358,700	381,000	404,400	426,700

Manpower shown in total man-months: E - Engineers
T - Technicians

Costs shown are costs to OCR or one-half of total c

F IGT HYDROGASIFICATION PROCESS

14-01-0001-381 A.G.A. PB-23a

1966 — 1967

Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul

2nd Year ← → 3rd Year

le size
ath fixed
operatior
11,300
12,000

7
Study hydrogasification of various coals, lignite, under most suitable conditions
E-214
T-63
DS-$27,200
ODC- $7,000

8
Perform any support work necessary for phase III
Double check data on which pilot plant is being designed

3
Establish pretreatment properties of various coals
E- 54
T-274
DS-$12,600
OCD- $4,500

ification runs

4
Produce material needed for any hydrogasification runs

position
6,150
1,000

6
Run small pilot unit and develop kinetic equations for design
E-10
T-19
DS-$11,500
ODC-$5,250

7
Perform any support work necessary for phase III

2
Establish tests and develop correlations to enable estimation of worth of coal for hydrogasification
E-14
T-11
DS $10,200
ODC $500

Laboratory and Pilot Plant Phase I-a8, b4, c7 II-a2	Engineers 24 Technicians 165	Salaries $40,400 Other Direct Costs 11,400	

ork, and
design

2
Perform analysis of past data and new data in any areas necessary for phase III work

mine areas where additions
as flowsheets updated

1
Make preliminary reactor design based on process concept and hydrogasification results
E-6
T-0
DS-$2,800
ODC-0

2
Work with subcontractor on design of reactors for hydrogasification and methanation
E-4
T-0
DS-$1,800
ODC-0

flowsheet

8
Revision and refinement of capital and operating costs
E-6
T-0
DS-$2,800
ODC-0

9
Work with subcontractor on capital and operating costs for pilot plant
E-9
T-0
DS-$4,100
ODC-0

1
Develop pilot plant flowsheet with heat and material balances
E-14
T- 0
DS-$7,700
ODC- 0

Select architect-engineer subcontractor

2
With assistance of subcontractor work on phase III as outlined in Contract No. 14-01-0001-381.*
E-34
T- 0
DS-$16,300
ODC- 0

2

* $95,500 available for architect-engineer subcontractor and engineering consultants.

D FEE)

)	28,100	23,100	23,300	23,300	23,300	23,300	23,300	23,300	15,800	15,800	15,800	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700
00	22,300	23,400	22,300	25,000	32,600	25,000	26,600	27,200	22,900	23,700	19,200	21,600	21,400	22,900	39,200					
00	381,000	404,400	426,700	451,700	484,300	509,300	535,600	563,100	586,000	609,700	628,900	650,700	672,100	695,000	734,200					

Me costs is [illegible] of overall of total costs. DS Direct Salaries, $
ODC - Other Direct Costs, $

IGT-MPR--3/67

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report – March 1967
to
Office of Coal Research
Contract No. 14-01-0001-381

Summary

1. Eight hydrogasification tests were conducted during March. Two tests were made with Indiana Sixth vein high-volatile-content bituminous coal, four with West Virginia Sewell seam medium-volatile-content bituminous coal, and two with Pocahontas No. 4 seam low-volatile-content bituminous coal.
2. Ten coal pretreatment tests were conducted: three with Indiana Sixth vein coal and seven with West Virginia Sewell seam coal.
3. The life study of a methanation catalyst has exceeded 500 hours, producing, at a pressure of 1000 psig, a gas with less than 0.1% carbon monoxide.
4. A preliminary economic analysis of an electrothermal hydrogen producer and hydrogasifier combination indicates a gas price of 50¢-51¢/million Btu at an electric power cost of 3-3.5 mills/kwhr.

GOOD!

Hydrogasification

We conducted eight hydrogasification tests this month in the balanced-pressure pilot unit. Two tests (Runs HT-160 and HT-161) were conducted with a lightly pretreated Indiana Sixth vein high-volatile-content bituminous coal. Four tests (Runs HT-162, HT-163, HT-164, and HT-165) were conducted with a lightly pretreated West Virginia Sewell seam medium-volatile-content bituminous coal. In the remaining two tests we conducted this month (Runs HT-166 and HT-167), the feed coal was a lightly pretreated Pocahontas No. 4 seam low-volatile-content bituminous coal. Both of the tests conducted with Indiana coal were successful. Of the tests conducted with West Virginia coal, one was successful. In the three other tests with this coal, the coal agglomerated in the reactor. One test with Pocahontas coal was successful, while the other was unsuccessful because of agglomeration of the material in the reactor.

Run HT-160 was the third test in the series conducted in the pilot unit with lightly pretreated Indiana bituminous coal. We reacted the coal in a 3-1/2-ft fluidized bed with a hydrogen-steam mixture at 1000 psig and a nominal coal bed temperature of 1700°F. The hydrogen/coal feed ratio was 25% of the stoichiometric ratio, and the steam concentration in the feed gas was 30 mole percent. The test lasted 5 hours, with 2-1/4 hours at steady state. About 30% of the carbon was gasified, producing a gas of 560 Btu/SCF (nitrogen-free basis).

We conducted the last test in the current series with Indiana bituminous coal (Run HT-161) in a 3-1/2-ft fluidized bed at a pressure and a temperature similar to those of Run HT-160. However, the hydrogen/coal feed ratio was 20% of the stoichiometric ratio, and the steam concentration in the feed gas was 50 mole percent. The total and steady-state test durations were similar to those of Run HT-160. Carbon gasification was about 35%, and the product gas heating value was 560 Btu/SCF (nitrogen-free basis).

Run HT-162 was the first hydrogasification test with a Sewell seam West Virginia bituminous coal. In this test, we fed the coal at a nominal 48 lb/hr and reacted it in a 3-1/2-ft fluid bed with 530 SCF/hr of hydrogen (25% of the stoichiometric hydrogen/coal ratio) and with 25 lb/hr of steam (50 mole percent concentration). The coal bed temperature was a nominal 1700°F, while the free-fall section above the bed was controlled to 1300°F. About 45 minutes after the start of coal feeding – some 30 minutes before the time estimated for the coal bed to reach this level – the coal bed radiation gage indicated a coal bed at the 3-1/2-ft level. We turned on the coal bed discharge screw soon after this, but the coal would not flow. The coal removed from the reactor after shutdown of the test was only lightly agglomerated. Agglomeration was estimated to have started 2 ft above the hydrogen-steam feed tube. Laboratory tests showed that the bulk of the pretreated coal used in this test was nonagglomerating.

Test conditions for Run HT-163 were similar to those of Run HT-162, except that the free-fall section of the reactor tube was heated to 1500°F. We modified the start-up procedure so that, initially, the hydrogen/coal ratio would be 75% of the stoichiometric ratio and the hydrogen concentration in the feed gas would be 85%. The objective of the 1500°F free-fall temperature and the start-up modification was to increase, as much as possible, the reaction of the coal in the free-fall zone and to thereby reduce the chances of agglomeration when the coal particles reached the coal bed. The temperature and start-up modifications were partially successful. A coal bed was established, and one coal discharge cycle was completed. Following this, an agglomerated layer of coal formed at the top of the coal bed which kept coal from moving past this level. We terminated the run 2 hours after coal feeding was started.

For Run HT-164 we made changes to allow a longer free-fall reaction time before the coal reached the coal bed. The free-fall section of the reactor was increased by lowering the top of the hydrogen-steam tube 31 in. Also, we limited the coal bed depth to 2 ft so we could add an additional 18 in. to the length of the free-fall zone. The test was started as a free-fall operation. About 1/2 hour after we started coal feeding, and before the full coal and steam feed rates were reached, coal began to agglomerate about 2 ft above the end of the hydrogen-steam feed tube. We had to stop the test because coal would not flow past the level of agglomeration.

Following the same procedures established for Run HT-164, Run HT-165 was a successful hydrogasification run with medium-volatile-content West Virginia bituminous coal. In the first part of the run, the coal was hydrogasified in the free-fall zone for a steady-state period of 1 hour. In the second part of the run, the coal was reacted in a 2-ft fluid bed for 2-1/2 hours.

We resumed the hydrogasification of a Pocahontas bituminous coal in Run HT-166, which was based on the demonstrated success of the test procedures used in Run HT-165. In two earlier attempts at hydrogasifying Pocahontas coal (Runs HT-150 and HT-151, January 1967 Progress Report), agglomeration occurred to a moderate degree in the reactor. Run HT-166 was, therefore, conducted at test conditions, and with procedures similar to those of Run HT-165. Within a period of 1-1/2 hours, the coal feed rate was 48 lb/hr, and the steam rate was 12 lb/hr (25 mole percent). In the next 10 minutes, while the steam rate was being increased to 18 lb/hr, the radiation bed-level gage indicated holdup of coal in the reactor. The coal had agglomerated in the vicinity of the hydrogen-steam feed tube. However, the agglomeration was much less severe than in the two earlier tests with the Pocahontas coal. About 37% of the coal was processed before shutdown.

Test conditions for Run HT-167 were similar to those of Run HT-166. However, to maintain a high hydrogen concentration until a smooth free-fall operation of at least 1 hour was obtained, we kept the steam rate at 6 lb/hr (12 mole percent) for this period. The steam rate was increased by 6 lb/hr at 25 minute intervals until the 25 lb/hr rate was reached. This was followed by a successful free-fall test lasting 1 hour. For the second phase of the run, a 2-ft fluid bed was established. However, 10 minutes after this bed height was reached, the coal would no longer discharge from the reactor, and the test had to be shut down. As in Run HT-166, the coal had agglomerated at the hydrogen-steam feed tube.

Coal Pretreatment

We ran 10 pretreatment tests in the fluidized-bed pretreater to study minimum pretreatment conditions and to supply the hydrogasification unit with nonagglomerating feed char. The results of these tests are shown in Table 1.

Table 1. NOMINAL CONDITIONS FOR THE PRETREATMENT TESTS

Run No.	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Avg Bed Temp, °F
FP-110A	0.16	1427	21.0	813
FP-111*	0.20	1450	21.0	810
FP-111A	0.20	1479	21.0	806
FP-112	0.32	1484	21.0	817
FP-113	0.33	1378	21.0	844
FP-114	0.15	1538	21.0	863
FP-115	0.26	1328	21.0	844
FP-116	0.30	1194	21.0	785
FP-117	0.17	1314	21.0	851
FP-118	0.20	1475	18.67	843

* Intended run conditions, but not attained.

Bituminous coal, crushed and screened to −16+80 mesh, was used for all 10 runs. For the first three runs, high-volatile-content coal from the Indiana Sixth vein was used. For the remaining seven runs, a medium-volatile-content bituminous coal from the West Virginia Sewell seam was used. This coal was obtained from the New River Coal Co.'s Lochgelly mine. The runs with the Indiana coal were to supply the hydrogasifier with nonagglomerating feed and to confirm the best conditions for pretreating this coal. We made the runs of the West Virginia coal to find operating conditions that would pretreat the coal and make it free-flowing, and then to optimize these conditions for minimum pretreatment and maximum capacity.

Run FP-110A ran for 5 hours and was terminated when the feed supply ran out. The product char was free-flowing with 23% volatile matter. We stopped Run FP-111 before steady state was reached because the feed coal bridged in the hopper and caused the reactor temperature to rise rapidly, causing agglomeration in the coal bed. Run FP-111A was to repeat FP-111 and was stopped after 4-3/4 hours when the feed ran out. The char was free-flowing with 23% volatile matter. These three runs conclude the pretreatment investigation of the Indiana coal.

Because the chemical analyses of West Virginia Sewell coal is somewhat different from those of the high-volatile-content bituminous coals, we subjected it to four exploratory runs to find a set of conditions that would both pretreat it and make it nonagglomerating. During the third run, we had some difficulty controlling the bed temperature. Run FP-112 ran for 4-3/4 hours and was stopped when the feed ran out. We ran the bed temperature at two levels that averaged 817°F. The product char was lightly caked with 16.5-18% volatile matter. Run FP-113 had run for 2-1/2 hours when the bed began to defluidize and channel. The char was lightly caked with 16.9-17.3% volatile matter. Run FP-114 had temperature control problems after 1-1/2 hours and was shut down after a hot spot occurred which agglomerated the bed. However, 1 hour of steady-state data was obtained. The char was lightly caked with 15.3% volatile matter. The coal residence time was very short compared to Runs FP-112 and FP-113; thus, the high temperature was not enough to pretreat the coal. Run FP-115 ran for 5 hours until the feed ran out. We had no control problems. The product char was free-flowing with 16.5% volatile matter. We combined the chars from Runs FP-112, FP-113, and FP-114 to use as a feed for Run FP-116, in which we wanted to make some free-flowing coal for use in the hydrogasifier. However, the conditions used were not quite severe enough; the product char was very lightly caked with 16.5-17.5% volatile matter. Run FP-117 was an attempt to reproduce the conditions of Run FP-115, but after 1-3/4 hours the bed bottom developed a hot spot, causing a shutdown. Attempts to control the temperature in the bed included using a very high coal rate near the end of the run and also decreasing the air feed rate. Apparently, once the hot spot has started, it cannot be controlled by either of these two methods. The product char was lightly caked with 15.4% volatile matter. Run FP-118 was again an attempt to reproduce Run FP-115 and again a hot spot occurred early in the run. We used a small flow of nitrogen to control the temperature. Rather than adjusting either the coal rate or air feed rate, the nitrogen rate was adjusted throughout the run to control the temperature. The product char was lightly caked with 15.6-16.8% volatile matter. This medium-volatile-content coal is definitely harder to pretreat than the high-volatile-content coal that we have previously pretreated. Pretreatment conditions for this coal are as yet not well defined because the temperature control problem has not allowed us to reproduce a good run.

High-Temperature Methanation

We have checked the ability of the Ni-0116T catalyst to methanate a gas mixture expected in the proposed pilot plant. The Ni-0116T catalyst is more active than the Ni-0104T, probably because it contains more nickel (65% compared to 58% for Ni-0104T) and its specific surface area is higher. A major advantage of Ni-0116T over Ni-0104T is its higher (by a factor of 3) crush strength.

The life study with Harshaw Ni-0104T 1/8 in. catalyst, mentioned last month, has exceeded 500 hours at a space velocity of 2500 hr^{-1} and a maximum bed temperature of 850°F. The 4% CO feed is being methanated to 0.1% CO. When we increased the space velocity to 5000 hr^{-1} on two occasions, the product gas CO composition remained at 0.1% CO at steady state.

Coal Characterization

Calorimetry

We completed our determination of the heat of reaction of hydrogen and coal (Ireland mine) and chars at 1500°F and 1000 psia. The results indicate that the exothermic heat of reaction at 1500°F is about 10% higher than that at 1300°F. The materials studied are the raw coal, pretreated coal, and low-temperature and high-temperature residue from the hydrogasifier. The heat capacity of coal is being determined at 1500°F.

Economic Studies

We made some rough estimates to evaluate the effect of process changes on the cost of pipeline gas made from coal by hydrogasification when the hydrogen used is made by the electrothermal fluid-bed gasification of char. Our estimates are based on figures from earlier, more detailed cost estimates. The major modification is the feeding of hot synthesis gas directly to the hydrogasifier. On the basis of equilibrium calculations and the availability of electric power to the gasifiers at process voltage at 3-3.5 mills/kwhr, a 20-year average gas price of 50¢ to 51¢/million Btu is possible. This includes by-product credit for sulfur and coal fines.

During the month no inventions were made in the course of the work.

Approved Jack Huebler Signed Frank Schora
Jack Huebler, Associate Director Frank Schora, Assistant Director

..PROGRAM FOR DEVELOPMENT OF IGT

OCR: Contract No. 14-01-0001-381

1964: Aug, Sep, Oct, Nov, Dec
1965: Jan, Feb, Mar, Apr, May, Jun, Jul (1st Year); Aug, Sep, Oct, Nov, Dec, Jan, Feb (2nd Year)

PHASE I

a. Hydrogasification

1. Establish rate-controlling areas of operation
E-13
T-36
DS-$15,800
ODC- $5,500

2. Study effect of degree of pretreatment
E- 6
T-18
DS-$7,500
ODC-$2,500

3. Study effects of variables to establish kinetics of fixed-bed system
E-13
T-36
DS-$15,800
ODC- $4,000

4. Study fluidization and establish rates for fluid-bed operation
E-10½
T-27
DS-$12,600
ODC- $3,000

5. Study H2O-O2-C System
E-3
T-9
DS-$3,000
ODC-$1,000

6. Study particle size effect for both fixed and fluid-bed operatio
E- 9
T-27
DS-$11,300
ODC- $2,000

Stud

b. Coal Pretreatment

1. Modify coal pretreatment unit
E- 3
T-11
DS-$4,250
ODC-$6,000

2. Study effects of pretreatment variables on char properties
E- 4½
T- 22½
DS-$10,400
ODC- $4,000

3. Establish pretreatment prop
E-
T-27
DS-$1
OCD-

Produce material required for hydrogasification runs

c. Methanation

1. Review state of the art, and design lab test unit
E-7
T-0
DS-$3,800
ODC-0

2. Build and shake down lab unit
E-3
T-9
DS-$4,150
ODC-$3,500

3. Lab scale catalyst evaluation
E- 5
T-12
DS-$6,850
ODC-$1,250

4. Study effects of gas composition
E- 5
T-10
DS $6,150
ODC-$1,000

5. Design and construct small pilot unit
E- 5
T-18
DS-$8,500
ODC-$18,500

d. Coal Characterization

1. Develop techniques and study petrographic characteristics of coal, char, and spent hydrogasified residue. Determine heats of reaction of coal and chars
E-13
T- 8
DS-$8,900
ODC- $20,500

2. Establish tests and develop estimation of worth of coal
E-14
T-11
DS-$19,
ODC-$50

PHASE II

a. Analysis of Experimental Data

1. Analyze and correlate data from hydrogasification, coal pretreatment, and methanation work, and recommend conditions for additional tests. Develop kinetic equations to use in reactor design
E-32
T- 0
DS-$13,800
ODC- 0

b. Process Concept Development

1. Develop simple math model of process based on present data
E-5
T-0
DS-$2,300
ODC-0

2. Improve and expand model when additional data becomes available and determine areas where additional data are needed. Modify process concept as necessary and keep process flow-sheets updated
E-20
T- 0
DS-$9,200
ODC-$1,500

c. Reactor Design Studies

Make prelim process conce

d. Economic Evaluation

1. Cost data review and extension
E-7
T-0
DS-$3,200
ODC-0

2. Develop simple flow sheet
E-2
T-0
DS-$900
ODC-0

3. Detailed flow sheet, energy and material balances
E-3
T-0
DS-$1,400
ODC-0

4. Process design of various sections
E-4
T-0
DS-$1,800
ODC-0

5. Cost estimate of equipment, plant and operating expenses
E-3
T-0
DS-$1,400
ODC-0

6. Report Preparation
E-2
T-0
DS-$900
ODC-0

7. Revision and updating of flowsheet
E-6
T-0
DS-$2,800
ODC-0

PHASE III

a. Preliminary Pilot Plant Design

TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)

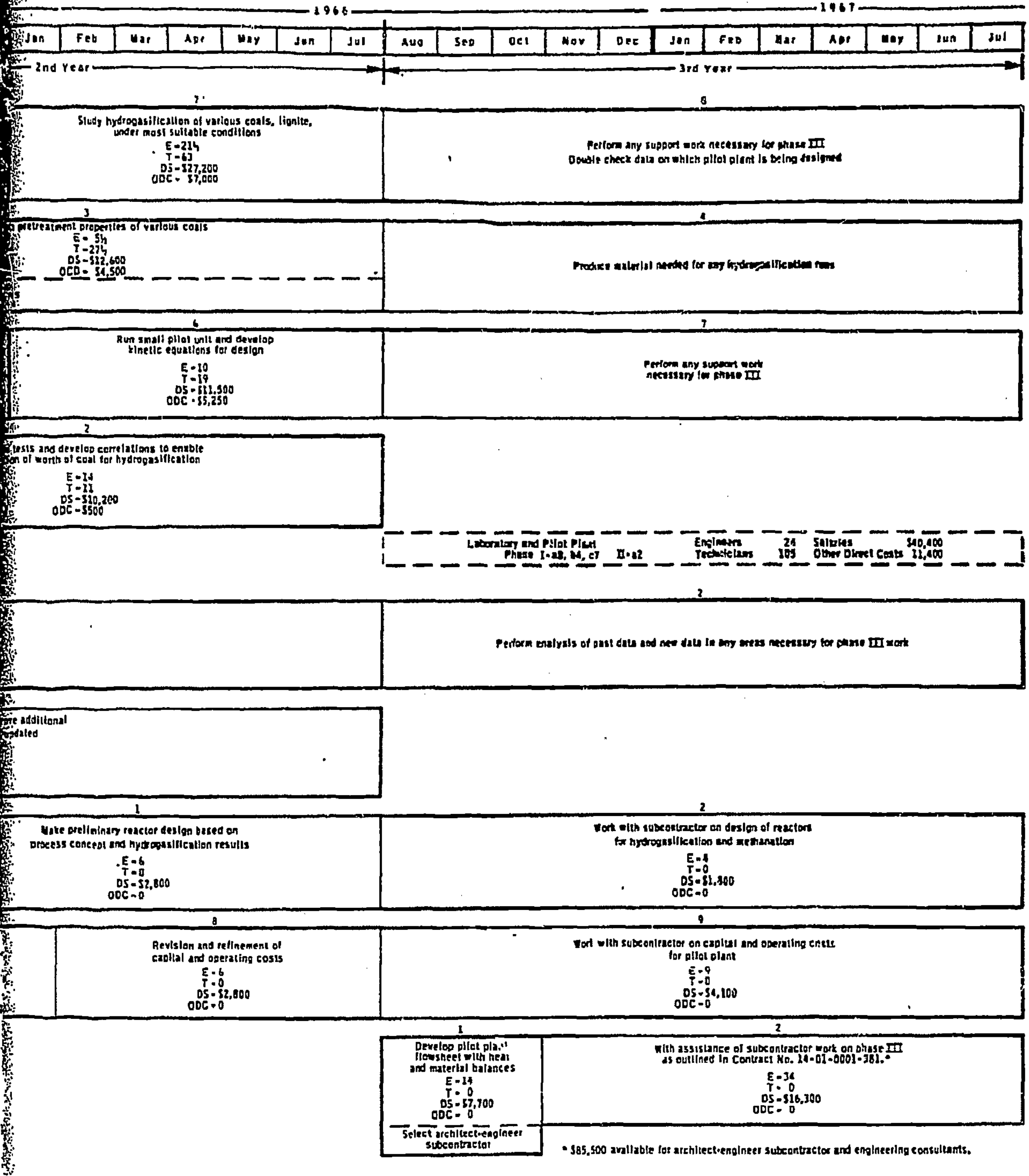
	TOTAL																			
Planned	855,400	23,500	23,500	23,500	23,800	22,400	24,100	24,300	24,700	23,700	23,300	23,800	30,300	29,300	27,800	27,400	28,100	28,100	23,300	23,300
Actual		10,000	15,000	23,300	14,500	23,000	21,100	19,800	28,900	23,400	30,200	24,500	22,900	25,500	21,900	26,900	27,800	22,300	23,400	22,300
Cumulative		10,000	25,000	48,300	62,800	85,800	106,900	126,700	155,600	179,000	209,200	233,700	256,600	282,100	304,000	330,900	358,700	381,000	404,400	426,70

Manpower shown in total man months: E - Engineers
T - Technicians

Costs shown are costs to OCR or one-half of total

GT HYDROGASIFICATION PROCESS

01-381 A.G.A. .PB-23a



Study hydrogasification of various coals, lignite, under most suitable conditions
E-21½
T-63
DS-$27,200
ODC- $7,000

Perform any support work necessary for phase III
Double check data on which pilot plant is being designed

pretreatment properties of various coals
E- 5½
T-27½
DS-$12,600
ODC- $4,500

Produce material needed for any hydrogasification runs

Run small pilot unit and develop kinetic equations for design
E-10
T-19
DS-$11,500
ODC-$5,250

Perform any support work necessary for phase III

tests and develop correlations to enable on of worth of coal for hydrogasification
E-14
T-11
DS-$10,200
ODC-$500

Laboratory and Pilot Plant Phase I-a8, b4, c7 II-a2
Engineers 24 Salaries $40,400
Technicians 105 Other Direct Costs 11,400

Perform analysis of past data and new data in any areas necessary for phase III work

re additional pdated

Make preliminary reactor design based on process concept and hydrogasification results
E-6
T-0
DS-$2,800
ODC-0

Work with subcontractor on design of reactors for hydrogasification and methanation
E-4
T-0
DS-$1,800
ODC-0

Revision and refinement of capital and operating costs
E-6
T-0
DS-$2,800
ODC-0

Work with subcontractor on capital and operating costs for pilot plant
E-9
T-0
DS-$4,100
ODC-0

Develop pilot plant flowsheet with heat and material balances
E-14
T- 0
DS-$7,700
ODC- 0

Select architect-engineer subcontractor

With assistance of subcontractor work on phase III as outlined in Contract No. 14-01-0001-381.*
E-34
T- 0
DS-$16,300
ODC- 0

* $85,500 available for architect-engineer subcontractor and engineering consultants.

23,300	23,300	23,300	23,300	23,300	23,300	23,300	15,800	15,800	15,800	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700
23,400	22,300	25,000	32,600	25,000	26,600	27,200	22,900	23,700	19,200	21,800	21,400	22,500	39,200	40,800				
404,400	426,700	451,700	484,300	509,300	535,900	563,100	586,000	609,700	628,900	650,700	672,100	695,000	734,200	775,000				

or one-half of total costs. DS - Direct Salaries, $
ODC - Other Direct Costs, $

2

IGT-MPR--4/67

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - April 1967

to

Office of Coal Research
Contract No. 14-01-0001-381

Summary

1. Six hydrogasification runs were conducted during the month. All ran until their scheduled shutdown.
2. Initial runs with a synthesized raw electrothermal reactor-type gas (no preshifting) indicated that operation is similar to that with a hydrogen-steam mixture.
3. Three pretreatment runs were made with West Virginia Sewell medium-volatile-content bituminous coal. Hot spots in the bed developed in two of these runs. The third run produced sufficient material for hydrogasification runs with this coal. The present series of pretreatment runs has been concluded.
4. A life study on our selected methanation catalyst was voluntarily terminated after 1419 hours on-stream. No decrease in activity was noted during the run.
5. A preliminary economic analysis indicates a gas price of 38¢ per million Btu based on 8¢ per million Btu lignite.

Hydrogasification

We conducted six hydrogasification tests this month in the balanced-pressure pilot unit. Three tests (Runs HT-168, HT-169, and HT-171) were conducted with lightly pretreated Pittsburgh No. 8 seam bituminous coal from the Ireland mine and a simulated producer gas, with steam as the gaseous feed medium. One test (Run HT-173) was conducted with the same lightly pretreated Pittsburgh No. 8 seam coal, but with a hydrogen-steam feed gas. Two tests (Runs HT-170 and HT-172) were conducted with a lightly pretreated West Virginia Sewell seam medium-volatile-content bituminous coal. The feed gas in these tests was a hydrogen-steam mixture. All six tests were successful.

Run HT-168 was the first test in a series conducted in the pilot unit with a producer gas-steam mixture as feed gas. The simulated producer gas was prepared by mixing hydrogen, carbon monoxide, and carbon dioxide into high-pressure storage. Mole fractions of these gases were as follows: hydrogen 58%, carbon monoxide 37%, and carbon dioxide 5%. The objective of this test was to study the effect of a typical producer gas on carbon gasification and on the hydrogasification product gas heating value. The Pittsburgh seam bituminous coal was fed at a nominal rate of 52 lb/hr and reacted with 530 SCF/hr of the simulated producer gas and 25.3 lb/hr of steam. Tests were conducted with 3-1/2-ft and with 7-ft fluidized coal beds in this run. Although we wanted a coal bed temperature of 1700°F, a temperature of 1600°F was actually realized. The reasons for this were the relatively low feed gas preheat temperature of 1000°F, and inoperation of the lowest reactor furnace heating zone. The run lasted 4-1/2 hours. Preliminary results showed no significant difference in the results of the operations at the two investigated coal bed levels. At both coal bed levels about 20% of the carbon was gasified, and a product gas of 460-465 Btu/SCF (nitrogen-free basis) was produced. About 57% of the carbon monoxide in the feed gas was shifted to hydrogen.

Operating conditions of Run HT-169 were similar to those of Run HT-168. The objective of this test was to get the coal bed temperature up to 1700°F. To do this, the bottom of the coal bed was raised 31 inches higher than its position in Run HT-168 so that it would not be affected by the inoperative heating zone at the bottom of the reactor tube. Also, the feed gas preheat temperature was increased to 1200°F. The coal bed, which was maintained at 3-1/2 ft throughout this test, reached temperatures of 1650°-1675°F. The run lasted 5-1/2 hours, then terminated when the coal feed supply was used up. Based on the as yet incomplete analytical results, hydrogasification results of this test are not significantly different from those of Run HT-168.

We conducted a third test with the simulated producer gas and pretreated Pittsburgh seam bituminous coal (Run HT-171) at conditions conducive to higher carbon conversions. The coal feed rate was a nominal 26 lb/hr so that with a dry producer feed gas rate of 530 SCF/hr, the hydrogen/coal ratio was 25% of the stoichiometric ratio. Producer gas composition was similar to that of Run HT-168. Steam concentration in the feed gas was 50 mole percent. The run lasted 6 hours, with 2-1/4 hours of this at steady state. Preliminary results indicate only a marginal increase in the carbon gasified over that of Runs HT-168 and HT-169.

Since we experienced difficulty in getting the coal bed temperature up to 1700°F in the three tests with simulated producer gas, we wanted to establish with some certainty what effect the inoperation of the lowest reactor heating zone had on this. Run HT-173 was therefore conducted with pretreated Pittsburgh seam coal and a hydrogen-steam feed gas mixture. The lowest reactor heating zone was not operating. Coal and gas feed rates were similar to those of Run HT-168. Temperatures of 1700°-1800°F were easily maintained in the 3-1/2-ft fluidized coal bed throughout the test. This indicates that in the tests with the simulated producer gas, the exothermic hydrogasification reaction rates, at the conditions investigated, were too low to hold the coal bed temperature at 1700°F.

The study of the hydrogasification of a medium-volatile-content bituminous coal from the West Virginia Sewell seam was resumed in tests conducted in Runs HT-170 and HT-172. In previous tests with this coal, pretreatment was inadequate, and the coal agglomerated in the hydrogasifier. The batch of coal prepared for the currently reported runs was pretreated to a sufficient degree so that it did not agglomerate. In both tests, the coal was reacted in a 3-1/2-ft fluidized bed, controlled to 1700°-1800°F, with a hydrogen-steam feed gas mixture. In Run HT-170, the hydrogen/coal ratio was 25% of the stoichiometric ratio, and the steam concentration was 30 mole percent. In Run HT-172, the hydrogen/coal ratio was reduced to 20% of the stoichiometric ratio, and the steam concentration was increased to 50 mole percent. Duration of both runs was about 5 hours with 2 to 2-1/2 hr of this at steady state.

Coal Pretreatment

During the month three pretreatment tests were made in the fluidized-bed pretreater to study minimum pretreatment conditions and to supply the hydrogasification unit with nonagglomerating feed char. We also used the pretreater as a fluidized drier for a subbituminous coal. Results are shown in Table 1.

Table 1. NOMINAL CONDITIONS FOR PRETREATMENT TESTS

Run No.	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Avg Bed Temp, °F
FP-119*	0.30	1200	21.0	835
FP-119A*	0.30	1100	21.0	830
FP-120	0.37	1188	21.0	817

* Intended run conditions, but not attained.

West Virginia Sewell medium-volatile-content bituminous coal, crushed and screened to –16+80 mesh, was used for all three runs. We dried the Colorado subbituminous coal, crushed and screened to –16+80, in the pretreater in an extended run. This coal was from Colorado's Erie District No. 16; it was obtained from Imperial Coal Co.'s Eagle mine.

In Run FP-119 we attempted to overcome temperature-control problems by cutting down on the air feed rate and the bed temperature. The run was shut down before steady state was attained when a hot spot occurred at the bottom of the bed. In Run FP-119A we again tried to control temperature by further cutting the air feed rate and the bed temperature, but again we were unsuccessful.

The feed for Run FP-120 was lightly caked residue from Runs FP-117, FP-118, and FP-119. The run, which lasted 8-1/4 hours, produced 800 lb of free-flowing char for the hydrogasifier. We decided to suspend further testing of the Sewell coal because Run FP-120 produced enough char for hydrogasifier testing. The Sewell coal can be made free-flowing by reducing the volatile matter to a conservative level of less than 15%.

The pretreater was run continuously for 32 hours as a fluidized drier for the Colorado subbituminous coal. The coal was dried from 20.6% to 3.1% moisture content at 225°F, using hot nitrogen as the fluidizing gas.

This completes the pretreatment program. No more runs are planned at present except to supply the hydrogasification unit with feed as needed.

High-Pressure Methanation

The life study with Harshaw Ni-0104T 1/8-in. catalyst was ended after 1419 hours on-stream. No change in activity was noted during the study. At 1100 hours the feed gas space velocity was raised from 5000 hr^{-1} to 6800 hr^{-1}. At 1200 hours it was increased further to 8700 hr^{-1}. CO and C_2H_6 conversions remained essentially complete after these two space velocity increases. There was some CO_2 breakthrough, however; about 0.4% CO_2 was found in the product at 8700 hr^{-1} space velocity. The feed gas contained about 1.0% CO_2. We plan to save this catalyst for possible further testing. The life study system is now to be used to evaluate a hydrodesulfurization catalyst.

The stirred reactor runs have indicated that a pore diffusion resistance becomes important at higher temperatures for large catalyst pellets (1/4 in.). We crushed 1/4-in. pellets of nickel-on-kieselguhr catalyst Ni-0101 (which is the unreduced form of Ni-0104) to check the effect of particle size on the methanation rate. This effect appears to become unimportant at 550°F for particle sizes in the range between 20 and 40 mesh. The use of particles of this size will allow us to study the true methanation kinetics.

Calorimetry

We completed the determination of the heat of reaction of hydrogen and North Dakota lignite and that of hydrogen and partially gasified North Dakota lignite at 1500°F and 1000 psia. We also completed the calibrations required for the determination of the heat capacity of coal and chars at 1500°F. The measurements of the heat capacity of Ireland mine coal and chars continue.

Process Economics

The study of the economics of producing hydrogen by means of an electrothermal fluidized bed is continuing. A rough cost estimate was made for the production of gas from lignite at a 250 million CF/day level. Based on 8¢/million Btu lignite price the cost of producing gas is approximately 38¢/million Btu.

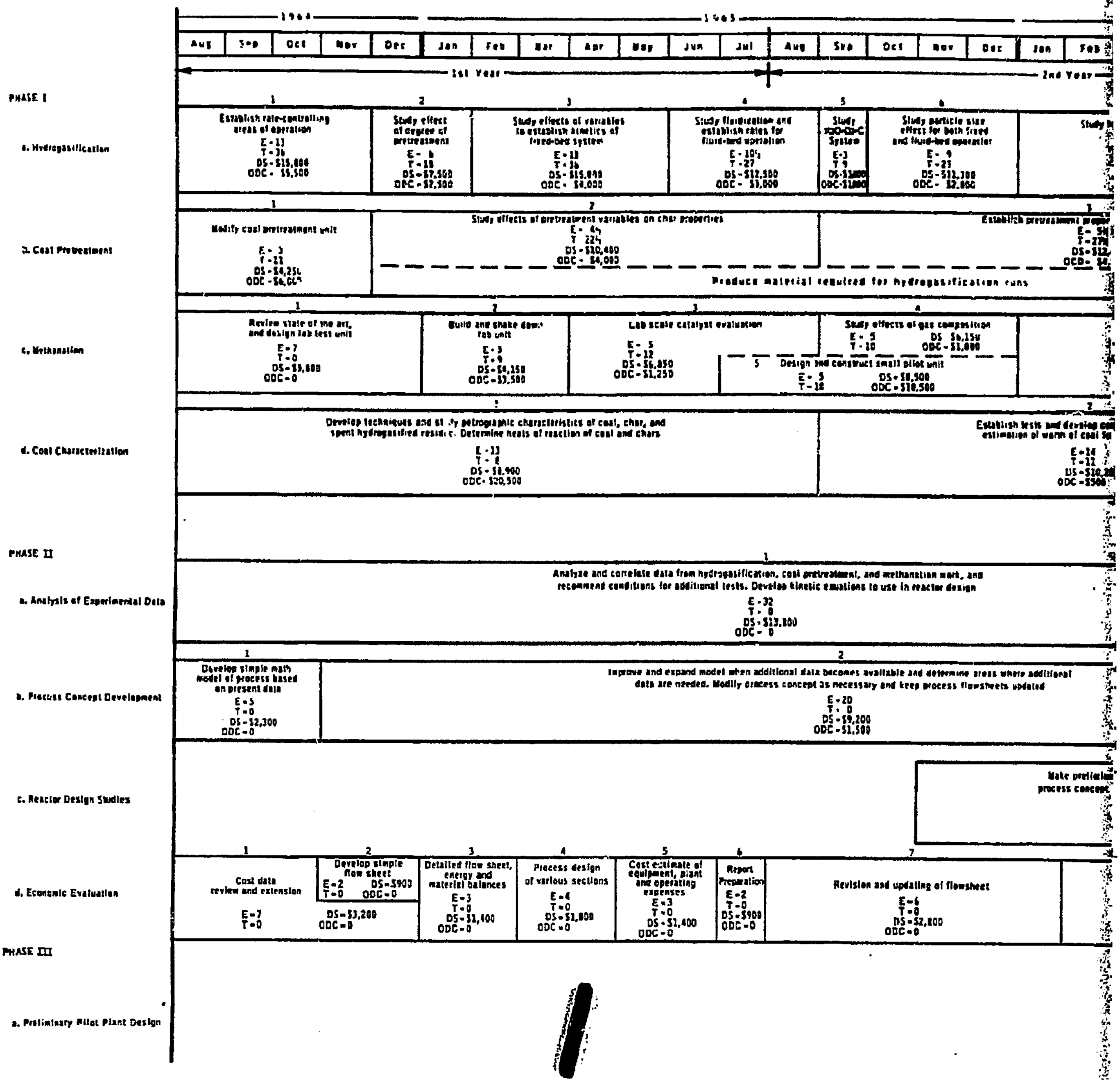
The program is now about 1-1/2 months ahead of schedule. Based on the present rate of effort, funding under the current contract will be exhausted in mid-June.

During the month no inventions were made in the course of the work.

Approved Jack Huebler Signed Frank Schora
Jack Huebler, Associate Director Frank Schora, Assistant Director

PROGRAM FOR DEVELOPMENT OF IGT

OCR: Contract No. 14-01-0001-381



PHASE I

a. Hydrogasification

- 1 Establish rate-controlling areas of operation E - 11 T - 36 DS - $15,800 ODC - $5,500
- 2 Study effect of degree of pretreatment E - 6 T - 18 DS - $7,500 ODC - $2,500
- 3 Study effects of variables to establish kinetics of fixed-bed system E - 13 T - 36 DS - $15,800 ODC - $4,000
- 4 Study fluidization and establish rates for fluid-bed operation E - 10½ T - 27 DS - $12,500 ODC - $3,000
- 5 Study H₂O-CO₂-C System E-3 T 9 DS-$1,800 ODC-$1,000
- 6 Study particle size effect for both fixed and fluid-bed operation E - 9 T - 23 DS - $11,300 ODC - $2,800
- Study

b. Coal Pretreatment

- 1 Modify coal pretreatment unit E - 3 T - 22 DS - $4,250 ODC - $6,000
- 2 Study effects of pretreatment variables on char properties E - 4½ T 22½ DS - $10,400 ODC - $4,000
- 3 Establish pretreatment proper E - 5 T - 27 DS - $12, ODC - $4,
- Produce material required for hydrogasification runs

c. Methanation

- 1 Review state of the art, and design lab test unit E - 7 T - 0 DS - $3,800 ODC - 0
- 2 Build and shake down lab unit E - 3 T - 9 DS - $4,150 ODC - $3,500
- 3 Lab scale catalyst evaluation E - 5 T - 12 DS - $6,850 ODC - $1,250
- 4 Study effects of gas composition E - 5 T - 10 DS $6,150 ODC - $1,000
- 5 Design and construct small pilot unit E - 5 T - 18 DS - $8,500 ODC - $10,500

d. Coal Characterization

- 1 Develop techniques and study petrographic characteristics of coal, char, and spent hydrogasified residue. Determine heats of reaction of coal and chars E - 13 T - 8 DS - $8,900 ODC - $20,500
- 2 Establish tests and develop co estimation of warm of coal fo E - 14 T - 12 DS - $10,2 ODC - $500

PHASE II

a. Analysis of Experimental Data

- 1 Analyze and correlate data from hydrogasification, coal pretreatment, and methanation work, and recommend conditions for additional tests. Develop kinetic equations to use in reactor design E - 32 T - 0 DS - $13,100 ODC - 0

b. Process Concept Development

- 1 Develop simple math model of process based on present data E - 5 T - 0 DS - $2,300 ODC - 0
- 2 Improve and expand model when additional data becomes available and determine areas where additional data are needed. Modify process concept as necessary and keep process flowsheets updated E - 20 T - 0 DS - $9,200 ODC - $1,500

c. Reactor Design Studies

- Make prelimin process concept

d. Economic Evaluation

- 1 Cost data review and extension E - 7 T - 0 DS - $3,200 ODC - 0
- 2 Develop simple flow sheet E - 2 T - 0 DS - $900 ODC - 0
- 3 Detailed flow sheet, energy and material balances E - 3 T - 0 DS - $1,400 ODC - 0
- 4 Process design of various sections E - 4 T - 0 DS - $1,800 ODC - 0
- 5 Cost estimate of equipment, plant and operating expenses E - 3 T - 0 DS - $1,400 ODC - 0
- 6 Report Preparation E - 2 T - 0 DS - $900 ODC - 0
- 7 Revision and updating of flowsheet E - 6 T - 0 DS - $2,800 ODC - 0

PHASE III

a. Preliminary Pilot Plant Design

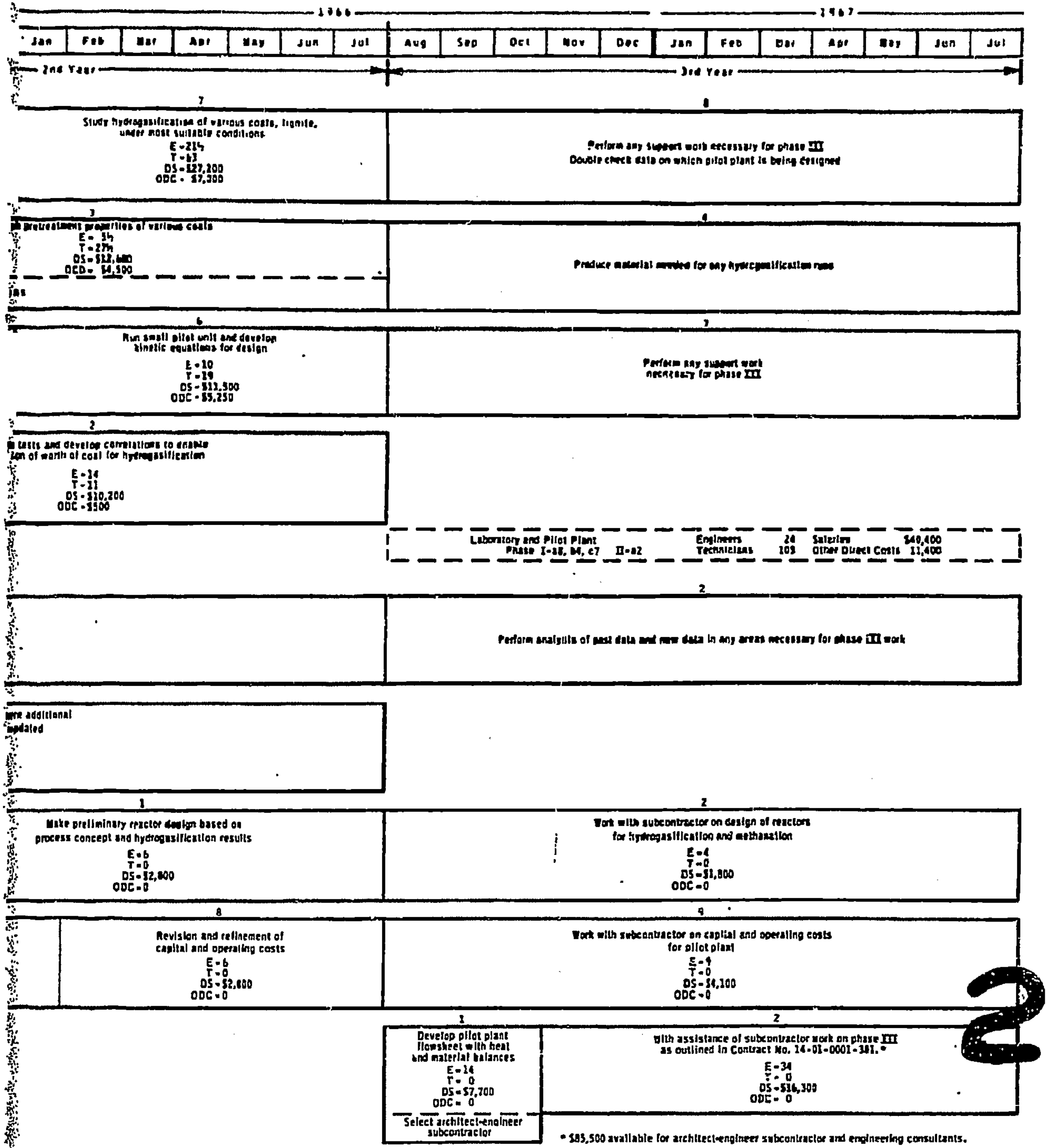
	TOTAL	TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)																		
Planned	855,400	23,500	23,500	23,500	23,800	22,400	24,100	24,300	24,700	23,700	23,300	23,800	30,300	29,300	27,800	27,400	28,100	28,100	23,300	23,300
Actual		10,000	15,000	23,300	14,500	23,000	21,100	19,800	28,900	23,400	30,200	24,500	22,900	25,500	21,900	26,900	27,800	22,300	23,400	22,300
Cumulative		10,000	25,000	48,300	62,800	85,800	106,900	126,700	155,600	179,000	209,200	233,700	256,600	282,100	304,000	330,900	358,700	381,000	404,400	426,70

Manpower shown in total man months: E = Engineers
T = Technicians

Costs shown are costs to OCR or one-half of total

-GT HYDROGASIFICATION PROCESS

01-381 A.G.A. PB-23a



Study hydrogasification of various coals, lignite, under most suitable conditions
E - 21½
T - 63
DS - $27,200
ODC - $7,300

Perform any support work necessary for phase III
Double check data on which pilot plant is being designed

pretreatment properties of various coals
E - 3½
T - 27½
DS - $12,680
ODC - $4,500

ies

Produce material needed for any hydrogasification runs

Run small pilot unit and develop kinetic equations for design
E - 10
T - 19
DS - $13,300
ODC - $5,250

Perform any support work necessary for phase III

tests and develop correlations to enable ion of worth of coal for hydrogasification
E - 14
T - 11
DS - $10,200
ODC - $500

Laboratory and Pilot Plant
Phase I-a8, b4, c7 II-a2
Engineers 24
Technicians 109
Salaries $40,400
Other Direct Costs 11,400

Perform analysis of past data and new data in any areas necessary for phase III work

re additional
updated

Make preliminary reactor design based on process concept and hydrogasification results
E - 6
T - 0
DS - $2,800
ODC - 0

Work with subcontractor on design of reactors for hydrogasification and methanation
E - 4
T - 0
DS - $1,800
ODC - 0

Revision and refinement of capital and operating costs
E - 6
T - 0
DS - $2,800
ODC - 0

Work with subcontractor on capital and operating costs for pilot plant
E - 9
T - 0
DS - $4,100
ODC - 0

Develop pilot plant flowsheet with heat and material balances
E - 14
T - 0
DS - $7,700
ODC - 0

Select architect-engineer subcontractor

With assistance of subcontractor work on phase III as outlined in Contract No. 14-01-0001-381.*
E - 34
T - 0
DS - $16,300
ODC - 0

* $85,500 available for architect-engineer subcontractor and engineering consultants.

2

23,300	23,300	23,300	23,300	23,300	23,300	23,300	15,800	15,800	15,800	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700
23,400	22,300	25,000	32,600	25,000	26,600	27,200	22,900	23,700	19,200	21,800	21,400	22,900	39,200	40,800	36,800			
404,400	426,700	451,700	484,300	509,300	535,800	563,100	586,000	609,700	628,900	650,700	672,100	695,000	734,200	775,000	811,800			

r one-half of total costs, DS - Direct Salaries, $
ODC - Other Direct Costs, $

IGT-mPR--5/67

DEVELOPMENT OF THE IGT HYDROGASIFICATION PROCESS

Progress Report — May 1967
to
Office of Coal Research
Contract No. 14-01-0001-381

Summary

1. Eight hydrogasification tests were made during the month.
2. West Virginia Sewell seam coal has been run successfully with pretreatment.
3. Colorado subbituminous coal has been run successfully without pretreatment although it does show some agglomerating tendencies under certain conditions.
4. A hydrodesulfurization catalyst has been established as being a poor methanation catalyst — a desirable feature for a desulfurization system.
5. Work on the preliminary design of the hydrogasification pilot plant is on schedule.

Hydrogasification

We conducted eight hydrogasification tests this month in the balanced-pressure pilot unit. One of these tests (Run HT-174) was the last of the current series conducted with lightly pretreated West Virginia Sewell seam medium-volatile-content bituminous coal. The seven other tests (Runs HT-175 through HT-181) were conducted with an untreated, but dried, Colorado subbituminous coal from the Eagle mine. Two of the eight tests were successful: the test with the West Virginia coal and one of the tests with the Colorado coal. Two other tests with the Colorado coal were partially successful. In the other four tests with this coal, the coal agglomerated before steady-state operation could be established.

The current studies of the hydrogasification of a medium-volatile-content bituminous coal from the West Virginia Sewell seam were completed with Run HT-174. In this test the lightly pretreated coal was reacted in a 3-1/2-ft fluidized bed with a hydrogen-steam feed gas mixture. The coal was fed at a rate of 48 lb/hr and reacted with 530 SCF/hr of hydrogen and 25 lb/hr of steam. At these flow conditions the hydrogen/coal ratio was 25% of the stoichiometric ratio, and the steam concentration in the feed gas was 50 mole percent. The run lasted 4-3/4 hours, 2 hours of this time at steady state. A product gas with a heating value of 496 Btu/SCF (nitrogen-free basis) was produced as 28% of the carbon was gasified.

We initiated studies of the hydrogasification of untreated Colorado subbituminous coal in the reactor with Run HT-175. Before using this coal it was dried from an as-received moisture content of 19.5% to one of 3.1%. When tested in the laboratory it showed only minimal tendencies to agglomerate. Operating conditions of Run HT-175 called for reaction of the coal in a 3-1/2-ft fluidized bed with hydrogen and steam. The steam concentration in the feed gas was 50 mole percent, and the hydrogen/coal ratio was 25% of stoichiometric. Shortly after the 3-1/2-ft coal bed was established, the coal began to agglomerate in the reactor tube and would not discharge. The coal feed rate at this time was only one-half of the planned rate of 55 lb/hr; the elapsed feeding time was 2 hours. Agglomeration of the coal started near the top of the feed gas distributor.

For Run HT-176, the planned flow rates were similar to those of Run HT-175. However, the start-up procedure was modified so that free-fall operation at the full flow rates would be established first before continuing on to a fluidized-bed operation. After 55 minutes of feeding coal, the test had to be terminated when the coal agglomerated at the end of the coal injection tube. A reduced purge nitrogen flow rate through the coal injection, occassioned by a moderate-size leak in the coal feed hopper vent line, allowed the coal to hang up in the injection tube and agglomerate.

For the next run with the Colorado coal, Run HT-177, the top heating zone of the reactor was controlled to 1200°F instead of 1300°F as in the previous runs. This would reduce the rate at which the coal was being preheated and the rate of the hydrogasification reaction at the top of the reactor. Agglomeration of the coal at the end of the coal feed tube was again experienced in this test after 48 minutes of feeding coal. Partially responsible for this agglomeration was the inoperation of the mechanically driven spiral stirrer used for promoting the flow of coal through the coal injection tube.

We obtained partially successful hydrogasification operation with the Colorado coal in Run HT-178. Run conditions were similar to those of Run HT-175. The run was started up with the coal being reacted in free-fall as it was fed at 30 lb/hr. Holdup of the coal in the reactor was indicated by the coal bed level gage 1-3/4 hr after coal feeding was started. A coal bed began to build up as the coal would no longer flow through the reactor. Operation was continued for another hour till the coal bed reached 11.25 ft. The run was shut down when the coal would not discharge from the reactor. Responsible for the coal holdup was the accumulation of oversized agglomerated coal particles on the discharge screw. There was no evidence of bridging in the reactor by agglomerated coal.

For Run HT-179 we lowered the position of the hydrogen feed distributor by 31 inches in order to obtain a longer effective free-fall reactor length. We also modified the operation conditions from those used in the previous tests. The upper two reactor heating zones were controlled to 1200°F. The hydrogen/coal ratio was increased to 35% of stoichiometric and the steam concentration was reduced to 30 mole percent. Flow rates for this run were 55 lb/hr coal, 743 SCF/hr hydrogen, and 15 lb/hr steam. The run was started and continued with the coal being reacted in free-fall. The run was completely successful as no difficulties developed with agglomeration of the coal or with coal feeding and discharging. It was terminated after 6 hours when the coal feed supply was depleted. Two free-fall steady-state operating conditions were obtained in this run, one at a coal feed rate of 30 lb/hr and one at 55 lb/hr.

After demonstrating successful free-fall operation with untreated Colorado subbituminous coal we resumed our attempts, in Run HT-180, at reacting this coal in a fluidized bed. Flow conditions and start-up were similar to those of Run HT-179. Successful free-fall operation at full flow rates was established and continued for 1/2 hr. The rate of coal discharge from the reactor was then reduced so that a coal bed would be established. Soon after, this further discharging of coal ceased as coal began to hold up in the reactor. A coal bed of 7 ft was reached when the coal feed screw stopped. Agglomeration of coal near the coal injection tube choked off flow from the tube and jammed the feed screw. The run lasted 2-1/2 hours, 30 minutes of this at fluid-bed conditions.

To minimize chances for coal agglomeration at the top of the reactor for Run HT-181, we set the control temperature of the top reactor heating zone at 1100°F. This would lower the rate of coal preheat and move the zone of hydrogasification reaction away from the coal feed tube. Conditions for this run were similar to those of Run HT-179. The test was started with the coal fed at 30 lb/hr and reacted in free-fall with 530 SCF/hr of hydrogen and 6 lb/hr steam. Within 1 hour after the start, the hydrogen rate was increased to 650 SCF/hr, and the steam rate to 15 lb/hr. Before any increase in the coal feed rate was made, the coal feeder jammed and the run had to be terminated. Coal had agglomerated in the reactor tube near the coal feed tube and stopped the coal flow. There was no coal agglomeration in the lower sections of the reactor. It is difficult to establish the influence of the hydrogen/coal ratio on the agglomeration of the Colorado coal experienced in Runs HT-180 and HT-181. In both of these runs, the hydrogen/coal ratio was over 55% of stoichiometric. There is a possibility that the availability of hydrogen was sufficiently high to extend the hydrogasification zone into the upper parts of the reactor, accompanied by a temperature increase of the upper zones of the reactor.

Coal Characterization

Calorimetry

The heat capacity of coal and chars was determined at 1500°F. Measurements continued at 1300°F.

Petrography

Work on the petrographic examination of coals, pretreatment products, and hydrogasification residues continued. Mounting and polishing work on the five high-volatile-content bituminous coals is nearly completed. Point-count analyses of the maceral composition of coal from the Ireland mine, Broken Arrow mine, and No. 5 Block were made. Photomicrographs of the Broken Arrow and No. 5 Block coals were obtained.

Methanation

We have begun a more detailed study of the kinetics of the reactions involved in methanation. The study consists of systematic variation of total pressure and partial pressures of the components under low conversion conditions. The effects of these changes on the reaction rate allow us to develop a more reliable mathematical model of the kinetics. To attain low conversions, a differential reactor system has been devised. Converting the present reactor system to this system required minor equipment modifications.

The first hydrodesulfurization tests with 13% CO feed show that the methanation rate on cobalt-molybdate catalyst is low. Desulfurization data have not been fully analyzed.

Girdler has sent a sample of G-41 chromia-alumina desulfurization catalyst for our tests. This catalyst converts organic sulfur compounds to H_2S in the presence of appreciable concentrations of CO and CO_2. It has special application for removal of COS and CS_2 from producer or coke oven gas.

Engineering Economics

Preparation continued of the detailed report on the economics of pipeline gas production with hydrogen made by electrothermal gasification of spent char. Tables, graphs, and flow sheets have been drawn, and a draft of the text is largely completed.

Calculations of the economics of pipeline gas production for the CO_2 Acceptor, BCR 2-Stage, and Kellogg Molten Salt processes have been made following the A.G.A. accounting procedure via the computer program. The various calculations have been approved by the respective contractors. These will be tabulated together with IGT's process and submitted to OCR and the other contractors with a set of recommendations for a common bases for costs of synthetic gas plants. This work is an outgrowth of the contractors' meeting January 17-18, 1967.

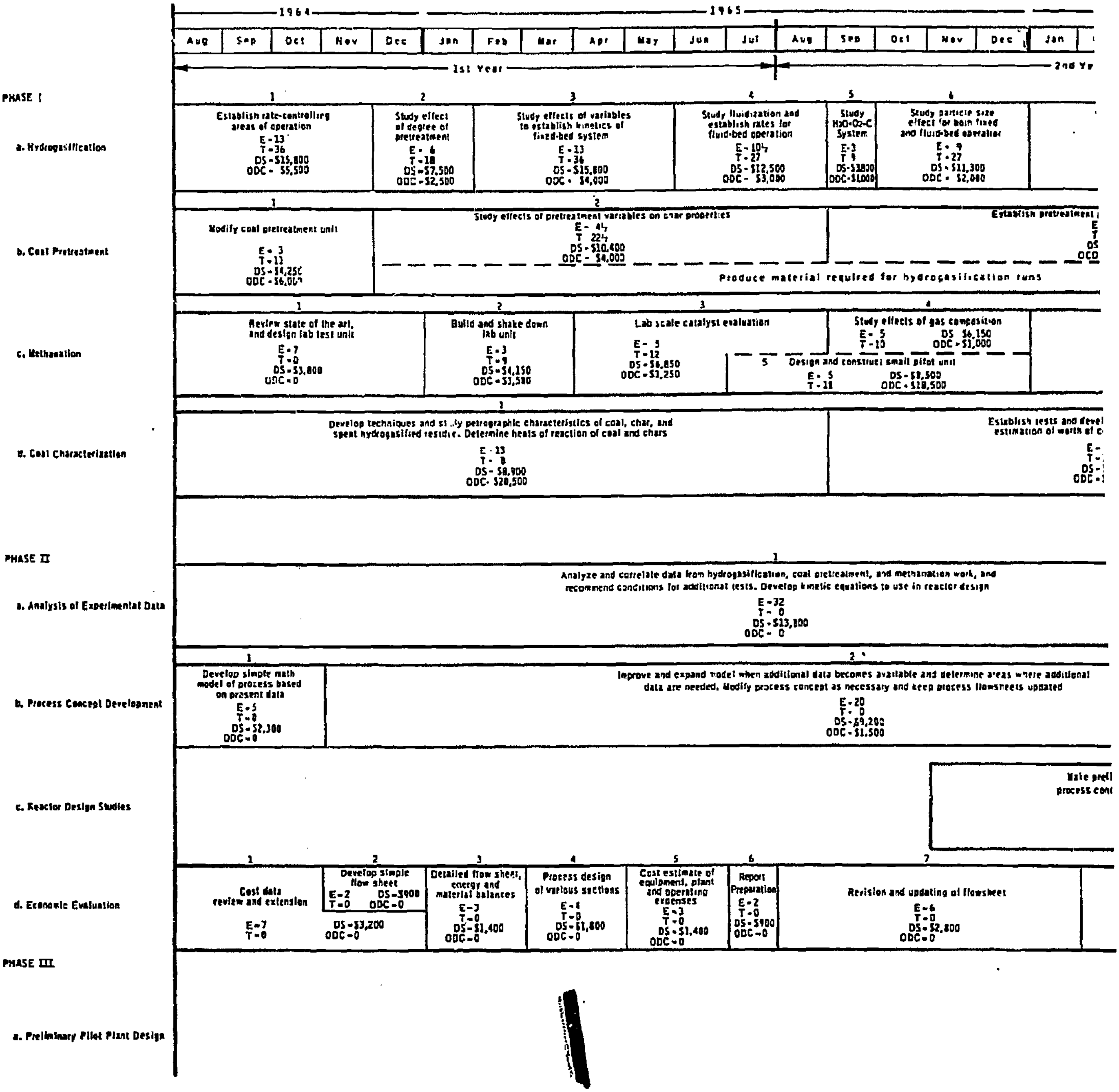
Because of the change in computers from an IBM 1620 to an IBM 1800, the A.G.A. accounting procedure was reprogrammed. It has been made more flexible and the output so arranged that all numbers are clearly labeled, making the calculation suitable for inserting in a report.

No new inventions were made during the course of this work during the month of May.

Approved Jack Huebler Signed Frank Schora
Jack Huebler, Associate Director Frank Schora, Assistant Director

PROGRAM FOR DEVELOPMENT OF IG

OCR: Contract No. 14-01-0001-381



PHASE I

a. Hydrogasification

1 Establish rate-controlling areas of operation
E - 13
T - 36
DS - $15,800
ODC - $5,500

2 Study effect of degree of pretreatment
E - 6
T - 18
DS - $7,500
ODC - $2,500

3 Study effects of variables to establish kinetics of fixed-bed system
E - 13
T - 36
DS - $15,800
ODC - $4,000

4 Study fluidization and establish rates for fluid-bed operation
E - 104
T - 27
DS - $12,500
ODC - $3,080

5 Study H_2O-O_2-C System
E-3
T 9
DS-$3800
ODC-$1000

6 Study particle size effect for both fixed and fluid-bed operation
E - 9
T - 27
DS - $11,300
ODC - $2,080

b. Coal Pretreatment

1 Modify coal pretreatment unit
E - 3
T - 11
DS - $4,250
ODC - $6,000

2 Study effects of pretreatment variables on char properties
E - 44
T 224
DS - $10,400
ODC - $4,000

Establish pretreatment
E
T
DS
OCD

Produce material required for hydrogasification runs

c. Methanation

1 Review state of the art, and design lab test unit
E - 7
T - 0
DS - $3,800
ODC - 0

2 Build and shake down lab unit
E - 3
T - 9
DS - $4,150
ODC - $3,580

3 Lab scale catalyst evaluation
E - 5
T - 12
DS - $6,850
ODC - $3,250

4 Study effects of gas composition
E - 5 DS $6,150
T - 10 ODC - $3,000

5 Design and construct small pilot unit
E - 5 DS - $8,500
T - 18 ODC - $18,500

d. Coal Characterization

1 Develop techniques and study petrographic characteristics of coal, char, and spent hydrogasified residue. Determine heats of reaction of coal and chars
E - 23
T - 8
DS - $8,900
ODC - $20,500

Establish tests and devel
estimation of worth of c
E -
T -
DS -
ODC -

PHASE II

a. Analysis of Experimental Data

1 Analyze and correlate data from hydrogasification, coal pretreatment, and methanation work, and recommend conditions for additional tests. Develop kinetic equations to use in reactor design
E - 32
T - 0
DS - $13,800
ODC - 0

b. Process Concept Development

1 Develop simple math model of process based on present data
E - 5
T - 8
DS - $2,300
ODC - 0

2 Improve and expand model when additional data becomes available and determine areas where additional data are needed. Modify process concept as necessary and keep process flowsheets updated
E - 20
T - 0
DS - $9,200
ODC - $1,500

c. Reactor Design Studies

Make preli
process con

d. Economic Evaluation

1 Cost data review and extension
E - 7
T - 0
DS - $3,200
ODC - 0

2 Develop simple flow sheet
E - 2 DS - $900
T - 0 ODC - 0

3 Detailed flow sheet, energy and material balances
E - 3
T - 0
DS - $1,400
ODC - 0

4 Process design of various sections
E - 4
T - 0
DS - $1,800
ODC - 0

5 Cost estimate of equipment, plant and operating expenses
E - 3
T - 0
DS - $1,400
ODC - 0

6 Report Preparation
E - 2
T - 0
DS - $900
ODC - 0

7 Revision and updating of flowsheet
E - 6
T - 0
DS - $2,800
ODC - 0

PHASE III

a. Preliminary Pilot Plant Design

TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)

	TOTAL																			
Planned	855,400	23,500	23,500	23,500	23,800	22,400	24,100	24,300	24,700	23,700	23,300	23,800	30,300	29,300	27,800	27,400	28,100	28,100	23,300	23,30
Actual		10,000	15,000	23,300	14,500	23,000	21,100	19,800	28,900	23,400	30,700	24,500	22,900	25,500	21,900	28,900	27,900	22,300	25,400	22,30
Cumulative		10,000	25,000	48,300	62,800	85,800	06,900	126,700	155,600	179,000	209,200	233,700	256,600	282,100	304,000	330,900	358,700	381,000	404,400	426,7

Manpower shown in total man months: E - Engineers
T - Technicians

Costs shown are costs to OCR or one-half of tota

IGT HYDROGASIFICATION PROCESS

1-0001-381 A.G.A. PB-23a

1966 1967

2nd Year 3rd Year

tior

7
Study hydrogasification of various coals, lignite, under most suitable conditions
E = 214
T = 63
DS = $27,200
ODC = $7,000

8
Perform any support work necessary for phase III
Double check data on which pilot plant is being designed

3
Establish pretreatment properties of various coals
E = 54
T = 274
DS = $12,600
ODC = $4,500

ion runs

4
Produce material needed for any hydrogasification runs

6
Run small pilot unit and develop kinetic equations for design
E = 10
T = 19
DS = $11,500
ODC = $5,250

7
Perform any support work necessary for phase III

2
Establish tests and develop correlations to enable estimation of worth of coal for hydrogasification
E = 14
T = 11
DS = $10,200
ODC = $500

Laboratory and Pilot Plant
Phase I-a8, b4, c7 II-a2
Engineers 24
Technicians 105
Salaries $40,400
Other Direct Costs 11,400

2
Perform analysis of past data and new data in any areas necessary for phase III work

eas where additional
sheets updated

1
Make preliminary reactor design based on process concept and hydrogasification results
E = 6
T = 0
DS = $2,800
ODC = 0

2
Work with subcontractor on design of reactors for hydrogasification and methanation
E = 4
T = 0
DS = $1,800
ODC = 0

eel

8
Revision and refinement of capital and operating costs
E = 6
T = 0
DS = $2,800
ODC = 0

9
Work with subcontractor on capital and operating costs for pilot plant
E = 9
T = 0
DS = $4,100
ODC = 0

1
Develop pilot plant flowsheet with heat and material balances
E = 14
T = 0
DS = $7,700
ODC = 0

Select architect-engineer subcontractor

2
With assistance of subcontractor work on phase III as outlined in Contract No. 14-01-0001-381.*
E = 34
T = 0
DS = $16,300
ODC = 0

2

* $85,500 available for architect-engineer subcontractor and engineering consultants.

c	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
,100	23,300	23,300	23,300	23,300	23,300	23,300	23,300	15,800	15,800	15,800	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700
300	23,400	22,300	25,000	32,600	25,000	26,500	27,200	22,900	23,700	18,200	21,800	21,400	22,900	39,200	40,800	36,600	37,500		
,000	404,400	426,700	461,700	484,300	509,300	535,900	563,100	586,000	609,700	328,900	650,700	672,100	695,000	734,200	775,000	811,600	849,300		

to OCR or one-half of total costs. DS = Direct Salaries, $
ODC = Other Direct Costs, $

IGT-MPR--6/67

DEVELOPMENT OF THE IGT HYDROGASIFICATION PROCESS

Progress Report - June 1967
to
Office of Coal Research
Contract No. 14-01-0001-381

Summary

1. Four hydrogasification tests using Colorado subbituminous coal were performed during June.
2. Colorado subbituminous coal has been run with complete success only after pretreatment at 650°F with air. Agglomeration without pretreatment is only slight.
3. Work has started on the design of the 100-kw, high-pressure electrothermal hydrogen producer.
4. The initial design study with Bechtel is nearing completion.

Hydrogasification

We conducted four hydrogasification tests this month in the balanced-pressure pilot unit. The hydrogasification performance of Colorado subbituminous coal, from the Eagle mine, was studied in these tests (Runs HT-182 through HT-185). In two of these tests, the feed was an untreated, but dried coal. Performance of the raw coal in these tests was only marginal because of a tendency for light agglomeration. For the two other tests, the coal was lightly pretreated with air at 650°F (Pretreatment Run FP-122). The tests with the pretreated Colorado coal were completely successful as there were no difficulties with coal hold-up or agglomeration.

With Run HT-182 we continued our efforts, started last month, to hydrogasify untreated Colorado subbituminous coal in a fluidized bed. To minimize chances of significant coal agglomeration before the desired feed rates were reached, we controlled the rate of reaction of the coal during start-up. The control temperatures of the top two reactor heating zones were set for 1200°F to reduce the rate of coal preheat. Also, the hydrogen-to-coal ratio was continually adjusted so as not to exceed 30% of the stoichiometric ratio. These measures were effective for 1-1/4 hours, after which the coal began to hang up in the reactor tube and would not discharge. We continued the test for another 1-1/2 hours until agglomeration of the coal near the coal feed tube stopped further coal feeding. During this period, operating data were obtained for a fixed coal bed hydrogasification operation. For this period the coal feed rate was 55 lb/hr, the hydrogen rate was 600 SCF/hr (a hydrogen/coal ratio) of 28% of the stoichiometric ratio), and the steam rate was 15.3 lb/hr (a concentration of 36% in the feed gas).

Flow rates for Run HT-183 were somewhat altered from those of Run HT-182 in an effort to better control the coal reaction rate. The hydrogen-to-coal ratio was set at 25% of the stoichiometric ratio (55 lb/hr of coal and 530 SCF/hr of hydrogen), and the steam rate was set at 50 mole percent of the hydrogen-steam feed mixture (25 lb/hr of steam). We started establishing steady-state free-fall operation at these rates before proceeding to fluid-bed operation. After about 1-1/2 hours of free-fall operation, coal began to hold up in the reactor tube about 2 ft above the hydrogen-steam feed tube. This plug soon broke, and free-fall operation continued. However, 1 hour later, the coal began to agglomerate at the top of the reactor tube where it eventually choked off further coal feed. Approximately 1 hour of free-fall operation at steady flow conditions was obtained in this 3-hour run. During this 1-hour period, the coal rate was 42 lb/hr, the hydrogen rate was 530 SCF/hr, and the steam rate was 25 lb/hr. A screening of the coal residue from the reactor showed that 8.7% of the 22.2 lb removed was of 1/2-in. size or larger, indicating light agglomeration.

The marginal performance of the untreated Colorado subbituminous coal in eight out of the nine tests conducted in the hydrogasification pilot unit indicated that the coal would have to be pretreated for fluid-bed hydrogasification operation. The coal was lightly pretreated in the pilot plant fluidized-bed pretreater with air at a temperature of 650°F. This pretreated coal was used as a feed in Runs HT-184 and HT-185 with completely successful results.

In Run HT-184 we fed the coal at 55 lb/hr and reacted it in a 3-1/2-ft fluidized bed with 530 SCF/hr of hydrogen (25% of the stoichiometric hydrogen/coal ratio) and with 25 lb/hr of steam (50 mole percent of the hydrogen-steam mixture). The bottom of the coal bed was 31 in. above the furnace bottom so that as much of the reactor length as possible would be available for free-fall. The coal bed was controlled to 1700°F, while the reactor above the coal bed was held at 1200°-1300°F. Over 2 hours of steady-state operation was obtained at the planned conditions in this 5-1/2-hour run.

The free-flowing performance of the pretreated Colorado subbituminous coal showed that a long free-fall reactor length was no longer necessary for operation. Accordingly, for Run HT-185 the hydrogen-steam feed tube was returned to the standard level of 62 in. above the bottom of the furnace. The pretreated coal was fed at 78 lb/hr and reacted in a 3-1/2-ft fluidized bed with 745 SCF/hr of hydrogen and 15.1 lb/hr of steam. At these flow conditions, the hydrogen-to-coal ratio was 25% of the stoichiometric ratio, and the steam concentration was 30 mole percent. After nearing 2 hours of steady-state operation, we voluntarily terminated the test. Total operating time with coal feed was 4 hours.

Coal Pretreatment

During the month we conducted two pretreatment tests in the fluidized-bed pilot plant pretreater to destroy the agglomerating tendencies of a Colorado subbituminous coal and make it a suitable hydrogasification feed. Earlier studies at IGT in a laboratory-size pretreater showed that subbituminous coals required only mild pretreatment to destroy their agglomerating characteristics. We therefore pretreated the coal with air at temperatures in the range of 600°-650°F. Nominal pretreatment conditions are shown in the table below.

Table 1. NOMINAL CONDITIONS FOR PRETREATMENT TESTS

Run No.	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Avg Bed Temp, °F
FP-122	0.39	1200	21.0	650
FP-123	0.37	1200	21.0	600

The pretreated coal from Run FP-122 was successfully fed and gasified in the pilot plant hydrogasification unit in Runs HT-184 and HT-185.

Coal Characterization

Calorimetry

The heat capacities of coal (Ireland mine), chars, and lignite have been determined at 1500° and 1300°F, and are being determined at 1000°F.

Methanation

Sulfur analyses for the runs made with Harshaw cobalt-molybdate catalyst indicate that there is not much conversion of COS to H_2S at space velocities of 500-1000 hr^{-1} and temperatures of 550°-850°F. We plan to test Girdler G-41 chromia-alumina catalyst this month.

Engineering Economics

Work on the cost estimates associated with the use of an electrothermal fluidized hydrogen source is nearing completion for a 250 million CF/day plant.

Work Planned

A detailed work plan is now being prepared to cover the work associated with Amendment No. 1 of the contract.

1. The specifications for a nominal 2-Mw power source for use in the pilot plant are now being prepared.

2. A 100-kw, high-pressure electrothermal reactor is now being designed. Initial studies indicate that a metallic reactor wall with a central electrode may be the preferred configuration for this reactor size. We have held discussions with people from Iowa State.

3. An area at our Crawford Avenue pilot plant is being cleared for installation of the electrothermal reactor.

4. Work is underway on an investigation of a slurry feed system for the pilot plant. The use of such a system is being considered because of the problems associated with a lock hopper system. Both water and benzene are being considered as slurry media.

5. Two additional tests on Colorado subbituminous coal are being planned for the pilot unit hydrogasifier. After these runs, this unit will be used to start investigation of the producer gas generator to be associated with the MHD generator power source. Also further evaluation of the direct use in the hydrogasifier of a CO-H_2 mixture as generated in the electrothermal reactor will be made.

   Prior to this work, however, we plan to make some fluidized-char-bed resistance measurements necessary for the design of the electrothermal reactor.

6. An alternate design of the hydrogasifier for the pilot plant is now being evaluated. Bechtel's initial design has lead to a hydrogasifier configuration that involves internally insulated piping in the second-stage hydrogasifier, which we believe will lead to high costs. An alternate design is being considered in which the first-stage hydrogasifier with its associated solids transfer lines is enclosed in a pressure shell, thereby allowing use of external insulation. We believe this is a considerably less expensive system.

7. An economic evaluation of a 400 million CF/day plant using a lignite feed will be developed. This estimate will be made assuming an MHD generator supplying power to an electrothermal hydrogen producer.

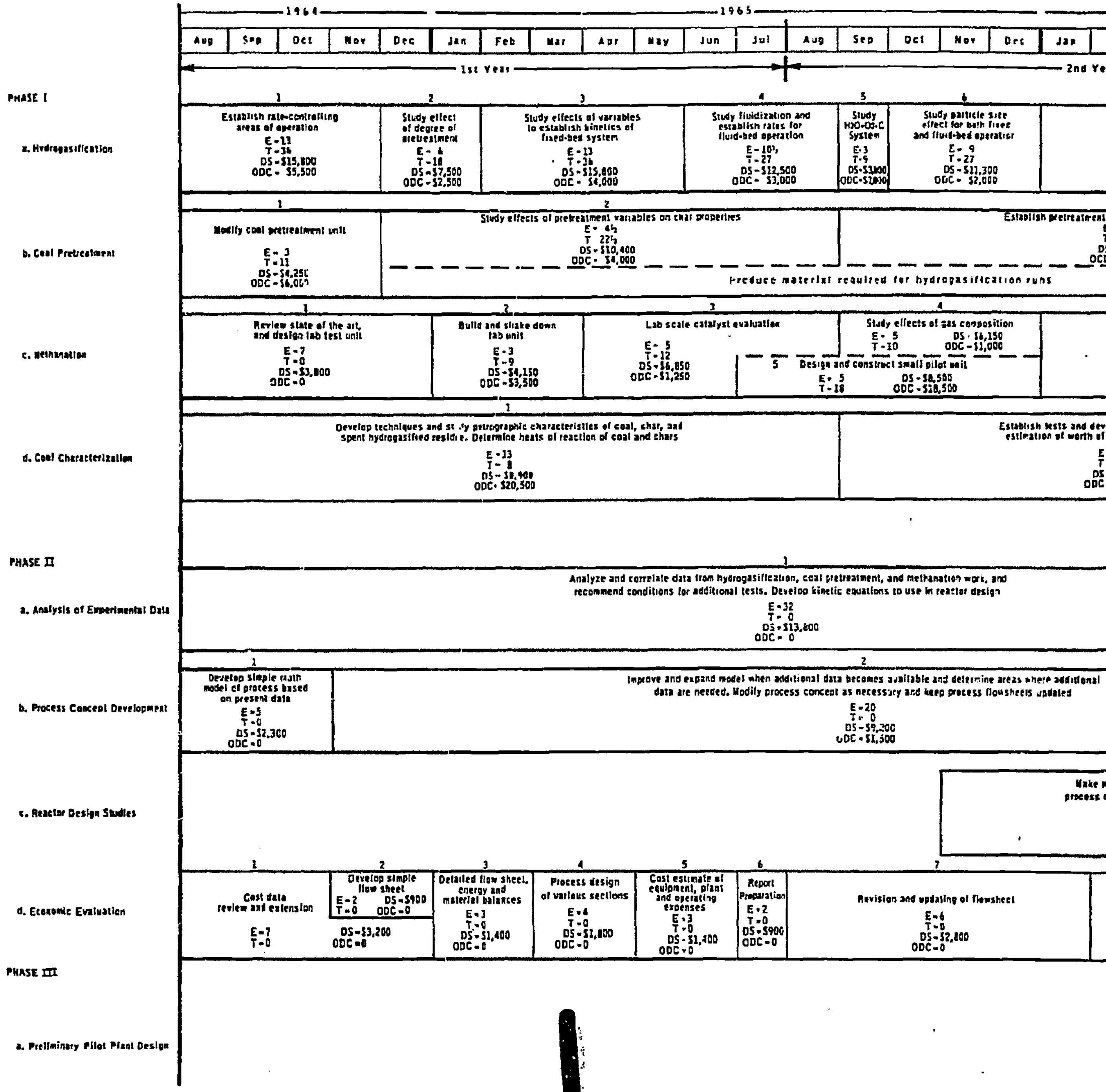
During the month no inventions were made during the course of the work.

Approved Jack Huebler
Jack Huebler, Associate Director

Signed Frank Schora
Frank C. Schora, Assistant Director

.PROGRAM FOR)EVELOPMENT OF IG

.OCR: Contract No. 14-C. 0001-381



TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)

	TOTAL																		
Planned	855,400	23,500	23,500	23,300	23,800	22,400	24,100	24,300	24,700	23,700	23,300	23,800	30,300	29,300	27,800	27,400	28,100	28,100	23,300
Actual		10,000	15,000	23,300	14,500	23,000	21,100	19,800	28,900	23,400	30,200	24,500	22,900	25,500	21,900	26,900	27,800	22,300	23,400
Cumulative		10,000	25,000	48,300	62,800	85,800	106,900	126,700	155,600	179,000	209,200	233,700	256,600	282,100	304,000	330,900	358,700	381,000	404,400

Manpower shown in total man months: E - Engineers
T - Technicians

Costs shown are costs to OCR or one-half o

IGT HYDROGASIFICATION PROCESS

001-381 A.G.A. PB-23a

1966 — 1967

2nd Year — 3rd Year

Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul

7

Study hydrogasification of various coals, lignite, under most suitable conditions
E-214
T-63
DS-$27,200
ODC- $7,000

8

Perform any support work necessary for phase III
Double check data on which pilot plant is being designed

3

ish pretreatment properties of various coals
E- 54
T-274
DS-$12,600
OCD- $4,500

uns

4

Produce material needed for any hydrogasification runs

6

Run small pilot unit and develop kinetic equations for design
E-10
T-19
DS-$11,500
ODC-$5,250

7

Perform any support work necessary for phase III

2

sh tests and develop correlations to enable
ation of worth of coal for hydrogasification
E-14
T-11
DS-$10,200
ODC-$500

Laboratory and Pilot Plant
Phase I-a8, b4, c7 II-a2
Engineers 24
Technicians 105
Salaries $40,400
Other Direct Costs 11,400

2

Perform analysis of past data and new data in any areas necessary for phase III work

here additional
updated

1

Make preliminary reactor design based on process concept and hydrogasification results
E-6
T-0
DS-$2,800
ODC-0

2

Work with subcontractor on design of reactors for hydrogasification and methanation
E-4
T-0
DS-$1,800
ODC-0

8

Revision and refinement of capital and operating costs
E-6
T-0
DS-$2,800
ODC-0

9

Work with subcontractor on capital and operating costs for pilot plant
E-9
T-0
DS-$4,100
ODC-0

1

Develop pilot plant flowsheet with heat and material balances
E-14
T- 0
DS-$7,700
ODC- 0

Select architect-engineer subcontractor

2

With assistance of subcontractor work on phase III as outlined in Contract No. 14-01-0001-381.*
E-34
T- 0
DS-$16,300
ODC- 0

2

* $85,500 available for architect-engineer subcontractor and engineering consultants.

Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
23,300	23,300	23,300	23,300	23,300	23,300	23,300	15,800	15,800	15,800	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700
25,400	22,300	25,000	32,600	25,000	26,600	27,200	22,900	23,700	19,200	21,800	21,400	22,900	39,200	40,800	36,800	37,500	15,500	
404,400	426,700	451,700	484,300	509,300	535,900	563,100	586,000	609,700	628,900	650,700	672,100	695,000	734,200	775,000	811,800	849,300	*864,800	

R or one-half of total costs, DS = Direct Salaries, $
ODC = Other Direct Costs, $

*PLANNED 855,400 INCREASED TO 883,700.

IGT-MPR--7/67

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report – July 1967

to

Office of Coal Research
Contract No. 14-01-0001-381

Summary

1. Two runs during the month with Colorado subbituminous coal completed the tests with this coal.
2. Three pretreater runs were made to supply material to the hydrogasifier.
3. Work on finding a suitable hydrodesulfurization catalyst continues.
4. We have completed the design of the 500-kw high-pressure electrothermal reactor.
5. A design study for an alternate hydrogasifier configuration was initiated.

Hydrogasification

During the month of July, we made two hydrogasification runs with Colorado subbituminous coal. Run HT-186 was set up to use a hydrogen-to-coal ratio of 20% of stoichiometric reacting with a lightly pretreated coal. Blockage of the coal feed tube with lightly agglomerated char caused termination of the run at the end of the first hour. Because laboratory tests showed the pretreated coal to be agglomerating, it was passed through the pretreater again and a duplicate of Run HT-186 was attempted. This run, HT-187, was successful and lasted for a period of 6 hours.

The next run planned will use a mixture of synthesis gas reacting with lignite to establish a hydrogasifier effluent composition for use in the cost estimate to be made for the hydrogasifier electrothermal system with lignite feed.

Pretreatment

In association with the above two hydrogasifier runs, two pretreatment runs were made with Colorado subbituminous coal. We conducted a pretreatment run with Ireland mine coal to produce char for forthcoming runs with synthesis gas.

Methanation

We have tested Girdler G-41 desulfurization catalyst at temperatures of 550°-850°F and at a low space velocity. At the lower temperatures there appears to be more conversion of COS with this catalyst than with cobalt-molybdate. The higher temperature data have not yet been fully analyzed.

Engineering Economics

The final draft of the economic analysis for the hydrogasifier electrothermal configuration is in preparation. We are now preparing a process description for a study based on lignite using the new configuration — i.e., hydrogasifier, electrothermal reactor, MHD onsite power system.

Electrothermal Reactor

The design of a 6-in.-ID high-pressure reactor was essentially completed. This reactor will be based on the use of a nonconsumable electrode and will have a 500-kw power source. We have ordered a tantalum electrode for testing at Iowa State.

Power Source

We have prepared specifications for an MHD power source to be used onsite in the pilot plant with the electrothermal gasifier.

Pilot Plant Preliminary Design

Plans are under way to investigate an alternate design for the pilot plant hydrogasifier. The initial design was based on the use of lock hoppers and individually internally insulated first-stage reactor and associated transfer lines. The new concept to be studied makes use of a slurry feed system and relocation of the first-stage reactor and associated transfer lines with external insulation into a pressure vessel located above the second-stage hydrogasifier.

During the month no inventions were made in the course of the work.

Signed

Frank Schora, Assistant Director

IGT- QTPR--7-9/67

INSTITUTE OF GAS TECHNOLOGY - IIT CENTER - CHICAGO 60616

Project Status Report
For
OFFICE OF COAL RESEARCH
and
AMERICAN GAS ASSOCIATION

Report For Third Quarter, 1967

Project Title Pipeline Gas From Coal – Hydrogenation (IGT Hydrogasification Process)

OCR Contract No. 14-01-0001-381(1)   A.G.A. Project No. IU-4-1

I. Project Objective

The overall objective of this project is a process for production of pipeline gas from coal that is economically attractive for supplementing natural gas supplies. The present objective is the design, construction, and operation of a large integrated pilot plant to obtain scale-up data and operating experience. Developmental research, engineering studies, and economic evaluation are in progress to help attain this objective.

II. Achievements

COAL CHARACTERIZATION

The surface area of coal increased from 150 sq m/g in the pretreated coal to 500 sq m/g in the hydrogasified char. The high reactivity of the char is traceable to the large surface area.

Maceral analyses and reflectance measurements were completed for the seven coals studied in the pilot plant, including five high-volatile-content bituminous coals.

Investigations of coal-water and coal-benzene slurries showed that benzene (or light oil) should be an excellent choice for the slurry medium. A slurry system for introducing coal into our high-pressure system should cost less to construct and operate better than a lock hopper system. Such a feed system will be included in the large pilot plant being designed.

The heats of reaction of bituminous coal and hydrogen have been determined at 1000 psia between 1300° and 1500°F. The heat release during pretreatment was measured as a function of the degree of pretreatment. The heat capacities of bituminous coals at various stages of gasification have been measured between 600° and 1500°F.

ENGINEERING ECONOMICS STUDIES

By feeding hot synthesis gas from the electrothermal gasification of char to the hydrogasifier instead of hydrogen, a 20-year average gas price of 50¢-52¢/million Btu is projected. This assumes electric power available at 3-3.5 mills/kwhr and includes by-product credit for char, sulfur, and coal fines. A report on this study is being prepared.

The economics of all the current gas-from-coal processes were calculated according to A.G.A. accounting procedures using IGT's computer program, and issued as a separate report. A common basis for costs is suggested to simplify comparison.

HIGH-PRESSURE METHANATION

The life test using Harshaw Ni-0104T catalyst was successfully completed after 1420 hours. The catalyst performed excellently, showing no deactivation. Full CO conversion was achieved at a space velocity of 9000 hr^{-1}. The space velocity was limited by the existing equipment and gas supply, not by the catalyst. This catalyst will be used for methanation in the large pilot plant.

The results of tests with Harshaw Co-Mo-0601T and Girdler G-41 chromia-alumina catalysts indicate no conversion of COS to H_2S. These were considered for use as part of the sulfur removal train for the large pilot plant. Other catalysts are being tested.

PILOT PLANT STUDIES

The coal pretreatment program was completed in April, and no further pretreatment tests are planned. However, additional pretreatment runs will be made to furnish feed for the hydrogasifier.

The hydrogasification program for evaluating various coals was completed in July. We are currently making a detailed study of the hydrogasification behavior when synthesis gas is used in place of hydrogen. Four tests were made with lignite this month.

Equipment is being ordered for the construction of an electrothermal gasifier system with a maximum power input of about 300 kw.

DESIGN OF LARGE PILOT PLANT

The preliminary design by Bechtel is complete. Inspection revealed several areas in the design where modifications and improvements could result in substantial cost reductions. These are being examined. A specification book for bids is being prepared.

PROJECT STATUS CHANGE

An amendment of this contract was signed by the Secretary of the Department of Interior on June 12, 1967, for the design, construction, and operation of a large pilot plant. This amendment extends the contract by 5 years at a budget of $12.27 million. A.G.A. will continue to participate in the program at a funding rate of $300,000 per year, on a year-to-year basis. The 3-year program started in August 1964 terminated, and the amended contract became effective on or about June 12, 1967.

III. Problems

No major problems were encountered during this period.

IV. Recommendations

We recommend that the project proceed into the areas defined in the contract amendment.

V. Status of Funding

1. A.G.A. Funding

A.	1967 Funds Allocated	$324,626
B.	Funds Expended This Month (estimated)	$ 16,737
C.	Funds Expended to Date (estimated)	$274,400

2. OCR Funding

A.	Funds Expended This Month (estimated)	$ 35,714
B.	Funds Expended Since Contract Amendment No. 1 (estimated)	$133,487

As a result of personally reviewing the pertinent data and information reasonably available, it is our opinion that the project's objectives will be attained within the contract term and the funds allocated.

Approved ______________________ Signed ______________________
Frank Schora, Associate Director Bernard S. Lee, Manager

Jack Huebler
Jack Huebler, Research Director

Appendix. Achievements in September

COAL CHARACTERIZATION

Coal Slurry Properties

Investigation of coal properties that may affect coal feeding as a slurry was continued. We expect to use a process in which the pretreated coal is slurried with light oil (probably benzene) produced in the hydrogasification process: The slurry is pumped into a fluid bed at 600°F where the benzene is vaporized by the hot hydrogasifier product gas, the gas mixture is cooled with quench water, and the condensed benzene is separated and recycled. The separation of benzene and water in the presence of coal fines was investigated. Tentative conclusions are as follows:

1. The coal tends to be wetted preferentially by the benzene whether it is wetted first with water or with benzene.
2. The water and benzene rapidly separate (within 2 minutes) into a benzene layer and a water layer.
3. The benzene layer contains suspended coal that settles to the bottom of the layer.
4. The slurry has a tendency, especially on standing, to form a benzene-coal sludge that has about the same density as the water. This sludge may float on top or sink to the bottom of the water layer. After the coal is filtered out, the water and benzene quickly separate.

The behavior of tar produced during hydrogasification in the slurry feed process was also investigated. We expect that when the product gas is cooled to 600°F, part of the tar will condense on the feed coal. The characteristics of a typical tar obtained in Run HT-185 with Colorado subbituminous coal are summarized as follows:

Specific Gravity, 60/60	1.031
Distillation	
IBP, 80°C	
IBP - 160°C	21.5 wt %
160°-200°C	9.6 wt %
200°-300°C	28.8 wt %
300°-355°C	17.5 wt %
Pitch	20.4 wt %
Recovery	97.8 wt %

A flash vaporization curve was estimated for this tar, assuming that two parts (by weight) of benzene or light oil are required to slurry one part of pretreated coal. About 22 weight percent of the tar, equivalent to a cut point of 160°C, remained in the vapor phase at 600°F. Therefore, the recycle slurry liquid will consist mostly of benzene and alkyl benzenes. Dicyclic and higher boiling aromatics, which have greater solubility for coal than benzene and its homologs, will not be present.

In the pilot plant under design, where first-stage hydrogasification of the coal will be cocurrent rather than countercurrent as in the present pilot plant, the liquid products are expected to contain much less of the higher boiling constituents.

Petrography

Work on the petrographic examination of coals was continued. Analyses of the Sewell and Pocahontas No. 4 coals are reported in Table 1.

Calorimetry

The heat capacities of raw coal (Ireland mine), pretreated coal, high-temperature residue, low-temperature residue, and North Dakota lignite were determined at 1500°, 1300°, 1000°, 800°, and 600°F. The results are being analyzed. The heats of reaction of hydrogen and Colorado subbituminous coal, West Virginia coals, Illinois coal, Broken Arrow coal, and Indiana coal are being determined at 1300°F.

Table 1. PETROGRAPHIC ANALYSES OF SEWELL AND POCAHONTAS NO. 4 COALS

	Sewell Seam	Pocahontas No. 4
Maceral Analysis, vol %		
Vitrinite	82	89
Exinite	2	Trace
Fusinite and Semifusinite	5	5
Granular Micrinite	4	2
Other Inertinite	7	4
Total	100	100
Vitrinite Reflectance, %		
1.20 to 1.29	17	--
1.30 to 1.39	28	--
1.40 to 1.49	11	--
1.50 to 1.59	32	--
1.60 to 1.69	4	10
1.70 to 1.79	6	55
1.80 to 1.89	1	24
1.90 to 1.99	1	8
2.00 to 2.19	--	3
No. of Readings	102	149
Avg Reflectance, %	1.45	1.78

Table 2. FEATURES OF HYDROGASIFICATION TESTS

Feed Solids: Dried North Dakota Lignite, Glenharold Mine

Feed Gas: Steam-Hydrogen-Carbon Monoxide-Carbon Dioxide

Run No.	Purpose of Run	Results
HT-188	To establish the level of carbon dioxide produced when hydrogasifying lignite, at 1700°F, with a feed gas containing 50 mole percent steam, and the remainder a mixture of CO, CO_2, and H_2, in a fluidized bed	Partially successful. Feed screw jammed after brief period of steady-state operation.
HT-189	Same as HT-188	Feed screw jammed
HT-190	Same as HT-188	Feed screw jammed
HT-191	Same as HT-188	Feed gas preheat coil plugged

Before the lignite was hydrogasified, it was dried with air at 225°F in the pilot plant coal pretreatment unit in a fluidized bed. The moisture content was reduced from the as-received level of 33% to 5%. The synthesis gas was prepared by compressing the gas components in the desired proportions and storing the prepared mixture at pressure. The prepared gas had the following composition: hydrogen 50%, carbon monoxide 45%, and carbon dioxide 5%. This composition approximates the composition of a gas from an electrothermal char gasifier.

Run HT-188 lasted 2-1/2 hr with a steady-state period of 10 minutes. The test was stopped when the lignite feed screw jammed. The jamming was mechanical in nature and was caused by binding of the screw in the housing between the feed hopper and the reactor. Since binding of this nature was not experienced when feeding raw or pretreated bituminous and subbituminous coals, the shape of the lignite particles may have contributed to the screw's binding.

Test conditions for Run HT-189 were similar to those of Run HT-188. To reduce the chances of feed screw binding, we operated with the temperature at the top of the reactor at 1100°F, instead of 1200°F as in Run HT-188. We also increased the initial feed screw speed from 11 to 18 rpm. However, after only 7 minutes of lignite feeding, the feed screw jammed.

Before we started Run HT-190, we installed water cooling coils around the outside of the feed screw conveyor housing to keep the lignite from being heated, and possibly devolatilizing in the housing. To promote the flow of lignite through the housing, we polished the surfaces of the feed screw flights. In this test we were able to feed the lignite for 1 hr before the feed screw began to bind and forced termination of the test. After this test, we examined the feed screw and the interior surfaces of the screw housing. The root shaft of the screw was slightly bent so that the screw flights scraped the walls of the housing. For most of its length, the bottom of the housing was gouged to depths of 1/64-1/32 in.

For Run HT-191, we smoothed and polished the interior surfaces of the screw housing and installed a new feed screw with a tapered root shaft so that the volume between the flights gradually increases from the feed end to the discharge end of the screw. Before the run, we tested the operation of the screw with lignite at atmospheric pressure. A temporary binding of the screw occurred soon after the screw was filled with lignite, but the binding was cleared by increasing the rotational speed of the screw to 25 rpm. After this, the screw operated well at a speed of 11 rpm. The results of this feeding test indicate that the screw's binding during lignite feeding is primarily a mechanical problem. During the test, however, after steady-state operation was attained, the furnace coils for preheating the feed gas plugged, apparently because of carbon deposition from the carbon oxides in the gas.

The hydrogasification results of Run HT-188 will be presented when the analyses of this test are completed.

The operating conditions and results of Run HT-183 (June 1967 Report), Runs HT-184 and HT-185 (July 1967 Report), and Run HT-187 (August 1967 Report), are presented in Table 3. Chemical and screen analyses of the feeds and residues are given in Table 4. Liquid yields and compositions are shown in Table 5.

The feed for Run HT-183 was an untreated Colorado subbituminous coal. Because this untreated coal had a slight tendency to agglomerate, its performance in the hydrogasification reactor was only marginal, even with free-fall operation as in Run HT-183. Consequently, for the next three tests (Runs HT-184, HT-185, and HT-186), the coal was pretreated with air before hydrogasification.

The carbon gasification (34 weight percent) obtained in Run HT-183 is high for a free-fall operation, even though the hydrogen/coal ratio was 33% of the stoichiometric ratio. This may be explained by the somewhat higher reactivity of the raw coal, compared to the pretreated coal. There was no measurable feed steam decomposition.

With pretreated Colorado subbituminous coal, the highest carbon gasification and the highest hydrocarbon yield were obtained in Run HT-185 when it was hydrogasified with a hydrogen/coal ratio of 24% of the stoichiometric ratio, and a steam concentration of 31 mole percent in the feed gas. The carbon oxides yield in Run HT-185 was lower than those obtained in Runs HT-184 and HT-187 where the steam concentration in the feed gas was 51 mole percent. This lower carbon oxides yield is explained by Run HT-185's lower feed steam decomposition compared to Runs HT-184 and HT-187.

Table 5 illustrates the difference in the oil yields obtained with untreated and pretreated Colorado subbituminous coal. With the raw coal (Run HT-183), over 6% of the carbon in the coal was converted to oil, compared to 2.0-3.9% of the pretreated coal (Runs HT-184, HT-185, and HT-187).

Table 3. OPERATING CONDITIONS AND RESULTS OF HYDROGASIFICATION TESTS WITH UNTREATED AND PRETREATED SUBBITUMINOUS COALS IN HIGH-TEMPERATURE ADIABATIC REACTOR

Coal Source	Colorado Subbituminous Coal			
	Erie District No. 15 Eagle Mine	Pretreatment Run FP-122	Pretreatment Run FP-12[illegible]	Pretreatment Run FP-12[illegible]
Sieve Size, USC	10/80			
Run No.	HT-183	HT-184	HT-18[illegible]	HT-1[illegible]
Duration of Test, hr	3	5-1/2	[illegible]	[illegible]
Steady-State Operating Period, min^a	113-152	210-323	130-[illegible]	[illegible]
OPERATING CONDITIONS				
Bed Height, ft	Free-Fall^p	3.1	[illegible]	[illegible]
Reactor Pressure, psig	1035	101[illegible]	101[illegible]	[illegible]
Reactor Temperature, °F^b				
31-1/2 in.	--	--	--	--
36-3/4 in.	--	--	--	--
42 in.	[illegible]	1155	--	--
47 in.	1225	1375	--	--
52 in.	1310	[illegible]	--	--
57-1/4 in.	--	[illegible]	--	--
62-1/2 in.	1295	1500	[illegible]	[illegible]
67-3/4 in.	[illegible]	[illegible]	[illegible]	[illegible]
73 in.	[illegible]	[illegible]	[illegible]	[illegible]
78-1/4 in.	[illegible]	[illegible]	[illegible]	[illegible]
83-1/2 in.	[illegible]	--	[illegible]	[illegible]
89 in.	[illegible]	--	[illegible]	[illegible]
94-1/2 in.	[illegible]	--	[illegible]	1640
100 in.	[illegible]	--	[illegible]	[illegible]
104 in.	[illegible]	--	[illegible]	[illegible]
114 in.	[illegible]	--	--	--
124-1/2 in.	[illegible]	--	--	--
135 in.	[illegible]	--	--	--
145 in.	--	--	--	--
155-1/2 in.	[illegible]	--	--	--
166 in.	[illegible]	--	--	--
176 in.	1425	--	--	--
187-1/2 in.	[illegible]	--	--	--
197 in.	1290	--	--	--
207 in.	1225	--	--	--
217-1/2 in.	1210	--	--	--
Average	[illegible]	[illegible]	[illegible]	[illegible]
Coal Rate, lb/hr^c	[illegible]	[illegible]	[illegible]	[illegible]
Feed Gas Rate, SCF/hr^d	[illegible]	510.[illegible]	[illegible]	[illegible]
Steam Rate, lb/hr	25.12	25.[illegible]	15.50	[illegible]
Steam, mole % of hydrogen-steam mixture	[illegible]	[illegible]	31.2	[illegible]
Hydrogen/Coal Ratio, % of stoichiometric^e	[illegible]	21.[illegible]	[illegible]	[illegible]
Hydrogen/Steam Ratio, mole/mole	[illegible]	[illegible]	[illegible]	[illegible]
Bed Pressure Differential, in. WC	--	[illegible]	[illegible]	[illegible]
Coal Space Velocity, lb/cu ft-hr	[illegible]	[illegible]	[illegible]	[illegible]
Feed Gas Residence Time, min^f	[illegible]	[illegible]	[illegible]	[illegible]
Superficial Feed Gas Velocity, ft/sec^g	[illegible]	[illegible]	[illegible]	[illegible]
OPERATING RESULTS				
Product Gas Rate, SCF/hr	[illegible]	[illegible]	[illegible]	[illegible]
Net Btu Recovery, 1000 Btu/lb	[illegible]	[illegible]	[illegible]	[illegible]
Product Gas Yield, SCF/lb	[illegible]	[illegible]	[illegible]	[illegible]
Hydrocarbon Yield, SCF/lb	[illegible]	[illegible]	[illegible]	[illegible]
Carbon Oxides Yield, SCF/lb	[illegible]	[illegible]	[illegible]	[illegible]
Net Reacted Hydrogen, SCF/lb	[illegible]	[illegible]	[illegible]	[illegible]
Residue, lb/lb coal^h	[illegible]	[illegible]	[illegible]	[illegible]
Liquid Products, lb/lb coal^i	[illegible]	[illegible]	[illegible]	[illegible]
Net MAF Coal Hydrogasified, wt %^{j}	[illegible]	[illegible]	[illegible]	[illegible]
Carbon Gasified, wt %	[illegible]	[illegible]	[illegible]	[illegible]
Steam Decomposed, lb/hr^k	[illegible]	[illegible]	[illegible]	[illegible]
Steam Decomposed, % of steam fed	[illegible]	[illegible]	[illegible]	[illegible]
Steam Decomposed, % of total equivalent fed^m	[illegible]	[illegible]	[illegible]	[illegible]
Overall Material Balance, %	[illegible]	[illegible]	[illegible]	[illegible]
Carbon Balance, %	100.[illegible]	101.[illegible]	[illegible]	[illegible]
Hydrogen Balance, %	[illegible]	[illegible]	[illegible]	[illegible]
Oxygen Balance, %	[illegible]	[illegible]	[illegible]	[illegible]
PRODUCT GAS PROPERTIES				
Gas Composition, mole %				
Nitrogen	[illegible]	[illegible]	[illegible]	[illegible]
Carbon Monoxide	[illegible]	[illegible]	[illegible]	[illegible]
Carbon Dioxide	[illegible]	[illegible]	[illegible]	[illegible]
Hydrogen	41.0	[illegible]	[illegible]	[illegible]
Methane	15.2	[illegible]	[illegible]	[illegible]
Ethane	[illegible]	[illegible]	1.1	[illegible]
Propane	[illegible]	[illegible]	[illegible]	[illegible]
Butane	--	--	--	--
Benzene	[illegible]	0.3	[illegible]	[illegible]
Hydrogen Sulfide	--	--	--	--
Total	[illegible]	[illegible]	[illegible]	[illegible]
Heating Value, Btu/SCF^n	[illegible]	[illegible]	[illegible]	[illegible]
Specific Gravity (Air = 1.00)	[illegible]	[illegible]	[illegible]	[illegible]
Nitrogen Purge Rate, SCF/hr	[illegible]	[illegible]	[illegible]	[illegible]

Table 3, Cont. OPERATING CONDITIONS AND RESULTS OF HYDROGASIFICATION TESTS WITH UNTREATED AND PRETREATED SUBBITUMINOUS COALS IN HIGH-TEMPERATURE ADIABATIC REACTOR

a. From start of coal feed.

b. Tube wall temperatures. Bottom of coal bed at 31 in. in Run HT-184; 62 in. in Runs HT-185 and HT-187.

c. Operating conditions and results based on weight of dry feed.

d. Hydrogen.

e. Percent of the stoichiometric hydrogen/char ratio – the net feed hydrogen/char ratio required to convert all the carbon to methane.

f. Coal bed volume/(CF/min feed gas at reactor pressure and temperature).

g. (CF/sec feed gas at reactor pressure and temperature)/cross-sectional area of reactor.

h. By ash balance.

i. Includes condensed, undecomposed steam.

j. 100 (wt of product gas-wt hydrogen in-wt decomposed steam-wt nitrogen in/wt of moisture-, ash-free coal).

k. Computed as difference between steam feed rate and the measured liquid water rate leaving the reactor.

m. Computed as difference between the total equivalent steam feed rate (includes moisture content of feed char and bound water corresponding to oxygen content of feed char) and the measured liquid water rate leaving the reactor.

n. Gross, gas saturated at 60°F, 30-in.-Hg pressure. SCF: dry gas volume in SCF at 60°F, 30-in.-Hg pressure.

p. Free-fall length of 15.5 ft.

q. Based on 1.3702 cu ft free-fall volume.

Table 4. CHEMICAL AND SCREEN ANALYSES OF HYDROGASIFICATION FEEDS AND RESIDUES

Run No.	HT-183		HT-184		HT-1[illegible]		HT-187	
Sample	Feed	Residue	Feed	Residue	Feed	Residue	Feed	Residue
Proximate Analysis, wt %								
Moisture	4.1	0.5	0.3	0.7	0.3	0.[illegible]	0.[illegible]	0.6
Volatile Matter	34.5	3.4	31.1	2.5	31.1	[illegible]	25.2	3.3
Fixed Carbon	56.7	86.7	63.3	86.6	63.3	84.[illegible]	[illegible]	84.5
Ash	4.7	9.4	5.3	10.2	5.3	11.2	[illegible].1	11.6
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Ultimate Analysis (Dry), wt %								
Carbon	74.1	86.3	73.1	[illegible]	73.1	[illegible]	[illegible]	[illegible]
Hydrogen	5.44	1.30	[illegible]	1.42	[illegible]	1.[illegible]	[illegible]	1.12
Nitrogen	1.27	0.00	1.40	0.57	1.40	[illegible]	1.[illegible]	[illegible]
Oxygen	13.94	2.86	15.[illegible]2	1.44	15.[illegible]2	1.[illegible]	17.1[illegible]	2.0[illegible]
Sulfur	0.54	0.07	0.27	0.21	0.2	0.1[illegible]	[illegible]	[illegible]
Ash	4.91	9.47	[illegible].34	10.24	[illegible].34	11.2	[illegible].12	11.[illegible]
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.0	100.00
Screen Analysis, USS, wt %								
+20	2.9	9.4	3.1	1.0	3.1	2.2	3.[illegible]	[illegible]
+30	19.3	15.2	14.8	[illegible]	14.[illegible]	[illegible]	[illegible]	[illegible]
+40	30.2	30.1	32.9	[illegible]	35.[illegible]	[illegible]	22.[illegible]	[illegible]
+60	26.1	25.3	29.0	[illegible]	[illegible]	[illegible]	23.2	[illegible]
+80	12.3	10.4	12.9	13.[illegible]	12.9	1[illegible]	15.[illegible]	11.0
+100	4.4	3.5	3.5	4.8	3.[illegible]	[illegible]	[illegible]	[illegible]
+200	3.7	4.9	1.7	[illegible]	1.[illegible]	[illegible]	[illegible]	[illegible]
+325	0.5	0.8	0.2	[illegible]	0.2	[illegible]	0.[illegible]	[illegible]
-325	0.6	0.4	0.1	0.4	0.1	[illegible]	0.[illegible]	[illegible]
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Table 5. COMPOSITION OF HYDROGASIFICATION LIQUID PRODUCTS

Run No.	HT-183	HT-184	HT-185	HT-187
Sample	Condenser	Condenser	Condenser	Condenser
Liquid Products,* lb/lb coal	0.703	0.337	0.206	0.271
Composition of Liquid Products, wt %				
Water	91.9	93.0	83.0	93.6
Oil	8.1	7.0	17.0	6.4
Total	100.0	100.0	100.0	100.0
Composition of Oil Fraction, wt %				
Carbon	82.8	81.9	81.6	83.7
Hydrogen	5.41	6.63	6.69	6.42
Total	88.21	88.53	88.29	90.12
Carbon in Oil Fraction				
lb/lb coal	0.0470	0.01937	0.02857	0.01455
wt % of carbon in coal	6.34	2.65	3.91	1.98

* Includes condensed, undecomposed steam.

Table 6 summarizes the tests made in the third quarter of 1967. With the completion of the coal evaluation program, gasification studies will be designed to simulate various portions of the large pilot plant to provide needed design data. For this reason the bar chart summary used in previous reports during the coal evaluation program has been discontinued.

Table 6. HYDROGASIFICATION TESTS IN THIRD QUARTER 1967

Run No.	Solids Feed*	Gas Feed†	Nominal Temp, °F	Purpose of Run	Results	Remarks
HT-184	CSB	HS	1300-1700	To study the hydrogasification of a lightly pretreated Colorado subbituminous coal with a hydrogen-steam mixture containing 50 mole percent steam and a hydrogen/coal ratio of [illegible] of the stoichiometric ratio in a 3-1/2-ft fluidized bed	Successful	Colorado subbituminous coal requires mild pretreatment
HT-185	CSB	HS	1500-1800	Same as HT-184, except with a 30 mole percent steam concentration	Successful	
HT-186	CSB	HS	1500-1700	Same as HT-184, except with a hydrogen/coal ratio of 20% of the stoichiometric ratio	Coal agglomerated at top of reactor tube	
HT-187	CSB	HS	1500-1[illegible]00	Same as HT-184	Successful	Evaluation of Colorado coal completed. Completed planned coal evaluation program
HT-188	L	HSMP	1700-1[illegible]00	To establish the level of carbon dioxide produced when hydrogasifying lignite with a feed gas containing [illegible] mole percent steam, and the remainder a mixture of CO, CO₂, and H₂ in a fluidized bed	Partially successful: feed screw jammed after short steady-state period	
HT-189	L	HSMP	1700-1[illegible]00	Same as HT-188	Feed screw jammed	
HT-190	L	HSMP	1700-1[illegible]00	Same as HT-188	Feed screw jammed	
HT-191	L	HSMP	[illegible]00-1[illegible]00	Same as HT-188	Feed gas preheater coil plugged	Solved mechanical problem of screw

* CSB — Colorado subbituminous
L — North Dakota lignite

† HS — Hydrogen-steam
HSMP — [illegible]

IGT-MPR--8/67

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report – August 1967
to
Office of Coal Research
Contract No. 14-01-0001-381

Summary

1. Three hydrogasification tests were attempted during the month using lignite feed and a synthesis gas. All three tests were unsuccessful because of mechanical binding in the feed screw.
2. Design, procurement, and construction of the high-pressure electrothermal reactor are now well under way. The latest delivery date – that of the steam generator-superheater – will be December 1967.
3. The hydrogasifier design study, in cooperation with Bechtel, is now well under way.
4. Proposals on an MHD power package are expected from three organizations, and there is a possibility that there will be a fourth respondent.

Hydrogasification

We conducted three hydrogasification tests this month in the balanced-pressure pilot unit (Runs HT-188, HT-189, and HT-190). In these tests an untreated, but dried, North Dakota lignite from the Glenharold mine was hydrogasified with a synthesis gas. The objective of these tests was to obtain experimental data on the concentrations and quantities of carbon dioxide in the product gases. This data is needed to design the proposed 3 ton/hr pilot plant. Only one of the three tests (Run HT-188) was partially successful. In the two other tests, binding of the feed screw in its housing terminated the tests before steady-state operation was established. The probable cause of the binding appeared to be entrapment of lignite particles between the outer edges of the feed screw flights and the feed screw housing. This might have been caused by the deposition of oils and tars in the feed screw conveyor housing by the slow devolatilization of the lignite as its temperature was raised when it entered the reactor.

The lignite, after crushing to a –10+80 mesh size, was air-dried at 225°F in a fluidized bed. This reduced its moisture level of 35% as-received to 5% before hydrogasification. We reacted the lignite in a 3-1/2-ft fluidized bed with a feed gas mixture of steam and synthesis gas. The synthesis gas was premixed and then stored at pressure. It consisted of hydrogen, carbon monoxide and carbon dioxide, in molar concentrations of 50, 45, and 5%. The steam concentration in the feed gas was 50 mole percent, while the hydrogen/lignite ratio was 25% of the stoichiometric ratio. The maximum lignite bed temperature reached in Run HT-188 was 1500°F, although we had planned to run at 1700°F. This run, which lasted 2-1/2 hrs, with 10 minutes of this time at steady state, was ended when the lignite feed screw began to bind. On a nitrogen-free basis, the product gas contained 11.1 mole percent carbon monoxide and 35.2 mole percent carbon dioxide.

Test conditions of Run HT-189 were similar to those of Run HT-188. To reduce the possibility of the feed screw binding, we operated with the top-of-the-reactor temperature at 1100°F, instead of 1200°F as in the previous run. We also increased the initial feed screw rotational speed from 11 to 18 revolutions per minute so that lignite particles would not be so easily trapped between the screw flights and the conveyor housing. However, after only 7 minutes of feeding the feed screw jammed. The cause of the binding appeared to be the same as in Run HT-188.

We made a third attempt at hydrogasifying the lignite with synthesis gas in Run HT-190. Before this run, we installed water cooling coils around the outside of the screw conveyor housing to keep the lignite from devolatilizing there. To promote the flow of the lignite through the housing, the surfaces of the feed screw flights were polished. In this test, we were able to feed the lignite for 1 hour before the feed screw began to bind and the test had to be terminated. The feed screw and the interior surfaces of the screw housing were examined. The root shaft of the screw was found to be bent slightly so that as the screw rotated the flights scraped against the walls of the housing. For most of the length of the screw housing, we found the bottom of the housing interior to be gouged to depths of 1/64 to 1/32 inch. Before we make any more tests with lignite, we will replace the feed screw with a screw having a tapered root shaft, so that the space between the flights will be longer at the discharge end of the screw than at the feed end. Mechanical misalignment of the screw may have been the primary cause of difficulties encountered in the three tests.

High-Pressure Methanation

Runs with Girdler G-41 desulfurization catalyst at temperatures up to 850°F and 1500 hr^{-1} space velocity did not show significant conversion of COS in a typical methanator feed. We are contacting several catalyst manufacturers for other trial catalysts.

Electrothermal Gasification

The vessel design and layout of the pilot unit have been completed and fabrication of the primary vessels is in progress. Construction of the barricade is completed and the servicing decks are now being installed. The steam generator and superheater have been ordered with December 1967 set as the delivery date. Several firms are preparing estimates and bids for the power supply and associated controls. A study to find a suitable nonconsumable electrode material is being conducted with the assistance of Iowa State University.

Preliminary Hydrogasifier Design

A revision of the initial hydrogasifier configuration is now well under way. The design concept is to use a water-jacketed pressure vessel which will contain both hydrogasifier stages and a coal slurry drier. The feed system will be based on a light oil-coal slurry, pressurized through a mud pump, and dried at pressure. Solids will be discharged by first slurrying them with water and then reducing the pressure through a line constriction. By using these two systems, we will avoid the problems associated with lock hoppers.

Power Source

We have received definite word that three organizations will bid on the 2-megawatt power package with a possible fourth bidder. The three are Avco, Atomics International, and Dicks Associates. Dick Associates, we understand, will bid based upon a team which will include a division of Chrysler Corporation. It is possible that M. Jones, Associates, will also present a proposal, although this is not yet definite.

During the month, no new inventions were made during the course of the work.

Approved Jack Huebler — Signed Frank Schora

Jack Huebler, Research Director — Frank Schora, Associate Director

IGT-MPR--9/6

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report – ~~October~~ Sept. 1967
to
Office of Coal Research
Contract No. 14-01-0001-381

Summary

1. During the month, two hydrogasification runs were made employing an electrothermally produced type of gas to gasify lignite. One of these two runs was completely successful.
2. We have encountered difficulty in the use of hydrodesulfurization catalysts to convert COS to H_2S. Such a scheme had been considered as a possible cleanup approach for the hydrogasifier effluent. Further work will be done in this area.
3. Construction of the development-scale electrothermal fluidized-bed reactor is continuing, with the reactor proper nearing completion.
4. The preliminary design of the hydrogasification section of the plant should be completed before the end of October; bids will be requested at that time. It now appears certain that three organizations will present proposals for the fabrication of a 2000-kw MHD system for incorporation into the pilot plant.

Hydrogasification

During September, we conducted two hydrogasification runs in the high-pressure reactor to evaluate the use of synthesis gas as the hydrogasification medium. Prior to these runs, the problems with the coal feed system were corrected. The problem with this feed system in previous runs was caused by the mechanical binding of the screw.

The first of the two runs, HT-191, was terminated after 2 hours because of a carbon blockage in a gas-fired preheater in which the premixed synthesis gas underwent preheating. Under the conditions of operation, carbon deposition by reversion of carbon monoxide to carbon dioxide and carbon can easily occur.

The second of these runs, HT-192, was completely successful. It lasted a total period of 4 hours, 2 hours of which were at steady-state conditions. Both runs were carried out using lignite as the feed material. After completion of these runs, the top of the unit was modified to enable some test work necessary to establish the electrical characteristics of a fluidized bed of char at high pressure. These resistance measurements are necessary to properly specify the electrical conditioning system to be purchased for use with the process development-scale electrothermal reactor now under construction.

Electrothermal Gasification

Initial piping work has started in the area in which the electrothermal gasifier will be located, and specification of control equipment and instrumentation is complete. We have been informed that the reactor is ready for shipment from Houston and should be received in Chicago during the first quarter of October. Work at Iowa State University has indicated that a molybdenum electrode appears to be resistant to attack during the gasification of our char in the electrothermal gasifier. Weight lost from this unit over a several-hour run was not detectable. This appears encouraging. However, a search for possible electrode materials will be continued. We have obtained an electrical engineering consultant — Dr. Ralph Armington, a professor at IIT — to assist with the electrical aspects of the electrothermal gasification study.

Power Package

Proposals for a 2000-kw MHD power package have been received from Avco Corporation and Atomics International. These proposals are now being reviewed. A third proposal is expected from J. B. Dicks and Associates, Inc., in conjunction with Chrysler Corporation. We expect to receive this proposal on October 17, 1967. M. Jones and Associates has declined to present a proposal because of insufficient time. Discussions with Dr. Jones indicate that his organization does not have a clear concept of a channel design at this time. Discussions will be held with respondents in the near future prior to recommending a contractor for this section of the pilot plant.

Methanation

Work is being undertaken to identify a hydrodesulfurization catalyst capable of converting COS to H_2S under the conditions to be encountered in the hydrogasification gas-cleanup area. High-temperature runs with COS-containing gas indicate that conversion to H_2S does not occur at space velocities of 1000 to 2000 hr^{-1} using Girdler G-41 chromia-alumina catalyst. It is possible that carbon monoxide in the gas may be inhibiting the catalytic activity of this material.

Coal Characterization

In the area of coal characterization, we are studying the properties of the slurry produced by slurrying pretreated coal and raw coal with the light oil produced in the hydrogasification process. In our process configuration, we plan to pump this slurry into a high-pressure fluidized bed where the oil will be flashed off at a temperature of 600°F. This oil is then recovered and reused for slurrying. In establishing some of the initial properties of these slurries, we found that coal tends to be wetted preferentially by the light oil and that a water-light oil mixture with minor amounts of coal entrained separates rapidly into two distinct layers.

Calorimetry

The determination of the heats of reaction of coals of various origins and hydrogen is continuing so that we can obtain data on the various coals used in our hydrogasification program.

Preliminary Design

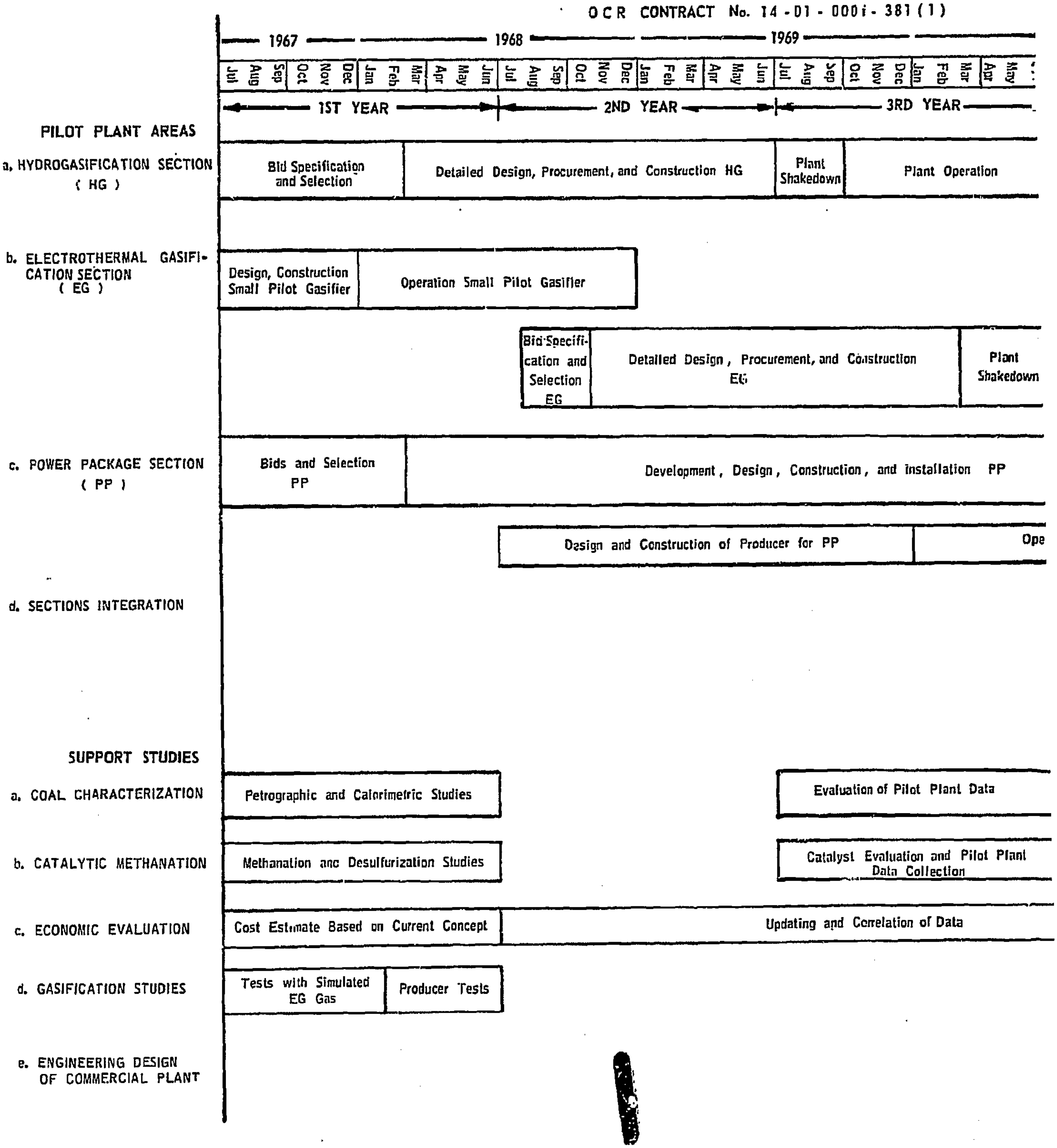
The finalized preliminary designs are now being prepared by Bechtel Corp. based on the new hydrogasifier configuration. Bechtel should have this work completed by mid-October. Based on this design, the preliminary design book is being completed and will be finished by mid- to late October. We will then request proposals for the detailed design, procurement, and construction of that portion of the plant covered by the preliminary design.

During the month, no new inventions were made in the course of the work.

Approved Jack Huebler Signed Frank Schora
Jack Huebler, Research Director Frank Schora, Associate Director

PILOT PLANT PROGRAM OF IGT HYDROG.

OCR CONTRACT No. 14-01-0001-381 (1)



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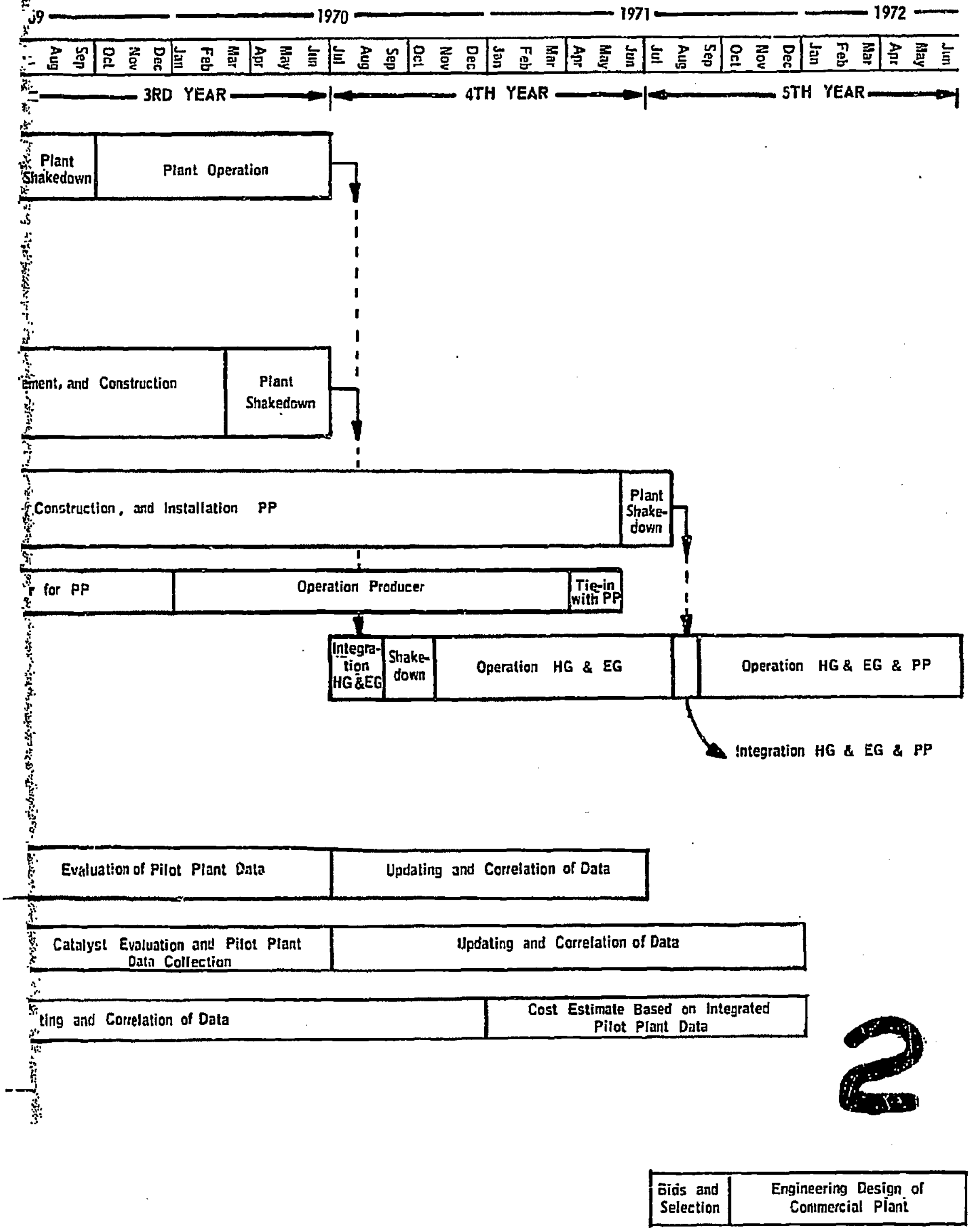

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DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - October 1967
to
Office of Coal Research
Contract No. 14-01-0001-381

Summary

1. Tests are continuing to determine the electrical conductivity characteristics of a fluidized bed of char at high pressures.
2. The electrothermal reactor vessel has been received and is being assembled. The high-pressure steam generation system is on order.

Hydrogasification

The furnace used both to generate steam and to preheat the feed gas is being repaired for leaks in the tubes. The two tube sections will be replaced. To minimize carbon deposition in the tubes when synthesis gas is the feed gas, the outlet of the steam-generating tubes will be joined to the inlet of the feed gas preheat tubes so that the synthesis gas will be preheated in the presence of steam. The furnace insulation support frame is also distorted and will be rebuilt.

We made additional tests in the hydrogasification pilot unit to establish the electrical conductivity characteristics of a fluidized bed of char at high pressure. Twelve tests were made in which resistance, voltage, and current measurements were taken with electrode immersion depths of 2-1/2 and 6 ft. The char was fluidized with nitrogen as the bed temperatures varied from 1300° to 1600°F, depending on the nitrogen flow rate.

Electrothermal Gasification

The reactor vessel has been received from the Gray Tool Co. and is being assembled. Mounting supports for the reactor and auxiliary vessels are being constructed. The high-pressure steam generator and superheater have been ordered. With the assistance of our consultant, Professor Ralph Armington of the IIT Electrical Engineering Department, we are conducting tests in the 4-in. hydrogasification reactor to define the electrical properties of the proposed system. The tests are being run at conditions simulating those of the electrothermal gasifier. The effects of electrode immersion depth, unit pressure, fluidizing gas velocity, and temperature have been studied. The purpose of these tests is to provide information that will enable us to acquire the most economical and versatile power supply for the test program. Several electrical companies are preparing bids using the information that is presently available, and we should have a power source ordered within a month.

Methanation

We have solved the problem of catalyst deactivation in the stirred reactor by placing a bed of nickel-kieselguhr catalyst and zinc oxide in the feed hydrogen-nitrogen stream before adding CO. Apparently a trace impurity in either the H_2 or N_2 poisoned the small amount of catalyst we use. We are ready to check the effect of particle size on the kinetics.

Calorimetry

The heat of reaction of hydrogen and the following coals were determined at 1300° and 1500°F.

1. Illinois coal
2. West Virginia Sewell coal
3. West Virginia Block No. 5 coal
4. Colorado subbituminous coal

The initial results indicate that the heats of reaction of these coals are about 2500 Btu/(lb coal reacted), compared to 1800 Btu/(lb coal reacted) for Ireland mine raw coal. These results are being analyzed further and additional data are being obtained.

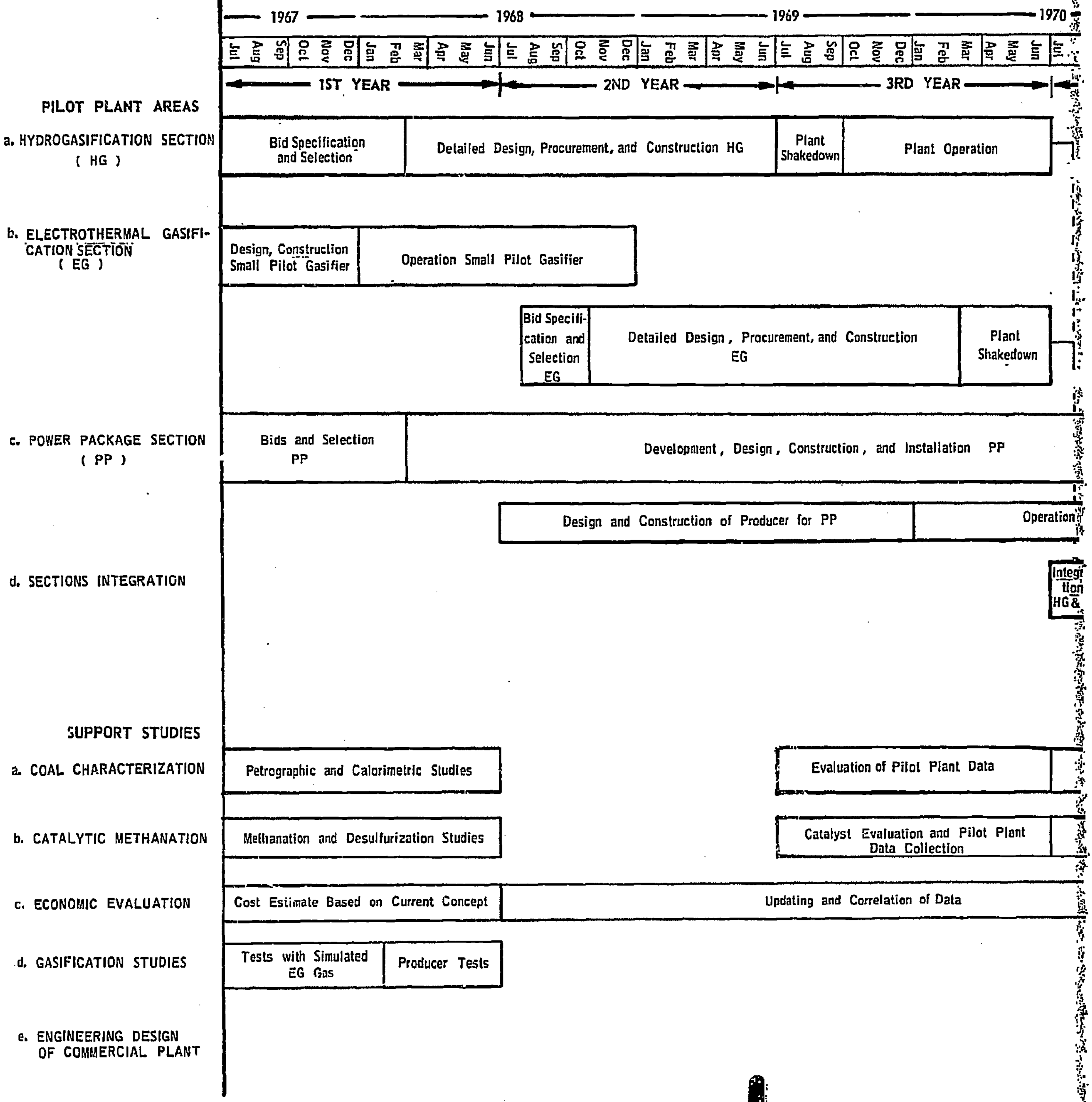
During the month, no inventions were made in the course of this work.

Approved Jack Huebler Signed

Jack Huebler, Research Director Frank Schora, Associate Director

PILOT PLANT PROGRAM OF IGT HYDROGASIF

OCR CONTRACT No. 14-01-0001-381 (1)



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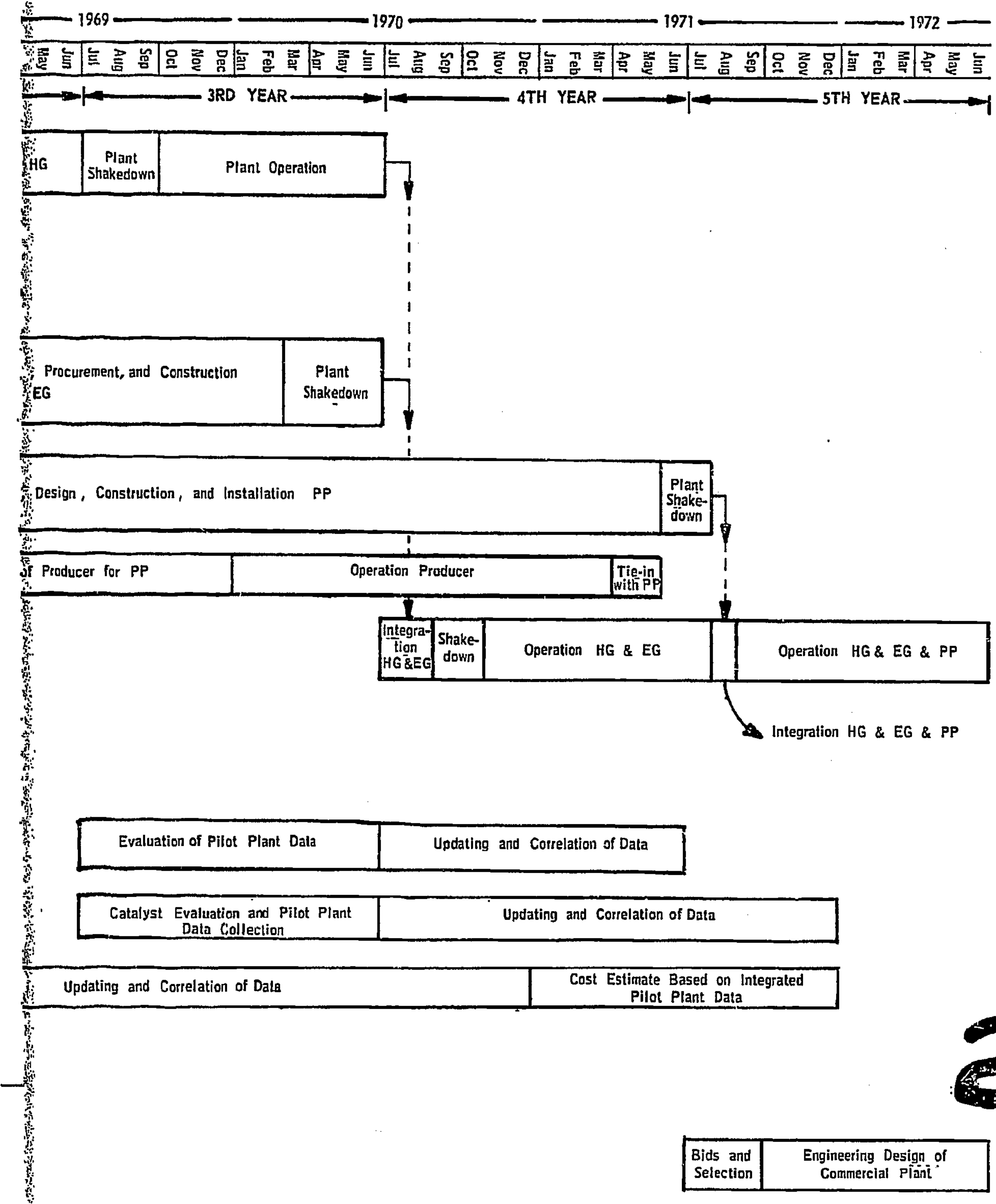

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IGT-MPR--11/6

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report – November 1967
to
Office of Coal Research
Contract No. 14-01-0001-381

Hydrogasification-Electrical Conductivity Tests

We conducted four electrical conductivity tests in the balanced-pressure hydrogasification reactor. These tests are a continuation of a study to establish the electrical characteristics of a fluidized bed of char at high pressure. The char in these tests was a partially hydrogasified Pittsburgh seam bituminous coal from the Ireland mine. It was fluidized with an equal-volume mixture of steam and nitrogen in a 2-1/2-ft bed maintained at temperatures of 1300° to 1700°F. Electrical flow measurements were made across the bed over a range of applied voltages, and over a range of linear gas velocities, so that char bed resistances could be computed. We conducted these tests at atmospheric pressure and at 200, 500, and 1000 psig to establish the effect of pressure.

Before we started the electrical conductivity tests with steam and nitrogen, we repaired the steam-generating and feed gas preheat furnace, and installed two new tube bundles in the reactor. One of the tube bundles is for steam generation, the other for feed gas preheat. The original set of tubes was deformed from excessive temperature exposure, and the steam-generating portion had a hole near a return bend that could not be repaired.

Electrothermal Reactor

All major items have been ordered with the exception of the power supply. This item should be placed on order in December. Supports are now being installed for the reactor vessel.

Engineering Economics Studies

Work is proceeding on the process design and cost estimate for a plant to make 500 billion Btu/day of pipeline gas from lignite.

A detailed material balance based on North Dakota lignite is nearly complete. This design includes slurry feed and a hydrogasifier system similar to the large pilot plant. Raw hot synthesis gas, generated by electrothermal gasification of lignite residue, will be used.

Methanation

Stirred reactor runs are continuing in order to determine more accurate kinetics for the reactions occurring in the methanator. The effect of pressure is being studied.

Calorimetry

The experimental program is near completion. A final report on the heat of reaction of hydrogen and coal and the heat capacity of coal is being prepared. The heat capacity of Al_2O_3 is being determined from 600° to 1500°F to check the calibrations at the beginning of this project.

Power Package

The three proposals received on the MHD power package were reviewed in November. Organizations submitting proposals were visited to talk with the people who would conduct the work. Our evaluation and recommendations will be submitted in December.

Pilot Plant

A preliminary screening of six engineering-construction firms was conducted to select organizations to bid on the detailed design, procurement, and construction of the pilot plant. These organizations were —

Ralph M. Parsons Company
Bechtel Corporation
Stearns-Roger Corporation
Blaw-Knox Company
J. F. Pritchard Company
Procon, Incorporated

All these organizations were invited to bid with the exception of Stearns-Roger Corp. We felt that this organization had very little experience in solids fluidization, which is a vital part of our system.

During the month, no new inventions were made in the course of the work.

Approved Jack Huebler Signed

Jack Huebler, Research Director Frank Schora, Associate Director

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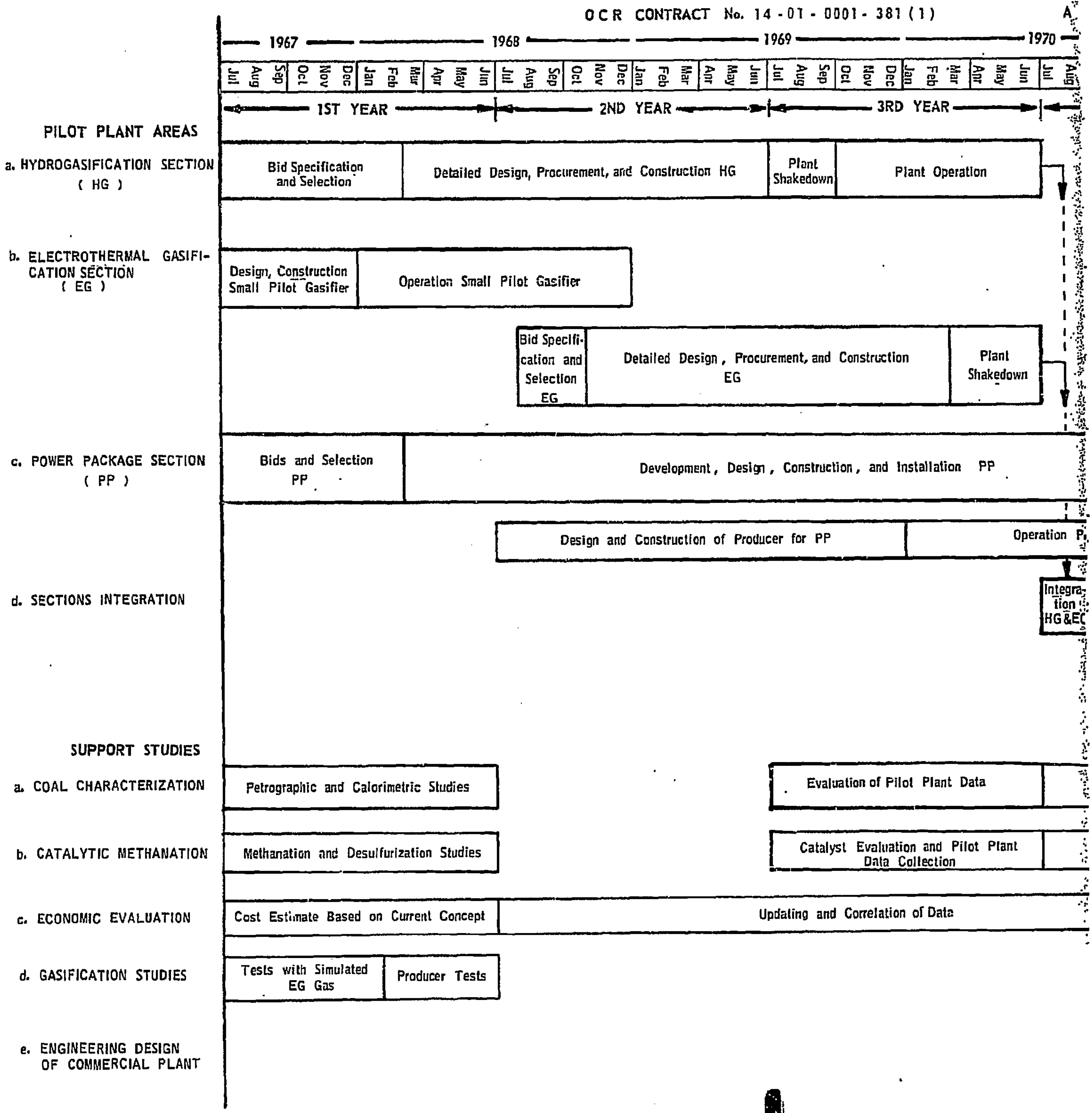

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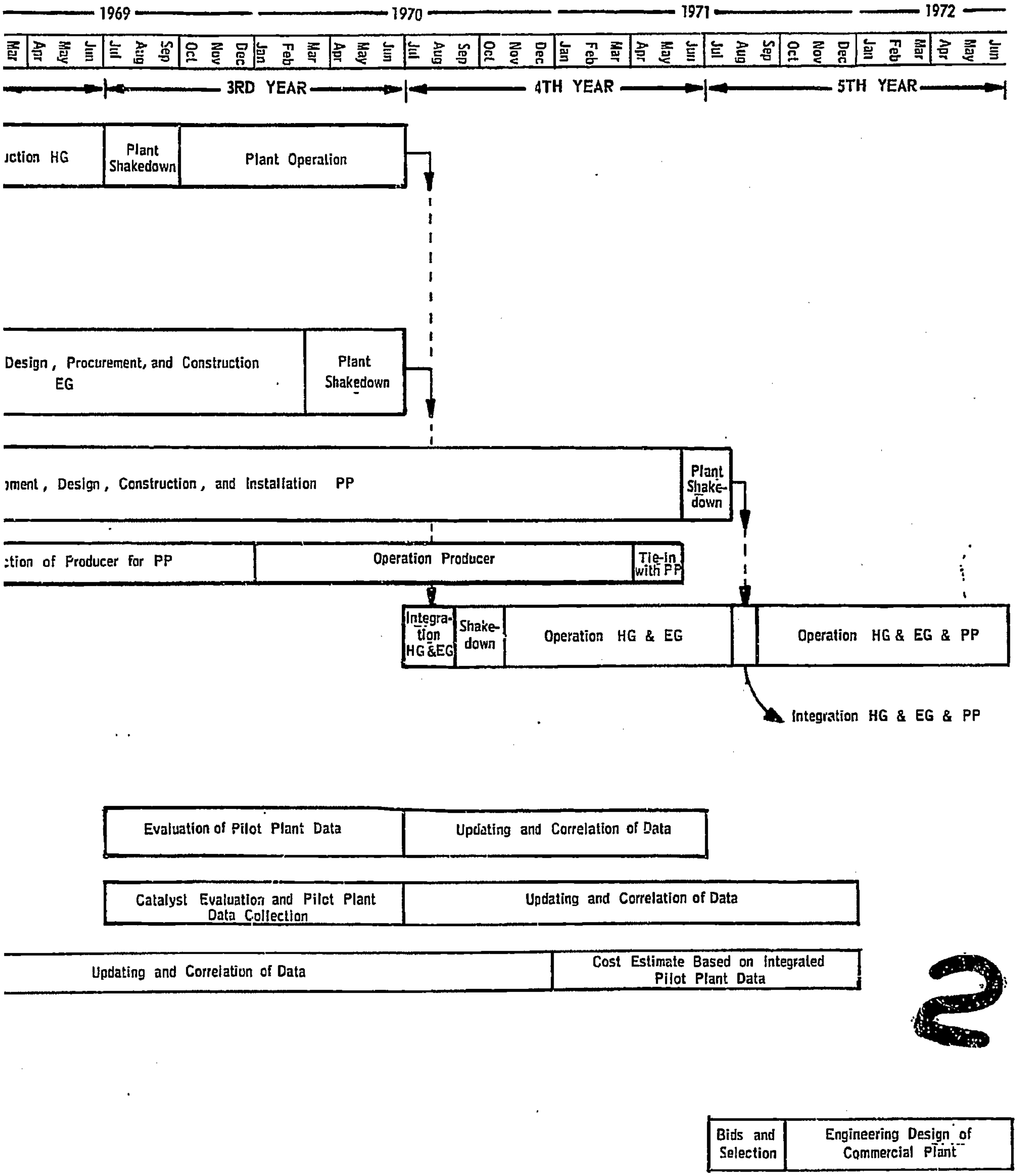

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DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS
Progress Report – December 1967
to
Office of Coal Research
Contract No. 14-01-0001-381(1)

Hydrogasification – Electrical Conductivity Tests

As a part of the study to establish the electrical characteristics of a fluidized bed of char at high pressure, we completed a series of five electrical conductivity tests in the balanced-pressure reactor. These tests are to establish power supply requirements for the 300-kw reactor. In these tests the char was fluidized with an equal-volume mixture of steam and nitrogen. Measurements were made of the resistivity of the char bed as electric power was applied to a 1/2-in.-diameter stainless steel rod immersed in the char to a depth of 1 ft. In four of these tests, we made measurements over a range of voltages up to 120 volts and over a range of fluidization gas velocities. To establish the effect of pressure, we conducted these tests at 200, 500, and 1000 psig.

To determine the electrical resistivity of the char bed at a higher voltage, we conducted several tests with 230-volt ac applied. At this high voltage, we measured an initial current flow of 250-800 amp for about 1 sec, followed rapidly by a rise to 1000 amp and above. These tests were conducted to examine the voltage-current relationship on an oscilloscope to determine if breakdown or arcing occurs at high current densities. It appears that such breakdown is not present.

Electrothermal Gasification

Discussions were continued with potential suppliers of the d-c power supply. We have requested quotations from three suppliers of used 600-volt d-c, 1000-amp motor generator sets. Such a unit appears to be best suited to match the load characteristics of the electrothermal reactor. Because of length of delivery and the initial cost, it is advisable to purchase a reconditioned unit. Such a unit will have a 1-year guarantee.

Power Package

We completed the review of proposals received for the MHD power package in December. Avco Corp. was recommended as the supplier.

Methanation

We are continuing runs to determine the effect of pressure on methanation rate. In the course of these runs we have noticed that CO_2 production is low and appears to decrease with increases in pressure. No ethane is formed with the 10% CO, 30% H_2, and 60% N_2 feeds over nickel-kieselguhr catalyst. Some evidence of a strong intraparticle diffusion effect has been noticed.

Coal Characterization

The bituminous coals that we have tested were reviewed by rank. This review showed a gap in the series between the West Virginia No. 5 Block with 39.0% volatile matter and the Sewell seam with 20.0% volatile matter. This occurred because the sample of Sewell seam coal that we obtained is of higher rank than expected. A 3-ton sample of Sewell seam coal from another mine, which is expected to have above 30% volatile matter, has been ordered.

We are obtaining reflectance readings on the hydrogasification residue from the Ireland mine, Illinois No. 6, Colorado subbituminous, and lignite coals.

Investigations of the solution of coal in benzene and any possible effect of solution on agglomeration after slurry feeding were continued.

During the month of December, no new inventions were made in the course of the work.

Approved Jack Huebler Signed
Jack Huebler, Research Director Frank Schora, Associate Director