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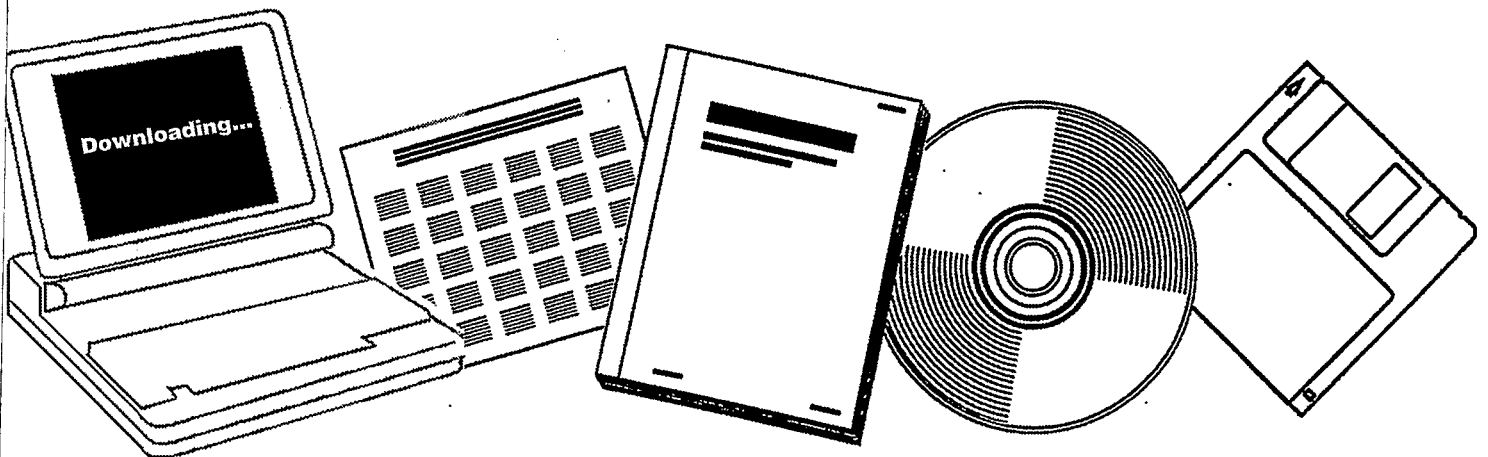
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**DEVELOPMENT OF IGT HYDROGASIFICATION
PROCESS. MONTHLY PROGRESS REPORTS,
JANUARY--OCTOBER; DECEMBER 1966**

**INSTITUTE OF GAS TECHNOLOGY, CHICAGO,
ILL**

1966



U.S. Department of Commerce
National Technical Information Service

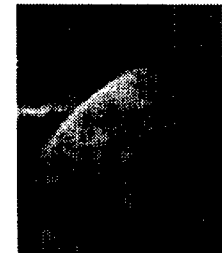
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FE--381-T-3

DEVELOPMENT OF IGT
HYDROGASIFICATION PROCESS

Monthly Progress Reports for the
Periods January - October and December 1966

Institute of Gas Technology
IIT Center
Chicago, Illinois 60616

Prepared for:

Office of Coal Research
U. S. Department of the Interior

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

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

CONTENTS

Monthly Progress Reports for each month January through
October and December 1966

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IGT-MPR--1/66

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS OFFICE OF COAL RESEARCH
RECEIVED
Progress Report - January 1966

to
Office of Coal Research
Contract No. 14-01-0001-581

1966 MAR 4 AM 11

DEPT OF THE INTERIOR

Hydrogasification

Five hydrogasification tests (Runs HT-89, HT-90, HT-91, HT-92, and HT-93) were made in the balanced-pressure pilot unit modified for fluid-bed operation. Tests were conducted with lightly pretreated Pittsburgh seam bituminous coal from the Ireland mine as the feed. The coal was reacted in a 5-1/2-ft-deep bed at 1500°F with a feed gas containing 45 mole percent hydrogen, 25 mole percent methane, and 30 mole percent steam. In Runs HT-89 to HT-92, the reaction was conducted in a fluidized bed; in Run HT-93 the reaction took place in a moving bed. All the tests were too short to reach steady-state operation, having had to be terminated in less than 1 hour because of difficulties in feeding the coal through the reactor feed tube.

The reasons for these difficulties varied for each run. In Run HT-88, reacted coal became compacted at the annulus between the feed gas distributing disk and the reactor wall. To remedy this situation for Run HT-89, the annulus was enlarged by reducing the gas distributor disk from 5-1/2 to 5-5/16 inches. And to eliminate coal agglomeration as a factor, the first 50 lb of coal fed to the reactor was a bituminous coal char with only 17% volatile matter. After about 1/2 hour of coal feeding, the coal injection tube became clogged and the feed screw jammed. The downward flow of coal through the injection tube, apparently, was sufficiently hindered by thermal updrafts in the reactor feed tube to create bridging in the tube. The normal nitrogen purge gas rate of 120 SCF/hr to the top of the reactor was not entirely adequate to keep the coal flowing.

In Run HT-90, the nitrogen purge rate to the feed tube was increased to 350 SCF/hr. Other operating conditions were similar to those of Run HT-89. The test had to be terminated 1/2 hour after coal feeding when an O-ring seal ruptured on one of the product gas filter bodies. Although the test was short, the increased nitrogen purge rate did improve the coal feeding, as coal did not plug in the injection tube.

Runs HT-91 and HT-92 were conducted at the same conditions as Run HT-88, except that the nitrogen purge rate to the top of the reactor was 350 SCF/hr. In both tests, the coal injection tube clogged 1/2 hour after the start of coal feeding. In Run HT-91, sharp variations in the reactor pressure were believed to be responsible for the blockage. In Run HT-92, a

sudden increase in the feed gas rate, which followed the addition of steam to the reactor, was apparently the cause of plugging.

Run HT-93 was conducted as a moving-bed test at coal and feed gas rates one-third of the those in the preceding fluidized-bed tests. The nitrogen purge rate was 350 SCF/hr. The objective of the test was to establish if the plugging of the coal injection tube, as experienced in fluidized-bed operation, was associated with high coal feed rates, or if other factors were responsible. The run proceeded smoothly for 53 minutes before the coal feed tube plugged, as previously. There were no pressure or flow disturbances that could account for the plugging. Apparently, the purge nitrogen did not provide the coal injection tube with sufficient gas sweep to keep the coal moving. To remedy this, the nitrogen will be injected through a nozzle at the top of the coal tube.

Coal Pretreatment

Four pretreatment tests were conducted in the fluidized-bed pretreatment pilot unit to supply the hydrogasification unit with nonagglomerating char.

Nominal conditions for the four tests are as follows:

Run No.	Solids Rate, lb/hr	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc. %	Bed Temp, °F
FP-50	45.4	1.03	646	9.4	754
FP-51	39.8	1.35	579	9.8	744
FP-52	41.4	1.55	589	9.8	730
FP-53	41.0	1.57	573	10.0	736

Ireland mine bituminous coal of -16, +80 mesh was used as the feed in the four tests. An air-nitrogen mixture of approximately 10% oxygen concentration was used as the pretreatment gas. Runs FP-50 and FP-51 were operationally successful, and were terminated at the depletion of the coal feed supply. Laboratory agglomeration tests showed the residues to be lightly caking but acceptable as feed for fluidized-bed hydrogasification tests. The desired run conditions during Run FP-52 were not reached due to the plugging of the internal cyclone. This caused erratic fluidization of the solids and subsequent termination of the test. Run FP-53 was conducted successfully; the laboratory agglomeration tests showed the char to be free-flowing.

The cyclone has been placed outside the reactor in the product gas off-take line between the reactor top and knockout drum. The cyclone and product lines are heated to facilitate solids separation of the off-take gas and to prevent the cyclone from plugging with solids.

Coal Characterization

Dynatech Corp. continued calibration of the heat-of-reaction calorimeter. An IGT staff engineer was sent to Dynatech for the initial calibration. At 1500°F and 1500 psia, the calorimeter body indicated nonuniform temperature. The instrument was modified by inserting a manually controlled heater plate at the bottom of the calorimeter receiver. Heat capacity of the calorimeter was calibrated in a helium atmosphere. The calibration curve exhibited an exponential decay instead of an expected straight-line decay, indicating the presence of convective heat transfer in the calorimeter and possible nonuniform temperature distribution around the calorimeter body. Calibration was also carried out in vacuum. The results were better than that in the helium calibration although exponential decay was also observed. The seal ring of the calorimeter body failed during hydrogen calibration. The calorimeter is being reassembled.

The drop calorimeter was calibrated for temperature rises of 14° and 5°F. It was then calibrated with alumina at 1200° and 1800°F. The calibrated heat capacity of alumina deviates from published values by less than 5%.

Coal Petrography

Residues from free-fall hydrogasification Runs HT-38 and HT-39, in which untreated Montour 4 coal was fed, were examined petrographically. The appearance of the particles, particularly the presence of many exterior wall perforations, is further evidence, by contrast, of the low reactivity of the pretreatment skin of pretreated feed particles.

A description of our apparatus and procedure for vacuum mounting of coal has been prepared and submitted to the ASTM D-5 task group that is working on standardization of coal petrographic methods.

Methanation

Further kinetic runs were made with Harshaw Ni-0104T catalyst to determine the dependence of the reaction rate on the partial pressures of the reactants and products. Data are now being analyzed.

Preliminary results indicate that the activity of Catalyst & Chemicals, Inc., nickel catalysts, C13-3 and C13-4, is much less than the activity of the Harshaw Ni-0104T.

Design has begun on a small fixed-bed reactor to study catalyst life. A tubular reactor of about 1-inch diameter should suffice for catalyst life study.

Engineering Economics

The study has been completed on the effect of carbon conversion on the 20-year average gas price (Table 1). The by-product char varied from 16.3% of the coal feed to 55.9%. The gas price reaches a minimum at about 35% byproduct char. Varying the credit taken for the byproduct char shifts the minimum only slightly. A separate memorandum will be issued with the complete results of this study.

Table 1.-EFFECT OF CARBON CONVERSION ON GAS PRICE

<u>Total Carbon Conversion, %</u>	<u>20</u>	<u>25</u>	<u>30</u>	<u>35</u>	<u>40</u>	<u>45</u>	<u>53</u>
Coal Feed, tons/day	30,804	26,436	23,160	19,812	17,216	15,612	13,296
Byproduct Char, tons/day	17,219	13,246	10,264	7,037	4,486	2,552	0
Byproduct Char, % of coal feed	55.9	50.1	44.3	35.4	25.9	16.3	0
Total Capital Investment, \$ million	158.04	145.52	140.45	138.94	145.71	144.45	147.22
20-Year Average Gas Price, \$ million Btu	58.5	55.6	54.0	53.7	54.8	55.3	57.8

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

Approved Jack Huebler Signed Frank C. Schora
 Jack Huebler, Associate Director Frank C. Schora, Manager

PROGRAM FOR DEVELOPMENT OF IGT H₂

OCR Contract No. 14-01-0001-381 A.G.A.

1964												1965																																																																																									
Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar																																																																																		
1st Year												2nd Year																																																																																									
PHASE I a. Hydrogasification b. Coal Pretreatment c. Methanation d. Coal Characterization	1			2			3			4			5			6			7																																																																																		
	Establish rate-controlling areas of operation E-13 T-36 DS-\$19,800 ODC-\$5,900			Study effect of degree of pretreatment E-5 T-16 DS-\$7,500 ODC-\$2,500			Study effects of variables to establish kinetics of fluid-bed system E-13 T-36 DS-\$15,600 ODC-\$4,000			Study fluidization and establish rates for fluid-bed operation E-10b T-27 DS-\$12,500 ODC-\$3,900			Study H ₂ O-D ₂ -C System E-3 T-6 DS-\$2500 ODC-\$1000			Study particle size effect for both fluid and fluid-bed operation E-9 T-27 DS-\$11,300 ODC-\$2,500			Study hydrogasification under actual conditions E-10 T-27 DS-\$12,500 ODC-\$3,900																																																																																		
	Modify coal pretreatment unit E-3 T-11 DS-\$4,250 ODC-\$6,000			Study effects of pretreatment variables on char properties E-4 T-22a DS-\$10,400 ODC-\$4,300						Establish pretreatment properties of various coals E-5 T-22a DS-\$12,400 ODC-\$4,300																																																																																											
	Review state of the art and design lab test unit E-7 T-0 DS-\$3,800 ODC-0			Build and shake down lab unit E-3 T-9 DS-\$8,150 ODC-\$3,500			Lab scale catalyst evaluation E-5 T-22 DS-\$6,850 ODC-\$1,250			Study effects of gas composition E-5 T-10 DS \$6,150 ODC-\$1,600			Design and construct small pilot unit E-5 T-18 DS-\$13,500 ODC-\$12,500			Run small pilot plant E-10 T-27 DS-\$12,500 ODC-\$3,900																																																																																					
	Develop techniques and study petrographic characteristics of coal, char, and spent hydrogasifier residue. Determine heats of reaction of coal and chars E-13 T-8 DS-\$2,500 ODC-\$2,500						Establish tests and develop correlations for estimation of worth of coal for hydrogasification E-14 T-11 DS-\$10,200 ODC-\$500																																																																																														
	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-12 T-0 DS-\$13,800 ODC-0																																																																																																				
	Develop simple math model of process based on present data E-5 T-0 DS-\$2,300 ODC-0			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 updating E-20 T-0 DS-\$9,200 ODC-\$1,500																																																																																																	
Make preliminary reactor design process concept and hydrogasifier design E-6 T-0 DS-\$2,800 ODC-0																																																																																																					
PHASE II a. Analysis of Experimental Data b. Process Concept Development c. Reactor Design Studies d. Economic Evaluation	1			2			3			4			5			6			7																																																																																		
	Cost data review and extension E-7 T-0			Develop simple flow sheet E-2 T-0 DS-\$3,900 ODC-0			Detailed flow sheets, energy and material balances E-3 T-0 DS-\$1,400 ODC-0			Process design of various sections E-4 T-0 DS-\$1,800 ODC-0			Cost estimate of equipment, plant and operating expenses E-3 T-0 DS-\$1,400 ODC-0			Report Preparation E-2 T-0 DS-\$900 ODC-0			Revision and updating of flow sheet E-5 T-0 DS-\$2,800 ODC-0																																																																																		
PHASE III a. Preliminary Pilot Plant Design	TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)																																																																																																				
	<table border="1" style="width: 100%; border-collapse: collapse; text-align: center;"> <thead> <tr> <th>TOTAL</th> <th>Aug</th><th>Sep</th><th>Oct</th><th>Nov</th><th>Dec</th><th>Jan</th><th>Feb</th><th>Mar</th><th>Apr</th><th>May</th><th>Jun</th><th>Jul</th><th>Aug</th><th>Sep</th><th>Oct</th><th>Nov</th><th>Dec</th><th>Jan</th><th>Feb</th><th>Mar</th> </tr> </thead> <tbody> <tr> <td>Planned</td> <td>855,400</td><td>23,500</td><td>23,500</td><td>23,500</td><td>23,800</td><td>22,400</td><td>24,100</td><td>24,300</td><td>24,700</td><td>23,700</td><td>23,300</td><td>23,800</td><td>30,300</td><td>29,300</td><td>27,800</td><td>27,400</td><td>28,100</td><td>20,100</td><td>23,300</td><td>23,300</td> </tr> <tr> <td>Actual</td> <td>10,000</td><td>15,000</td><td>23,300</td><td>14,500</td><td>23,000</td><td>21,100</td><td>19,800</td><td>28,900</td><td>23,400</td><td>30,200</td><td>24,500</td><td>22,900</td><td>25,900</td><td>21,900</td><td>26,900</td><td>27,800</td><td>22,300</td><td>23,400</td><td></td><td></td> </tr> <tr> <td>Cumulative</td> <td>10,000</td><td>25,000</td><td>48,300</td><td>62,800</td><td>85,800</td><td>106,900</td><td>126,700</td><td>155,600</td><td>179,000</td><td>209,200</td><td>233,700</td><td>256,600</td><td>282,100</td><td>304,000</td><td>330,900</td><td>358,700</td><td>381,000</td><td>404,400</td><td></td><td></td> </tr> </tbody> </table>																		TOTAL	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	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	20,100	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	25,900	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	
TOTAL	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar																																																																																	
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	20,100	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	25,900	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 of total costs.

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HYDROGASIFICATION PROCESS

31 A.G.A. PB-23a.

1966							1967										
Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
Year 1										3rd Year							

Study hydrogasification of various coals, flights, under most suitable conditions E-23 1/2 T-33 DS-327,200 ODC-37,300	Perform any support work necessary for phase III Double check data on which pilot plant is being designed
---	--

Obtain properties of various coals E-3 1/2 T-2 1/2 DS-512,500 ODC-34,500	Produce material needed for any hydrogasification runs
--	--

Run small pilot unit and develop kinetic equations for design E-10 T-19 DS-511,500 ODC-55,250	Perform any support work necessary for phase III
---	--

Develop correlations to enable both of cost for hydrogasification E-14 T-11 DS-510,200 ODC-4500

Laboratory and Pilot Plant Phase I-af, b4, c7	II-ac	Engineers Technicians	24	Salaries	140,400	Other Direct Costs	11,400
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Perform analysis of past data and new data in any areas necessary for phase III work
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Design

Make preliminary reactor design based on process forecast and hydrogasification results E-4 T-0 DS-52,800 ODC-0	Work with subcontractor on design of reactors for hydrogasification and methanation E-4 T-0 DS-51,600 ODC-0
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Revision and refinement of capital and operating costs E-5 T-0 DS-52,800 ODC-0	Work with subcontractor on capital and operating costs for pilot plant E-9 T-0 DS-34,100 ODC-0
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Develop pilot plant flowsheet with heat and material balances E-14 T-0 DS-57,700 ODC-0	With assistance of subcontractor work on phase III as outlined in Contract No. 14-01-0003-381. E-34 T-0 DS-516,300 ODC-0
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Select architect-engineer subcontractor * \$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	23,700	

Half of total costs: DS - Direct Salaries, \$
ODC - Other Direct Costs, \$

IGT-MPR--2/66

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - February 1966

to

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

Hydrogasification

Five hydrogasification tests (Runs HT-94, HT-95, HT-96, HT-97, and HT-98) were conducted in the balanced-pressure pilot unit modified for fluid-bed operation. A lightly pretreated Pittsburgh seam bituminous coal from the Ireland mine was reacted at a nominal temperature of 1300°F with a feed gas containing 45 mole percent hydrogen, 25 mole percent methane, and 30 mole percent steam. Runs HT-95 and HT-96 were conducted with a 3-1/2-ft fluidized coal bed, Run HT-94 with a 3-1/2-ft moving coal bed, and Runs HT-97 and HT-98 with a 7-ft moving coal bed. The coal feeding problems encountered in the earlier fluid-bed tests were, in general, eliminated in these tests by maintaining an adequate nitrogen purge rate to the coal injection tube. As a result, operating times for most of the tests were sufficiently long to yield operating data.

Run HT-94 was conducted with a moving coal bed to establish an adequate operating procedure that would remedy the earlier coal feeding problems encountered in the coal injection tube. A nitrogen purge tube, 1/8-inch ID, was installed to provide a high-velocity nitrogen jet at the mouth of the coal injection tube. Feed gas entered the reactor through a 3-5/16-inch-diameter porous metal disk at a velocity below that required for fluidization. Coal was fed at a rate of 22 lb/hr. The test lasted 3-1/2 hours and was terminated when difficulties developed in discharging solids from the reactor. Suspension of coal particles between the reactor walls and the gas distributor disk was found to be the cause of the difficulty. However, the test was long enough to indicate an improvement in the coal feeding techniques.

With successful coal feeding demonstrated in Run HT-94, a return to fluid-bed operation was made in Run HT-95. The 3-5/16-inch-diameter gas distributing disk was replaced with a 1/2-inch-diameter disk in order to increase the annular area between the disk and the reactor walls. However, when the small-diameter feed disk became partially plugged, the high feed gas rate required for coal-bed fluidization could not be maintained without an excessive pressure drop across the disk. A gas rate of approximately 80% of that required for fluidization was maintained in the test for 4 hours. The test was terminated when an adequate steam flow rate could no longer be maintained due to increasing pressure drop across the distributor disk.

For Run HT-96 a 2-7/8-inch-diameter feed gas distributing disk was installed. Operating conditions were similar to those of HT-95. The objective was to operate at fluidization gas velocities starting with a coal feed rate of 22 lb/hr and to eventually increase the coal feed rate to 65 lb/hr. The test was only partially successful, for it had to be terminated after 1-1/2 hours when the product gas line upstream of the bayonet filters became plugged with coal. The most likely cause of the plug was the flow of product gas with entrained coal particles up the solids return leg of the internal cyclone.

The objective of Run HT-97 was to partially hydrogasify, in a moving bed, a sufficient amount of coal to be used as feed in high-temperature fluid-bed tests. A 3/8-inch feed gas tube with eight 1/16-inch-diameter holes at the end was used, the tube extending up to the bottom of the furnace section. The solids return leg of the cyclone was removed and the opening was plugged. Approximately 70 lb of coal was processed in 3-1/4 hours before the test was stopped, due to an unexplained pressure upset in the reactor which caused the coal feed screw to jam.

Run HT-98 was a repeat of Run HT-97, with the same objective. The test lasted 9-3/4 hours - the longest hydrogasification run conducted in the balanced-pressure pilot unit. It was shut down when the product gas line upstream of the bayonet filter became filled with coal and restricted the outflow of the product gases.

Coal Pretreatment

Three pretreatment runs were conducted in the fluidized-bed pretreatment pilot unit to supply the hydrogasification unit with nonagglomerating char.

Nominal conditions for the tests were:

Run No.	Solids Rate, lb/hr	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Bed Temp, °F
FP-54	38.1	1.97	635	9.8	735
FP-55	38.0	2.05	615	9.7	721
FP-56	37.8	1.85	652	9.8	742

Ireland mine bituminous coal of -16+80 mesh was used as the feed in the three tests. An air-nitrogen mixture of approximately 10% oxygen was used as the pretreatment gas. Tests FP-54 and FP-56 were operationally successful and were terminated at the depletion of the coal feed supply. Laboratory agglomeration tests showed the char from FP-54 to be lightly caked, whereas the FP-56 char was free-flowing.

During Run FP-55 erratic bed movement and pretreatment gas channeling caused the bed to plug, which forced a shutdown of the test after 5 hours. Laboratory agglomeration tests showed the char to be caked.

Coal Characterization

Dynatech Corp. continued calibration of the heat-of-reaction calorimeter. The seal ring at the break of the calorimeter body has been a problem since testing began. Pressure-filled O-rings, vented O-rings, C-rings, and V-rings have at best taken the temperature cycle only three times before failure. On this basis, the downtime will be frequent and long. Also, part of the nonuniform temperature distribution in the calorimeter body could be due to the O-rings, which are sandwiched between the upper and lower body halves. The advantage of this design is that cleaning is easy. After careful evaluation of all the operational problems, it was decided that the calorimeter body halves should be welded into one piece. An opening for cleaning will be provided at the neck of the calorimeter drop tube at the lower part of the cold zone. The calorimeter has been modified and is now being assembled and pressure tested.

Coal Petrography

Arrangements have been made with J. A. Harrison and H. J. Gluskoter of the Illinois State Geological Survey to investigate etching of polished residue sections by means of electronically excited oxygen. This method for low-temperature ashing of organic materials has been used at the Survey on coal.¹ Application of this technique to polished sections of residue may make it possible to differentiate attrital vitrinite (which contains a great many small particles of inorganic matter) from non-attrital vitrinite.

An examination of products from both successful and unsuccessful pretreatment runs was initiated. The mode of agglomeration and particle properties characteristic of successful pretreatment will be studied.

During the month a patent memorandum was written which described an apparatus and method for determining the pore structure of porous solids. In this apparatus the amount of gas that flows into and out of the pores of a sample of a solid such as coal is instantaneously measured as the pressure of the surrounding gas is cyclically increased and decreased at a controlled rate. The size and volume of pores are determined from measurement of this flow at different rates of pressure oscillation.

¹ Gluskoter, H. J., "Electronic Low-Temperature Ashing of Bituminous Coal," Fuel 44, 285-91 (1965) July

Methanation

Methanation runs continued with Harshaw Ni-0104T nickel on kieselguhr and Girdler G-65 nickel on alumina catalysts. Higher temperature runs were emphasized (700°-900°F) to investigate possible carbon deposition and catalyst activity change. No noticeable carbon deposition was found with either catalyst up to 900°F. No data on activity change could be obtained because of an analytical equipment failure. At lower temperatures (500°-700°F) there is some production of ethane and propane. Above 700°F these hydrocarbons are cracked at a much higher rate than they are produced. At low space velocities (100 SCF/hr-lb cat.) and 900°F the ethane and propane were removed from the product with Ni-0104T as catalyst; with G-65, the product contained both hydrocarbons.

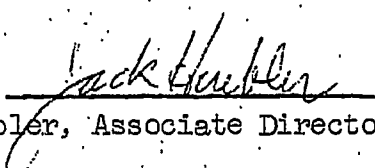
Four runs using 1/4-in. alumina spheres as catalyst were carried out with the 10:30 CO/H₂ feed gas at temperatures of 505°, 595°, 700°, and 804°F. These blank runs indicate that CO and CO₂ are not hydrogenated on alumina, but hydrocracking of ethane and propane does occur.

A new method of mounting catalyst pellets in the reactor has been investigated. Catalyst pellets are placed in a stationary annular screen basket which is placed in the reaction zone. Two propellers and a radial impeller are mounted on the shaft for gas circulation. With the catalyst stationary it is possible to measure the catalyst temperature accurately. The effect of agitator speed was checked by introducing feed at a constant rate to the reactor (Ni-0104T as catalyst) and varying shaft rpm at conditions to give high conversion of CO. No effect on conversion was found until the shaft speed was reduced to below 500 rpm. Pulse tracer tests indicate perfect mixing with this arrangement.

Equipment for catalyst life study is being ordered and construction of this facility has begun at the pilot plant.

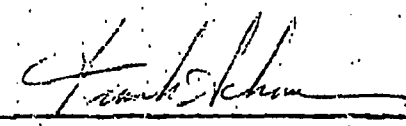
A 0.5% ruthenium on γ -alumina catalyst is now being tested. An ammonia synthesis catalyst, capable of producing longer chained hydrocarbons with low-temperature methanator feeds, has also been obtained. Results of tests with these catalysts will be available next month.

Approved



Jack Huebler, Associate Director

Signed



Frank Schora, Manager

PROGRAM FOR DEVELOPMENT OF IGT F

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

1964												1965											
1st Year												2nd Year											
Aug	Sept	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sept	Oct	Nov	Dec	Jan	Feb	Mar				
1												2											
PHASE I A. Hydrogasification Establish rate-controlling areas of operation E-13 T-34 DS-\$19,800 ODC-\$5,300												Study effect of degree of pretreatment E-6 T-18 DS-\$7,500 ODC-\$2,500											
Study effects of variables to establish kinetics of fixed-bed system E-13 T-36 DS-\$15,800 ODC-\$4,800												Study fluidization and establish rates for fluid-bed operation E-10 T-27 DS-\$12,500 ODC-\$3,800											
Study H ₂ O-DS-C System E-3 T-6 DS-\$3800 ODC-\$1200												Study particle size effect for both fixed and fluid-bed operation E-9 T-27 DS-\$13,300 ODC-\$2,000											
Study hydrogen under												Establish pretreatment properties of											
B. Coal Pretreatment Modify coal pretreatment unit E-3 T-11 DS-\$4,250 ODC-\$6,000												Study effects of pretreatment variables on char properties E-8 T-22 DS-\$10,400 ODC-\$4,000											
Produce material required for hydrogasification runs												Establish pretreatment properties of E-9 T-27 DS-\$13,300 ODC-\$2,000											
C. Methanation Review state of the art, and design lab test unit E-7 T-0 DS-\$3,800 ODC-0												Build and shake down lab unit E-3 T-9 DS-\$4,150 ODC-\$3,300											
Lab scale catalyst evaluation E-5 T-12 DS-\$6,850 ODC-\$1,250												Study effects of gas composition E-5 T-10 DS-\$6,150 ODC-\$1,000											
Design and construct small pilot unit E-5 T-18 DS-\$8,500 ODC-\$18,500												Run											
D. Coal Characterization Develop techniques and study petrographic characteristics of coal, char, and spent hydrogasified residue E-13 T-8 DS-\$8,900 ODC-\$20,500												Determine heats of reaction of coal and chars E-12 T-21 DS-\$10,200 ODC-\$500											
PHASE II a. Analysis of Experimental Data 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												Establish tests and develop correlations E-12 T-21 DS-\$10,200 ODC-\$500											
b. Process Concept Development Develop simple math model of process based on present data E-5 T-0 DS-\$2,300 ODC-0												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 preliminary reactor process concept and design E-6 T-0 DS-\$2,800 ODC-0												Make preliminary reactor process concept and design E-6 T-0 DS-\$2,800 ODC-0											
d. Economic Evaluation Cost data review and extension E-7 T-0 DS-\$3,200 ODC-0												Develop simple flow sheet E-2 T-0 DS-\$900 ODC-0											
Detailed flow sheet, energy and material balances E-3 T-0 DS-\$1,400 ODC-0												Process design of various sections E-4 T-0 DS-\$1,800 ODC-0											
Cost estimate of equipment, plant and operating expenses E-3 T-0 DS-\$1,300 ODC-0												Report Preparation E-2 T-0 DS-\$900 ODC-0											
Revision and updating of flowsheet E-6 T-0 DS-\$2,800 ODC-0												Make preliminary reactor process concept and design E-6 T-0 DS-\$2,800 ODC-0											
PHASE III A. Preliminary Pilot Plant Design												Make preliminary reactor process concept and design E-6 T-0 DS-\$2,800 ODC-0											

		TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)																			
		TOTAL																			
		23,500	23,500	23,500	23,800	22,400	24,700	24,300	24,700	23,700	23,300	23,100	30,300	29,300	27,800	27,400	28,100	28,100	23,300	23,300	23,300
Planned	\$55,400																				
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	28,900	21,900	26,900	27,600	22,300	22,400	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	285,500	307,400	334,300	358,700	381,000	403,400	426,700	

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

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

HYDROGASIFICATION PROCESS

PA PB-23a

1966							1967									
Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
3rd Year																

Gasification of various coals, lignite, or most suitable conditions. E-214 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.
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Gas of various coals	Produce material needed for any hydrogasification runs.
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Run a 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.
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Relations to enable hydrogasification	<table border="1" style="width: 100%; text-align: center;"> <tr> <td>Laboratory and Pilot Plant, Phase I-a8, b4, c7</td> <td>II-a2</td> <td>Engineers</td> <td>24</td> <td>Salaries</td> <td>540,400</td> </tr> <tr> <td></td> <td></td> <td>Technicians</td> <td>305</td> <td>Other Direct Costs</td> <td>11,400</td> </tr> </table>	Laboratory and Pilot Plant, Phase I-a8, b4, c7	II-a2	Engineers	24	Salaries	540,400			Technicians	305	Other Direct Costs	11,400
Laboratory and Pilot Plant, Phase I-a8, b4, c7	II-a2	Engineers	24	Salaries	540,400								
		Technicians	305	Other Direct Costs	11,400								

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

Reactor design based on 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
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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
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Develop pilot plant flowsheet with heat and material balances. E-14 T-0 DS-\$7,700 ODC-0	With assistance of subcontractor work on phase III as outlined in Contract No. 14-01-0001-181. E-30 T-0 DS-\$15,200 ODC-0
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* \$85,500 available for architect-engineer subcontractor and engineering consultants.

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

DS - Direct Salaries, \$
 ODC - Other Direct Costs, \$



IGT-MPR-3/66

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - March 1966

to

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

Hydrogasification

Seven hydrogasification tests (Runs HT-99, HT-100, HT-100A, HT-101, HT-102, HT-103, and HT-104) were conducted in the balanced-pressure pilot unit modified for fluid-bed operation. Three of these tests were conducted with a partially hydrogasified Pittsburgh seam bituminous coal in a simulation of the high-temperature hydrogasification stage. The coal was reacted in a 3-1/2-ft fluidized bed with a hydrogen-steam mixture containing 50 mole percent steam. In the four other tests, a lightly pretreated Pittsburgh seam bituminous coal from the Ireland mine was reacted in a 3-1/2-ft fluidized bed with a feed gas containing 45 mole percent hydrogen, 25 mole percent methane, and 30 mole percent steam. These four tests were a simulation of the low-temperature hydrogasification stage. Two of the tests (Runs HT-99 and HT-100A) with partially hydrogasified coal, and one of the tests (Run HT-104) with lightly pretreated coal, were successful. The other tests were terminated before steady-state operation was reached because of excessive leaks in the equipment or because of solids plugging in the reactor.

Run HT-99 was the first completely successful fluid-bed test. It lasted 3-3/4 hours and ended when the coal feed supply was used up. Although the target temperature for this run was 1700°F, the average bed temperature reached only 1310°F. The reasons for this were the high throughput rates, the limitation of the reactor furnaces, and the limitation of the feed gas preheat. The coal feed rate was 50 lb/hr, the hydrogen feed rate 635 SCF/hr, and the steam rate 30.2 lb/hr. Feed gas could not be preheated above 1000°F because of heat losses in the long feed gas transfer line. A modification of the product gas filtering equipment was made for Run HT-99. The internal cyclone was removed, and two 18-in.-long, porous metal bayonet filters were installed inside of the reactor. The outside bayonet filters were bypassed, and the product line was connected directly to the condenser from the top of the reactor.

To increase feed gas preheat, the feed gas transfer line was shortened and run directly from the gas-fired steam generator and gas preheater to the bottom of the reactor, bypassing the intermediate electric superheat furnaces. Auxiliary heating and insulation of the transfer line were improved so that the feed gas would enter the reactor at 1200°F. To improve heat transfer between the downflowing solids in the reactor and the

feed gas, a 108-1/2-in.-long, 1/2-in.-diameter U-tube was installed inside the reactor between the feed gas inlet and the feed gas distributor. The first test incorporating this modification had to be terminated 1/2 hr after coal feed was started because of excessive leaks in the new feed gas transfer line. However, the test demonstrated that a feed gas inlet temperature of 1250°F could be obtained with the new preheat system.

After the leaks were fixed, Run HT-100A was conducted at conditions similar to those of Run HT-99. The test lasted 4-1/4 hr, and was terminated when the coal feed supply was exhausted. A coal feed rate of 50 lb/hr was maintained for about 3 hr, and during this time the temperature of the fluidized bed reached 1500°F. The coal feed rate was then reduced to 40 lb/hr for the remainder of the test to investigate its effect on the bed temperature. After this reduction in the coal feed rate, the bed temperature increased to 1650°F, and was still rising when the test ended.

For Run HT-101, an annular feed gas distributor (3-1/2-in. OD x 1-3/4-in. ID) with a "Rigimesh" sintered disk was installed. This distributor replaced a 2-7/8-in.-diameter disk that was damaged in Run HT-100A. Run conditions for this test with lightly pretreated bituminous coal were: 1000 psig, 1300°F, 3-1/2-ft coal bed, 65 lb/hr coal feed rate, and hydrogen-methane-steam feed gas rate of 1640 SCF/hr. The run was started successfully at a coal rate of 22 lb/hr. After 40 min, the coal rate was increased to 65 lb/hr. Twenty minutes after this increase, and before the 3-1/2-ft coal bed level was reached, the coal feeder jammed. Packed coal at the throat of the coal feed tube caused the jamming. Agglomerated coal particles (as big as 1 in.) were also found in the reactor.

To provide for a higher rate of coal hydrogasification in suspension, and to lessen the tendency for agglomeration, the annular feed gas distributor was lowered from 108-1/2 to 62 in. above the bottom of the furnace for Run HT-102. This, in essence, increased the free-fall distance in the reactor. This test was conducted at conditions similar to those of Run HT-101. The test was shut down after 1 hr when coal agglomerated in the reactor just above the gas distributor. The agglomeration caused bridging and kept the coal from moving down in the reactor.

For Run HT-103 the annular gas distributor was removed and a 3/8-in. pipe, with a capped end, and with twelve 1/16-in.-diameter ports in the upper wall of the pipe, was installed. This was changed to minimize coal bridging due to agglomeration. The internal U-bend heat exchange tube in the feed gas line was removed to increase the free cross-sectional area in the lower end of the reactor. This tube was no longer necessary, since adequate preheat was provided by the gas-fired preheater after the transfer line was modified. The test was started at a coal feed rate of 22 lb/hr, and after 1 hr, it was increased to 65 lb/hr.

The run was shut down 15 min later because coal would no longer discharge from the reactor and the bed level began to rise. As in Run HT-102, agglomerated coal near the end of the feed gas tube caused bridging and stopped the coal flow.

Analysis of hydrogasification data from the low-temperature runs with pretreated bituminous coal and hydrogen-methane-steam feed gases in the current series of tests showed that carbon hydrogasification rates in the free-fall section of the reactor were apparently too low to keep some of the coal from agglomerating before it reached the bed. To increase the hydrogasification in the free-fall section, the temperature would have to be raised to at least 1300°F. To attain this temperature level, the reactor was preheated to 1500°F for Run HT-104. Furthermore, to maintain this temperature in the initial stages of the operation, the run was started at one-third of the coal and feed gas rates required for fluidized-bed operation (22 lb/hr coal, 380 SCF/hr hydrogen-methane, and 8 lb/hr steam). This minimized reactor cooldown during the bed buildup phase of operation. The run was continued at these low feed rates for 4 hr through about eight bed discharge cycles. The coal and feed gas rates were then increased, in two stages, to two-thirds of the usual fluidization rates. The test continued with these intermediate rates for 1 hr and then the rates were increased, again in two stages, to the fluidization rates. Successful fluid-bed operation was maintained for 3-3/4 hr until the test ended when the coal feed supply was exhausted. The test lasted a total of 8-3/4 hr. The reactor temperature in all three stages of the test was 1500°F. This temperature was maintained in the final fluidization stage with very little heat input from the furnace heaters, because most of the necessary heat was supplied by the preheated feed gas and the heat of the exothermic reaction. Residues from this run showed only negligible agglomeration of coal particles. The weight of the residue recovered indicated that approximately 30% of the coal was converted to gaseous products and to a small amount of liquid products.

Coal Pretreatment

Four pretreatment tests were conducted in the fluidized-bed pretreatment pilot unit to study minimum pretreatment conditions and to supply the hydrogasification pilot unit with non-agglomerating char.

Nominal conditions for the tests were:

Run No.	Solids Rate, lb/hr	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc. %	Bed Temp, °F
FP-57	40.2	1.53	624	9.72	749
FP-58	40.0	1.54	577	9.64	747
FP-59	49.4	1.21	635	5.28	737
FP-60	45.3	1.27	600	4.65	745

Ireland mine bituminous coal of -16+80 mesh was used as the feed in the four tests. Runs FP-57 and FP-58 were made with an air-nitrogen mixture having approximately 10% oxygen. The purpose of the two tests was to supply the pilot hydrogasification unit with nonagglomerating char. Laboratory agglomeration tests showed both residue chars to be slightly caked.

The objective of Runs FP-59 and FP-60 was to study the effect of a two-stage pretreatment process on the volatile content and agglomeration characteristics of the char produced. The tests were conducted with an oxygen/coal feed ratio of approximately one-half that of previously conducted one-stage tests (about 0.7 SCF/lb). The oxygen concentration of Run FP-59 was 5.28% and the O₂/coal feed ratio was 0.680 SCF/lb. The test was terminated when enough char was produced to conduct the succeeding tests. Laboratory agglomeration tests showed the char to be caked, and proximate analysis of the char indicated a 27.5% volatile content.

Run FP-60 was the second stage of this series of tests. The oxygen concentration in the pretreatment gas was 4.65% yielding an O₂/coal feed ratio of 0.617 SCF/lb. Laboratory agglomeration tests showed the char to be free-flowing. The volatile content of the char was 24.3%.

Coal Characterization

The heat-of-reaction calorimeter was assembled, pressure-tested, temperature-tested, and calibrated. The calorimeter was held at 1500°F for 3 weeks, and the mC_p (mass X heat capacity) of the calorimeter was calibrated once in a vacuum of 43 microns, twice in helium at 1500 psig, and twice in hydrogen at 1400 psig. The results correspond within 8%. An alumina sample was dropped into the calorimeter at 1500°F and 1500 psig of hydrogen. The measured heat absorbed is within 10% of the published heat capacity values. Two n-decane samples were dropped at 1500°F and 1500 psig of hydrogen, and temperature rises of 3.5° and 6.5°F were observed. Gas samples have been taken and the runs are now being analyzed.

The instrument was shipped from Cambridge, Massachusetts at the end of March and should arrive in Chicago during the first week of April.

The agglomerating tendency of Ireland mine pretreated coal was investigated. There was no indication of a cementing medium other than the coal particles themselves. It appears that the surfaces of some of the particles become liquid enough for them to partially fuse with other particles.

Particle densities and true densities were determined for several sieve fractions of pretreatment and hydrogasification residues, as part of a systematic examination of the coal at different stages of the process.

Work to improve mounting and polishing procedures was continued.

Methanation

Catalyst evaluation continued with the study of two catalysts capable of producing longer-chained hydrocarbons at low temperatures: 0.5% ruthenium on γ -alumina, and an ammonia synthesis catalyst. The yields of long-chain gaseous hydrocarbons with both catalysts were low (less than 2%), with the ruthenium outperforming the ammonia synthesis catalyst. More liquid products were obtained with the ruthenium, although liquid yields were low. Reaction temperatures with both catalysts varied between 350° and 900°F, and both low- and high-CO feed gases were used. At the same temperatures and feed rates, 94 grams of the ruthenium catalyst removed much more CO than either a 222- or 283-gram sample of the ammonia synthesis catalyst.

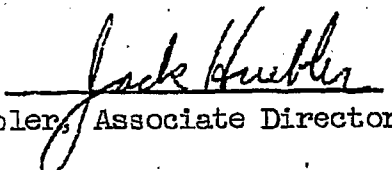
The low yields of longer chained hydrocarbons caused speculation that hydrogenolysis of these hydrocarbons might be occurring on the reactor walls. To study this effect, runs are being made without catalyst with both the regular feed gases and with a 50% H₂/50% natural gas (containing considerable C₂+) feed at 500°-900°F.

Equipment for catalyst life testing has been ordered, and although some equipment will not be received before June 1, preliminary operation of the unit should be possible by next month.

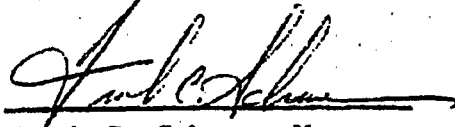
Investigation has begun on a preliminary determination of the type of equipment necessary for combustion of the spent hydrogasification char. Combustion of this char is necessary for both steam and hydrogen production. The possibility of running preliminary burning tests to establish combustion characteristics of the char was discussed with Dr. Essenhigh of Pennsylvania State University. Other people competent in this area are also being contacted.

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

Approved


Jack Huebler, Associate Director

Signed


Frank C. Schora, Manager

PROGRAM FOR DEVELOPMENT OF IGT H

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

	1964												1965															
	Aug	Sept	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sept	Oct	Nov	Dec	Jan	Feb	Mar								
	1st Year												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-\$8,000				4. Study fluidization and establish rates for fluid-bed operation E-10 ¹ T-27 DS-\$12,500 ODC-\$3,000				5. Study H ₂ O-DS-C System E-3 T-0 DS \$1800 ODC-\$1000				6. Study particle size effect for both fixed and fluid-bed operation E-9 T-27 DS-\$11,300 ODC-\$2,000				7. Study hydrogasification under normal conditions E-10 ¹ T-27 DS-\$12,500 ODC-\$3,000			
b. Coal Pretreatment	1. Modify coal pretreatment unit E-3 T-11 DS-\$2,250 ODC-\$6,000				2. Study effects of pretreatment variables on char properties E-4 ¹ T-22 ¹ DS-\$10,400 ODC-\$4,000												3. Establish pretreatment properties of WHI E-5 ¹ T-27 ¹ DS-\$12,600 ODC-\$4,500											
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-0 DS-\$4,150 ODC-\$3,500				3. Lab scale catalyst evaluation E-5 T-12 DS-\$5,850 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				6. Run small kinetic runs 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 correlations for estimation of worth of coal for hydrogasification E-14 T-11 DS-\$10,200 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-12 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	1. Make preliminary reactor process concept and hydrogasification flow-sheets E-4 T-0 DS-\$2,800 ODC-0																											
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-\$1,300 ODC-0				5. Cost estimate of equipment, plant and operating expenses E-3 T-0 DS-\$1,400 ODC-0				6. Reactor Preparation E-2 T-0 DS-\$900 ODC-0				7. Revision and updating of flow-sheet 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	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23
Planned	855,600	23,500	21,500	21,500	21,800	22,400	24,100	24,300	24,700	22,700	23,100	23,800	30,300	29,100	27,800	27,400	28,100	28,100	22,300	23,400	22,300	25,400	22,300	25,000
Actual		10,000	15,000	23,300	14,500	23,000	21,100	19,800	26,900	23,400	30,200	24,500	22,900	29,500	21,900	26,900	27,800	22,300	23,400	22,300	25,400	22,300	25,000	
Comulative		10,000	25,000	48,300	62,800	85,800	106,900	126,700	153,600	179,000	209,200	233,700	256,600	282,100	304,000	330,900	358,700	381,000	404,400	426,700	441,700			

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

Costs shown are costs to OCR of one-half of total costs. DDC

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GT HYDROGASIFICATION PROCESS

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

1966												1967						
Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
2nd Year												3rd Year						

Study hydrogasification of various coals, lignite, under most suitable conditions. E-214 T-67 DS-\$27,200 ODC-\$7,000	Perform any support work necessary for phase III Double check data on which pilot plant is being designed
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Pretreatment properties of various coals E-54 T-274 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-\$3,250	Perform any support work necessary for phase III
---	--

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

Laboratory and Pilot Plant Phase I-28, 34, 67 II-27	Engineers 24 Technicians 105	Salaries \$40,400 Other Direct Costs 11,400
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Perform analysis of past data and new data in any areas necessary for phase III work
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additional ted

Make preliminary reactor design based on process concept and hydrogasification results E-4 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
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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
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Develop pilot plant flow sheet with heat and material balances E-14 T-0 DS-\$7,700 ODC-0	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
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Select architect-engineer subcontractor * \$85,580 available for architect-engineer subcontractor and engineering consultants.

23,300	23,300	23,300	23,300	23,300	23,300	23,300	23,300	15,400	15,400	15,400	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700	
23,400	22,300	25,000																		
490	428,700	481,700																		

For example of total costs. DS - Direct Salaries, \$
 ODC - Other Direct Costs, \$



IGT-MPR-4/66

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - April 1966

to

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

Hydrogasification

Six fluidized-bed hydrogasification tests (Runs HT-105, HT-100, HT-107, HT-108, HT-109, and HT-110) were conducted in the modified balanced-pressure pilot unit. In five of these tests a lightly pretreated Pittsburgh seam bituminous coal from the Ireland mine was reacted in a 3-1/2-ft fluidized bed with a feed gas containing 45 mole percent hydrogen, 25 mole percent methane, and 30 mole percent steam. The sixth test was set up for hydrogasification of untreated Pittsburgh seam coal following start-up and 2-3 hour operation with pretreated coal. All of these tests were a simulation of a low-temperature hydrogasification stage. Successful fluid-bed operation was achieved in three of the tests (Runs HT-105, HT-106, and HT-109). The other tests were terminated before steady-state operation was reached because of solids plugging in the reactor.

Run HT-105 was started up following the same procedure used successfully in Run HT-104 (First Quarter, 1966, Project Status Report). This procedure involves preheating the reactor to 1500°F and starting the operation at one-third the coal and feed gas rates required for fluidized-bed operation (65 lb/hr coal, 1140 SCF/hr hydrogen-methane, and 23 lb/hr steam, at fluidized conditions). When fluidized-bed operation is reached, the reactor temperature is reduced to the desired temperature of 1300°F. The run lasted 7-1/2 hours with about 1 hour of operation at the fluidized-bed feed gas rates and a coal rate of 55 lb/hr. About 1/4 hour after the coal rate was increased to 65 lb/hr, the feed screw jammed when coal plugged in the 1-in.-diameter coal injection tube.

To improve the flow of coal through the coal injection tube for Run HT-106, the nitrogen purge tube to the top of the reactor was increased from 0.125 to 0.152 inch diameter. Run conditions and start-up for this run were the same as for Run HT-105. This run lasted 6-1/2 hours, terminating for the same reason as Run HT-105. Termination again was within 1/4 hour after full coal and feed gas fluidization were established.

Only brief periods of fluidized-bed operations at the desired coal feed rates were achieved in both Runs HT-105 and HT-106, but the start-up procedures were demonstrated to be effective.

For Runs HT-107 and HT-108 the coal injection feed tube and the insert body supporting the feed tube were removed. As this provided a full 4-inch-diameter cross-section for the coal to flow through from the end of the feed screw to the heated section of the reactor tube, any holdup in the feeding of coal would be minimized. Run conditions and start-up were the same as for Run HT-106, except that in Run HT-108 the reactor was preheated to 1700°F. Also, the upper product gas bayonet filter was removed for Run HT-108 to reduce product gas-coal contact near the feed screw outlet. Both tests were stopped within 45 minutes of starting the coal feed because of coal plugging in the unheated portion of the reactor tube. The apparent cause of plugging was a too rapid heatup of the coal by-product gas in the upper section of the reactor tube near the bayonet filter.

As operation without the coal injection tube was shown unfeasible, the coal injection tube was again installed for Run HT-109. Three product-gas sampling probes and three internal coal bed thermocouples, extending from the top of the reactor to the coal bed, were removed. We found that these had coiled; they were probably partially responsible for intermittent coal holdups in the reactor in previous operations. Reactor heating Zone 3 (from the top of the reactor), which we found not operating, was repaired by eliminating a short in a power input electrode. Run HT-109 was conducted at the same conditions as Run HT-106. It was fully successful in that a 1-1/2-hour steady-state operation at 1300°-1350°F bed temperature was achieved. Based on the weight of the residue recovered, approximately 35% of the coal had been reacted. Total operating time for the test was 5 hours. Shutdown was due to an inadvertent shutoff of the steam generator gas burner because of a faulty interlock device. This caused a partial condensation of the steam in the feed gas line, resulting in a pressure upset that caused a blockage in the coal injection tube.

After the successful demonstration of fluid-bed operation with pretreated bituminous coal, consideration was given to the use of untreated bituminous coal for fluid-bed hydrogasification. The scheme for untreated bituminous coal hydrogasification involved a start-up and the establishment of a fluid-bed regime using pretreated coal, to be followed by the addition of the raw coal. Run HT-110 was set up to do this by charging the bottom of the coal feed hopper with pretreated coal sufficient for 2-3 hours of operation, with untreated coal on top. Run conditions and start-up were the same as for Run HT-109. The test lasted only 1 hour; it was terminated when the feed screw jammed. This was caused by raw coal plugging in the coal injection tube and in the reactor tube about 6 ft below the top of the heated zone. Feeding of the raw coal apparently started about 50 minutes after the coal feed screw was started. Disarrangement of the original stratification of the coals in the hopper was the probable cause for the short time interval before the raw coal reached the feed

screw. This loss of stratification occurred when the feed hopper was being pressurized from the bottom.

Coal Pretreatment

Four pretreatment tests were conducted in the fluidized-bed pretreatment pilot unit to study minimum pretreatment conditions and to supply the hydrogasifier with nonagglomerating char.

Nominal conditions for the tests were:

<u>Run No.</u>	<u>Avg Solid Res Time, hr</u>	<u>Feed Gas Rate, SCF/hr</u>	<u>Oxygen Conc, %</u>	<u>Bed Temp, °F</u>
FP-61	2.19	609	9.79	746
FP-62	2.21	602	9.34	749
FP-63	1.23	595	9.67	748
FP-64	1.19	593	9.77	747

Bituminous coal from Consolidation Coal Co.'s Ireland mine was crushed to -16+80 mesh and used as the feed for these four runs. The operating conditions used were about the same as those used in FP-56 which produced a free-flowing char. Run FP-61 lasted for 3-3/4 hours; then the feed screw jammed against its housing. The residue char was found to be very lightly caked. Run FP-62 was stopped after 12 hours when the bottom of the fluidized bed began to defluidize. The char was found to be very lightly caked. In Run FP-63 the feed rate was increased to give a shorter solids residence time, while the other operating conditions remained the same. The run was terminated after 12 hours when the unit ran out of feed. The agglomeration test showed the char to be very lightly caked. The last run for this period was FP-64 which was a duplication of FP-63 with the run again being stopped when the feed supply was exhausted. The char was found to be free-flowing.

With Run FP-64, some innovations to the gas cleanup systems were made to protect the product gas meter from damage caused by entrained oils and tars. The water used for scrubbing in the venturi scrubber was changed to continuous flow rather than full recycle. The oils and tars that are being scrubbed out are now being collected and reported as part of the pretreater residues. A second Cuno filter was installed in parallel with the first to allow for the changing of filter cartridges during a run.

Methanation

The possibility of longer chained hydrocarbons being hydrogenolyzed on the reactor walls was investigated. A feed of 2.1% ethane, 0.4% propane, 47.7% hydrogen, and the balance methane and C₄+ was fed at low flow rates to the empty reactor over a temperature range of 790°-1010°F. Over 90% conversion

of ethane is possible with this feed and gas composition. There were no changes in the ethane, propane, or C₄+ compositions. This indicates that the absence of appreciable amounts of longer chained hydrocarbons in runs using iron and ruthenium catalyst is probably due to hydrogenolysis occurring simultaneously with the Fischer-Tropsch synthesis. Iron catalysts are known to catalyze hydrogenolysis at high pressures and temperatures with high hydrogen concentrations.

Catalyst reactivity data indicate the Harshaw Ni-0104 nickel on kieselguhr is the best commercial catalyst for cleanup methanation of hydrogasifier product. We will begin experiments to establish a rate model for cleanup methanation using this catalyst.

Further construction of the catalyst life testing reactor depends on delivery of controlling and recording instruments, which are expected this month.

Coal Characterization

The heat-of-reaction calorimeter was assembled at IGT and was vacuum and pressure tested. The mCp (mass X heat capacity) of the calorimeter is being determined at various temperatures. This calibration is to determine the behavior of mCp over the temperature range within which the heat-of-reaction of hydrogen and coal will be determined. Also, the single calibration performed at Dynatech Corp. will be double-checked.

The previous n-decane test was found to have an h_r (heat of reaction of n-decane and hydrogen) value of 3575 Btu/lb. Based on the heat of formation of methane at 25°C, the calculated h_r value is 3223 Btu/lb. This difference of 10% may be due to the inaccuracy of the estimated value of the heat of formation of methane at the actual experimental conditions.

Petrographic work on the systematic examination of coal at different stages of hydrogasification was continued. Hydrogasification residue is being examined by X-ray diffraction to determine whether any conversion to graphite has occurred. It was found that mineral matter, particularly clay, interferes with the determination and must first be removed.

We are now planning to include in the hydrogasifier run schedule several tests to investigate producing a producer gas from spent hydrogasifier char at high pressure. We plan to fluidize the bed of spent char with steam, nitrogen, and air to establish the rates of the reactions involved in production of carbon monoxide and hydrogen. This information will be very useful for better defining a program for the production of hydrogen from char.

No inventions were made during April in the course of this work.

Approved

Jack Huebler

Signed

Frank C. Schora

Jack Huebler, Associate Director

Frank C. Schora, Manager

PROGRAM FOR DEVELOPMENT OF IGT H

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

	1964												1965											
	AUG	SEP	OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC	JAN	FEB	MAR				
	1st Year												2nd Year											
PHASE I	1		2			3			4			5			6			7						
a. Hydrogasification	Establish rate-controlling areas of operation E-13 T-36 DS-\$19,800 ODC-\$5,500		Study effect of degree of pretreatment E-6 T-18 DS-\$7,300 ODC-\$2,500			Study effects of variables to establish kinetics of fixed-bed system E-13 T-36 DS-\$15,800 ODC-\$4,000			Study fluidization and establish rates for fluid-bed operation E-10 ^{1/2} T-27 DS-\$12,500 ODC-\$3,000			Study H ₂ O-Co-C System E-3 T-9 DS-\$1800 ODC-\$1000			Study particle size effect for both fixed and fluid-bed operation E-9 T-27 DS-\$11,500 ODC-\$2,000			Study hydrogasification under new						
b. Coal Pretreatment	Modify coal pretreatment unit E-3 T-11 DS-\$4,250 ODC-\$6,000		Study effects of pretreatment variables on char properties E-4 ^{1/2} T-23 ^{1/2} DS-\$10,400 ODC-\$4,000						Establish pretreatment properties of new E-5 ^{1/2} T-27 ^{1/2} DS-\$12,600 ODC-\$4,500						Produce material required for hydrogasification runs									
c. Methanation	Review state of the art and design lab test unit E-7 T-0 DS-\$3,800 ODC=0		Build and shake down lab unit E-3 T-9 DS-\$4,150 ODC-\$3,500			Lab scale catalyst evaluation E-5 T-12 DS-\$4,850 ODC-\$1,250			Design and construct small pilot unit E-5 T-11 DS-\$8,500 ODC-\$18,500			Study effects of gas composition E-5 T-10 DS-\$6,150 ODC-\$3,000			Run small kinetic									
d. Coal Characterization	Develop techniques and study petrographic characteristics of raw, char, and spent hydrogasified residue. Determine heats of reaction of coal and chars E-13 T-8 DS-\$9,800 ODC-\$20,500						Establish tests and develop correlations & estimation of worth of coal for hydrogasification E-14 T-11 DS-\$10,200 ODC-\$500																	
PHASE II	1																							
a. Analysis of Experimental Data	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	Develop simple math model of process based on present data E-3 T-0 DS-\$2,500 ODC=0												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-\$3,700											
c. Reactor Design Studies	Make preliminary reactor process concept and hydro E-6 T-0 DS-\$2,800 ODC=0																							
d. Economic Evaluation	Cost data review and extension E-7 T-0		Develop simple flow sheet E-2 T-0 DS-\$3,200 ODC=0		Detailed flow sheet, energy and material balances E-3 T-0 DS-\$1,400 ODC=0		Process design at various sections E-4 T-0 DS-\$1,800 ODC=0		Cost estimate of equipment, plant and operating expenses E-3 T-0 DS-\$1,400 ODC=0		Report Preparation E-2 T-0 DS-\$900 ODC=0		Revision and updating of flowsheet E-6 T-0 DS-\$2,800 ODC=0											
PHASE III	1																							
a. Preliminary Pilot Plant Design																								

	TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)																						
	TOTAL	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
Planned	\$55,400	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	23,300		
Actual	10,000	15,000	23,300	14,500	23,000	21,100	19,800	28,900	25,400	30,200	24,500	22,900	29,500	21,000	28,800	27,800	22,500	23,400	22,500	28,000			
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	330,800	358,700	381,000	404,400	428,700	481,700			

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

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

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ST HYDROGASIFICATION PROCESS

381 A.G.A. PB-23a

1966							1967										
Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
2nd Year										3rd Year							

<p>7</p> <p>Study hydrogasification of various coals, lignite, under most suitable conditions</p> <p>E=214 T=41 DS=\$27,200 ODC=\$7,000</p>	<p>8</p> <p>Perform any support work necessary for phase III</p> <p>Double check data on which pilot plant is being designed</p>
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<p>9</p> <p>Determine properties of various coals</p> <p>E=54 T=274 DS=\$12,480 ODC=\$2,500</p>	<p>9</p> <p>Produce material needed for any hydrogasification run</p>
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<p>6</p> <p>Run small pilot unit and develop kinetic equations for design</p> <p>E=10 T=14 DS=\$12,480 ODC=\$5,250</p>	<p>7</p> <p>Perform any support work necessary for phase III</p>
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<p>2</p> <p>Run and develop correlations to enable use of work of coal for hydrogasification</p> <p>E=14 T=11 DS=\$10,200 ODC=\$500</p>

<p>Laboratory and Pilot Plant Phase I-4B, 4C, 4D, 4E, 4F, 4G, 4H, 4I, 4J, 4K, 4L, 4M, 4N, 4O, 4P, 4Q, 4R, 4S, 4T, 4U, 4V, 4W, 4X, 4Y, 4Z</p>	<p>II-12</p>	<p>Engineers Technicians</p>	<p>24 107</p>	<p>Salaries Other Direct Costs</p>	<p>\$40,400 \$1,480</p>
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	<p>7</p> <p>Perform analysis of past data and new data to any extent necessary for phase III work</p>
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<p>Additional</p>

<p>5</p> <p>Make preliminary reactor design based on process concept and hydrogasification results</p> <p>E=4 T=0 DS=\$2,800 ODC=0</p>	<p>2</p> <p>Work with subcontractor on design of reactor for hydrogasification and combustion</p> <p>E=4 T=0 DS=\$2,800 ODC=0</p>
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<p>8</p> <p>Revise and refinement of capital and operating costs</p> <p>E=4 T=0 DS=\$2,800 ODC=0</p>	<p>2</p> <p>Work with subcontractor on capital and operating costs for pilot plant</p> <p>E=4 T=0 DS=\$2,800 ODC=0</p>
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<p>1</p> <p>Develop pilot plant flowsheet with heat and material balances</p> <p>E=14 T=0 DS=\$7,700 ODC=0</p> <p>Select mechanical engineer subcontractor</p>	<p>2</p> <p>With assistance of subcontractor work on phase III as outlined in Contract No. 24-D1-0001-381.</p> <p>E=34 T=0 DS=\$16,180 ODC=0</p> <p>\$45,500 available for architect-engineer subcontractor and engineering consultants.</p>
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300	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	22,500	25,900	32,600																	
104,400	426,700	481,700	484,300																	

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



967-MPR--5/66

DEVELOPMENT OF ICF HYDROGASIFICATION PROCESS

Progress Report - May 1966

to

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

Hydrogasification

Five hydrogasification tests (Runs HT-111, HT-111A, HT-112, HT-113, and HT-114) were conducted in the balanced-pressure pilot unit. Two of these tests were conducted with a partially hydrogasified Pittsburgh seam bituminous coal in a simulation of a high-temperature hydrogasification stage. The coal was reacted in a 3-1/2-ft-deep fluidized bed with a hydrogen-steam mixture containing 50 mole percent steam. In the three other tests, a lightly pretreated Pittsburgh seam bituminous coal was reacted in a 7-ft-deep fluidized bed in which the temperature at the top of the bed was held at 1300°F, and the temperature at the bottom at 1700°F. The purpose of these tests was to combine the low-temperature and the high-temperature stages in a single operation. One of the high-temperature tests (Run HT-111A) and one of the dual-temperature fluidized-bed tests (Run HT-113) were successful. The other tests were terminated before steady-state operation was reached because of plugs in the coal injection tube, or a plug in the product gas line.

In Run HT-111, partially hydrogasified Pittsburgh seam bituminous coal from low-temperature Runs HT-104 and HT-105 was reacted at 1700°F in a 3-1/2-ft fluidized bed with a mixture of hydrogen and steam. The coal feed rate was 40 lb/hr; the hydrogen/coal ratio was 30% of stoichiometric; and the steam concentration was 50 mole percent. This test had to be shut down 15 minutes after starting when the coal injection tube plugged. The plugging was caused by the caking of a small quantity of unpretreated bituminous coal that had remained in the feed screw housing from the previous test (Run HT-110) and was conveyed into the feed tube.

Run HT-111A was conducted under the same conditions as Run HT-111. It was a completely successful high-temperature run, which was terminated after 5-1/2 hours when the coal feed supply was nearly spent. Based on the weight of the residue recovered, approximately 25% of the coal was converted.

The objective of Run HT-112 was to hydrogasify a lightly pretreated Pittsburgh seam coal in a single stage with a temperature of 1300°F at the top and 1700°F at the bottom. In this test, the low-temperature and high-temperature stages were carried out in one operation. To do this, the top three zones of the reactor tube were preheated to 1300°F, and the bottom four zones

were preheated to 1700°F. The coal feed rate was 40 lb/hr; the hydrogen rate was 530 SCF/hr (35% of stoichiometric); and the steam rate was 25.3 lb/hr (50 mole percent of the feed gas mixture). After 1-3/4 hours the test had to be ended when the coal jammed in the injection tube. The jamming was caused by a pressure upset in the reactor induced by hydrogen-steam feed pulsations. Partially plugged distributing parts in the hydrogen-steam feed tube were responsible for the feed pulsations. Examination of the feed tube after the run showed that the parts were plugged by metal scale.

Run HT-113 was conducted under the same conditions as HT-112. It was a successful test of 5-1/2 hours duration. Termination was voluntary after a 3-1/2-hour steady-state period. Approximately 50% of the coal was converted to gaseous and liquid products, based on the weight of the recovered residue.

The objective of Run HT-114 was to maintain a temperature gradient through the coal bed as in HT-113, but to hydrogasify at a lower hydrogen/coal ratio. To maintain fluidization velocities, coal and hydrogen throughput rates were correspondingly increased to 60 lb/hr and to 600 SCF/hr, respectively. The hydrogen/coal ratio at these rates was 25% of stoichiometric. The steam rate was 28.6 lb/hr (50 mole percent). The test proceeded smoothly for 2-1/2 hours when a sudden failure of the product gas bayonet filter caused a shutdown just as steady state was being reached.

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.

Nominal conditions for the tests were:

Run No.	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Bed Temp, °F
FP-65	0.85	589	4.01	738
FP-66	1.07	689	3.81	738
FP-67	1.22	618	4.34	740
FP-68	0.66	831	17.01	734

Bituminous coal crushed and screened to -16+80 mesh from Consolidation Coal Co.'s Ireland mine was used as the feed for three of the runs. Run FP-67 used the char produced in Run FP-66 as feed. Run FP-65 was an attempt to start a two-stage test, but the run was stopped prematurely when the reactor top product line plugged with tars and fine coal particles. We found that the strip heaters around the reactor top had burned out and allowed the tars to condense on the reactor top and outlet. The char was caked. Run FP-66 was essentially a repeat of FP-65 but with a higher gas feed rate. Since fluidization during the start of the

run was not satisfactory, the gas rate was increased to correct this condition. Run FP-66 was shut down after 15-3/4 hours when the coal feed ran out. The char was found to be caked. It had 25-26% volatile matter. The second stage of this test was FP-67, which was operated at conditions similar to those of Run FP-66. The run was stopped after 14-1/2 hours when the feed char was used up. The product char was found to be free flowing and had 22-24% volatile matter.

The coal was subjected to the following conditions during the two-stage run:

<u>Run No.</u>	<u>Total Solids Res Time, hr</u>	<u>Total Oxygen/Coal Fed, SCF/lb</u>	<u>Total Gas/Coal Fed, SCF/lb</u>
FP-66-67	2.29	1.16	25.02
FP-59-60	1.85	1.58	16.09
FP-56	2.48	1.30	25.02

These combined conditions are somewhat less severe than those of the previous two-stage run (FP-59-60), which also produced a free-flowing char. But there does not seem to be any advantage in two-stage operation over the single-stage Run FP-56 which produced a free-flowing char.

Run FP-68 was made at high gas and solids rates and high oxygen concentration. The high oxygen concentration allowed the pretreater to run without any heat input because of the amount of exothermic heat produced during the reactor at these conditions. The run was terminated when the bottom of the fluidized bed began to defluidize and cool. The air-metering system would not allow the higher gas rates needed to correct the poor fluidization and still maintain temperature and oxygen concentration. The char was found to be lightly caked and had 27.2% volatile matter.

Methanation

Isothermal kinetic runs are being made with Harshaw Ni-0104T nickel-on-kieselguhr catalyst. Data obtained from these runs will be used to develop a methanation rate applicable to the three typical feed gases. The effects of temperature and pressure on the parameters of the rate law will then be determined.

The equipment for the catalyst life study was received, enabling completion of the unit. We expect catalyst life studies to begin in June.

Coal Characterization

The heat-of-reaction calorimeter was calibrated at 1000 psia hydrogen and temperatures of 800°, 900°, 1100°, and 1500°F. The results were satisfactory.

The heats of reaction of raw coal (Ireland mine) with hydrogen at 1000 psia were measured at 1000°, 1300°, and 1500°F. Data are being analyzed.

The coal was analyzed both before and after the reaction to obtain information on the percent of coal gasified. Gas samples were taken after each reaction to determine the gases produced.

The first series of tests is to study the effect of temperature on the heat of reaction of hydrogen and coal. A second series of tests will study the effect of partially gasified coals on the heat of reaction with hydrogen.

Dr. Essenhigh has been retained to perform some char reactivity tests to develop information for the design of a char producer. Also in this area of activity, Babcock & Wilcox (Operations) Limited has been contacted regarding its work with pulverized coal vortex combustion systems. In the U.S., all vortex units have been run as cyclone combustors with coarse coal. Due to the need for a small combustion volume (because of pressure considerations), a vortex system appears attractive at this time.

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

Signed



Frank C. Schora, Manager

PROGRAM FOR DEVELOPMENT OF IGT

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

1964												1965															
1st Year												2nd Year															
Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb									
PHASE I																											
a. Hydrogasification																											
1 Establish rate-controlling areas of operation E-13 T-34 DS-112,000 ODC-33,100				2 Study effect of degree of pretreatment E-6 T-16 DS-172,000 ODC-12,500				3 Study effects of variables in existing system E-13 T-36 DS-215,000 ODC-18,000				4 Study fluidization and establish rates for fluid-bed operation E-10 T-27 DS-112,500 ODC-33,000				5 Study H ₂ O-DS-C System E-3 T-9 DS-138,000 ODC-12,000				6 Study particle size effect for new fixed and fluid-bed operation E-5 T-27 DS-113,300 ODC-12,500				7 Study hydro			
b. Coal Pretreatment																											
1 Modify coal pretreatment unit E-3 T-11 DS-54,250 ODC-16,000				2 Study effects of pretreatment variables on char properties E-4 T-22 DS-110,400 ODC-14,000										3 Establish pretreatment procedure E-5 T-27 DS-112,000 ODC-14,500													
Produce material required for hydrogasification runs																											
c. Methanation																											
1 Reverse state of the art, and design lab test unit E-7 T-0 DS-13,800 ODC-0				2 Build and start down lab unit E-3 T-9 DS-14,150 ODC-12,500				3 Lab scale catalyst evaluation E-5 T-12 DS-16,850 ODC-11,250				4 Study effects of gas composition E-5 T-10 DS-56,150 ODC-12,000				5 Design and construct small pilot unit E-5 T-18 DS-14,500 ODC-11,500											
d. Coal Characterization																											
1 Develop techniques and study petrographic characteristics of coal, char, and spent hydrogasified resid. Determine heats of reaction of coal and chars E-13 T-3 DS-56,900 ODC-120,500														2 Establish costs and develop complete estimation of worth of coal for by													
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-12 T-0 DS-113,000 ODC-0																											
b. Process Concept Development																											
1 Develop simple math model of process based on present data E-5 T-0 DS-12,100 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 up-to-date E-28 T-0 DS-19,200 ODC-11,500																							
c. Reactor Design Studies																											
1 Make preliminary process concept and E-1 T-0 DS-0 ODC-0																											
d. Economic Evaluation																											
1 Cost data review and extension E-7 T-0		2 Develop simple flow sheet E-2 T-0 DS-13,200 ODC-0		3 Detailed flow sheet, energy and material balances E-3 T-8 DS-11,400 ODC-0		4 Process design of various sections E-4 T-8 DS-11,800 ODC-0		5 Cost estimate of equipment, plant and operating expenses E-3 T-0 DS-11,400 ODC-0		6 Report preparation E-2 T-0 DS-9,700 ODC-0		7 Revision and updating of flowsheet E-6 T-8 DS-12,800 ODC-0															
PHASE III																											
a. Preliminary Pilot Plant Design																											

		TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)																			
		TOTAL	23,500	23,500	23,500	23,900	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
Planned	855,400																				
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	28,900	27,800	22,300	23,400	22,300	2
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	4

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

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

HYDROGASIFICATION PROCESS

A.G.A. PB-23a

	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
	1st Year																

Run hydrogasification of various coals, lignite, under most favorable conditions E - 21 T - 61 DS - \$17,200 ODC - \$7,000	Perform any support work necessary for phase III Double check data on which pilot plant is being designed
--	--

Properties of various coals E - 59 T - 276 DS - \$17,600 ODC - \$6,500	Produce material needed for any hydrogasification run
--	---

Run small outlet unit and develop kinetic equations for design E - 10 T - 19 DS - \$11,600 ODC - \$5,350	Perform any support work necessary for phase III
--	--

Map correlations to enable coal for hydrogasification E - 11 T - 10 DS - \$10,200 ODC - 0	Laboratory and Pilot Plant Phase I-28, 84, c? II-aZ Engineers 28 Technicians 103 Salaries \$40,400 Other Direct Costs 11,400
---	--

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

Preliminary reactor design based on I and hydrogasification results E - 6 T - 0 DS - \$2,800 ODC - 0	Work with subcontractor on design of reactor 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	15,800	15,800	15,800	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700	23,700
OD	25,000	32,000	25,000														
700	461,700	484,300	509,300														

Costs: DS - Direct Salaries, \$
 ODC - Other Direct Costs, \$

2

IGT-MPR -- 6/66

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - June 1966

to

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

Hydrogasification

Six hydrogasification tests (Runs HT-115, HT-116, HT-117, HT-118, HT-119, and HT-120) were conducted in the balanced-pressure pilot unit modified for fluid-bed operation. In these tests a lightly pretreated Pittsburgh seam bituminous coal was reacted in a fluidized-bed reactor maintained at two temperature levels. The top of the reactor (the free-fall space above the bed) was controlled at 1300°F, and the bottom of the reactor (the fluidized-bed portion) was controlled at 1700°F. Feed gas was a mixture of hydrogen and steam containing 50 mole percent steam. Four of these tests (Runs HT-115, HT-116, HT-118, and HT-119) were partially successful in that steady-state operation was attained, but they were either of short duration, at a bed height below the desired 7 feet, or with feed gas rates less than those required for fluidization. Run HT-119 was terminated because of plugged feed gas tube ports, while the remaining tests were shut down because of coal feed injection tube plugging.

To help in preventing blockage of the coal feed, as experienced in the recent series of tests, a motor-driven stirrer was installed in the coal feed tube for Run HT-120 and run at a low speed (5-10 rpm). It extended the length of the feed tube and projected beyond the end by 1 inch. The stirrer was a 1/8-inch-diameter stainless steel rod formed into a spiral 3/4-inch OD with a 3-inch pitch. By operating continuously while coal was being fed, the stirrer would prevent deposits from building up inside of the feed tube. In this test, a plug developed in the feed gas transfer line and the run had to be terminated after 1-1/4 hours. The plug of metal scale developed at a feed gas transfer line fitting.

Coal Pretreatment

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

Nominal conditions for the tests were:

Run No.	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Avg Bed Temp, °F
FP-69	0.55	800	21.0	726
FP-70	0.52	1003	21.0	707
FP-71*	0.30	800	21.0	775
FP-71A	0.34	789	19.2	767
FP-72*	0.50	700	21.0	775
FP-72A	0.49	674	21.0	775

* Intended run conditions, but not attained.

Bituminous coal crushed to -16+80 from Consolidation Coal's Ireland mine was used as the feed for these runs. All six tests were attempts to produce free-flowing chars with a relatively high volatile content (26-28%) with only air as the feed gas. This series of runs demonstrated the higher throughput capacity that the pretreater unit is capable of achieving.

Runs FP-69 and FP-70 had a bed height of 7 feet. The remaining four runs had a bed height of only 4 feet to reduce the coal residence time and the amount of gas needed for good fluidization. These runs further demonstrated that the amount of exothermic heat of reaction produced with high oxygen concentrations is sufficient to maintain the bed temperature without additional heat input.

The first 100% air run was FP-69. The run was terminated after 4 hours because the product line became plugged with condensed tars and fine solids. The char was found to be heavily caked with 27-28% volatile matter.

Methanation

Methanation runs at 550°F have been carried out using the three typical hydrogasifier products as feeds. These runs cover a range of CO compositions in the methanation product of 1.0 to 4.5%. Several rate models are being tested for fit to these data. More data will be needed in the regions below 1.0% CO and above 4.5% CO at 550°F for accurate rate model determination.

The catalyst life test unit construction is complete and equipment calibration has begun.

Coal Characterization

The heat capacity of the calorimeter metal was determined at 200°, 1000°, 1300°, and 1500°F with the drop calorimeter. The results are slightly lower than those obtained from the heater calibration.

The heat-of-reaction calorimeter was disassembled for cleaning operations and thermocouple repairs. It was then re-

assembled and pressure tested. The heat of reaction of pretreated coal with hydrogen is being determined.

Engineering Economics Studies

A modification of the cost estimate for production of high-Btu gas by the IGT hydrogasification process is now under way to establish the effects of new experimental results on gas costs. Most changes are a result of using a fluidized bed rather than a moving bed for contacting gases and solids in the hydrogasifier section.

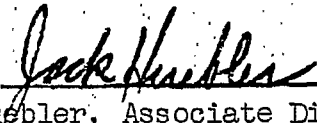
Preliminary contacts have been made with several engineering firms concerning the preliminary design of a 1-3 ton per hour pilot plant. Firms contacted are:

C. F. Braun & Co.
Bechtel Corp.
Foster-Wheeler Corp.
Cameron and Jones, Inc.
Stone and Webster, Inc.
Fluor Corp., Ltd.
The Lummus Company

We plan to invite two of these firms to submit formal proposals. Our selection of the two firms will be based on their experience with fluidization, solids handling, and high-pressure operation. Their interest in working in the area of coal technology will also be considered.

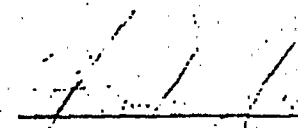
No new inventions were made during the month in the course of this work.

Approved



Jack Huebler, Associate Director

Signed



Frank C. Schora, Manager

PROGRAM FOR DEVELOPMENT OF IGT H²

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

	1964							1965												
	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar
	1st Year														2nd Year					
PHASE I	1		2			3			4		5		6			7				
a. Hydrogasification	Establish rate-controlling areas of operation E-13 T-36 DS-\$15,800 ODC-\$5,500		Study effect of degree of pretreatment E-8 T-18 DS-\$7,500 ODC-\$2,500			Study effects of variables to establish kinetics of fixed-bed system E-13 T-36 DS-\$12,800 ODC-\$4,000			Study fluidization and establish rates for fluid-bed operation E-10 ¹ T-27 DS-\$12,500 ODC-\$3,000		Study H ₂ O-DS-C System E-3 T-4 DS-\$3800 ODC-\$1000		Study particle size effect for both fixed and fluid-bed operation E-9 T-27 DS-\$11,300 ODC-\$2,000			Study hydrogasific under most				
b. Coal Pretreatment	Modify coal pretreatment unit E-3 T-11 DS-\$4,250 ODC-\$6,000		Study effects of pretreatment variables on char properties E-44 T-224 DS-\$10,400 ODC-\$4,000			Establish pretreatment properties of variol E-5 ¹ T-27 ¹ DS-\$12,800 ODC-\$4,500			Produce material required for hydrogasification runs											
c. Methanation	Review state of the art, and design lab test unit E-7 T-0 DS-\$3,400 ODC-0		Build and shake down lab unit E-3 T-9 DS-\$4,150 ODC-\$1,500			Lab scale catalyst evaluation E-5 T-12 DS-\$6,850 ODC-\$1,250		Study effects of gas composition E-5 T-10 DS-\$6,150 ODC-\$1,000		Design and construct small pilot unit E-5 T-18 DS-\$8,500		Run small kinetic								
d. Coal Characterization	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-\$6,900 ODC-\$20,500						Establish tests and develop correlations to estimation of worth of coal for hydrogasifi E-14 T-11 DS-\$10,200 ODC-\$500													
PHASE II	1																			
a. Analysis of Experimental Data	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	Develop simple math model of process based on present data E-5 T-0 DS-\$2,300 ODC-0					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 preliminary reactor des process concept and hydrogasifi E-6 T-0 DS-\$2,800 ODC-0																			
d. Economic Evaluation	Cost data review and extension E-7 T-0 DS-\$3,200 ODC-0		Develop simple flow sheet E-2 T-0 DS-\$300 ODC-0		Detailed flow sheet, energy and material balances E-3 T-0 DS-\$1,400 ODC-0		Process design of various sections E-4 T-0 DS-\$1,800 ODC-0		Cost estimate of equipment, plant and operating expenses E-3 T-0 DS-\$1,400 ODC-0		Report Preparation E-2 T-0 DS-\$900 ODC-0		Revision and updating of flowsheet E-6 T-0 DS-\$2,800 ODC-0			Revis capiti				
PHASE III	1																			
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	Mar
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	23,300
Actual		10,000	15,000	23,300	14,500	23,000	21,100	19,800	28,900	25,400	30,200	24,500	22,900	25,800	21,900	26,900	27,800	22,300	23,400	22,300	25,000
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	282,400	304,000	330,900	358,700	381,000	404,400	426,700	461,700

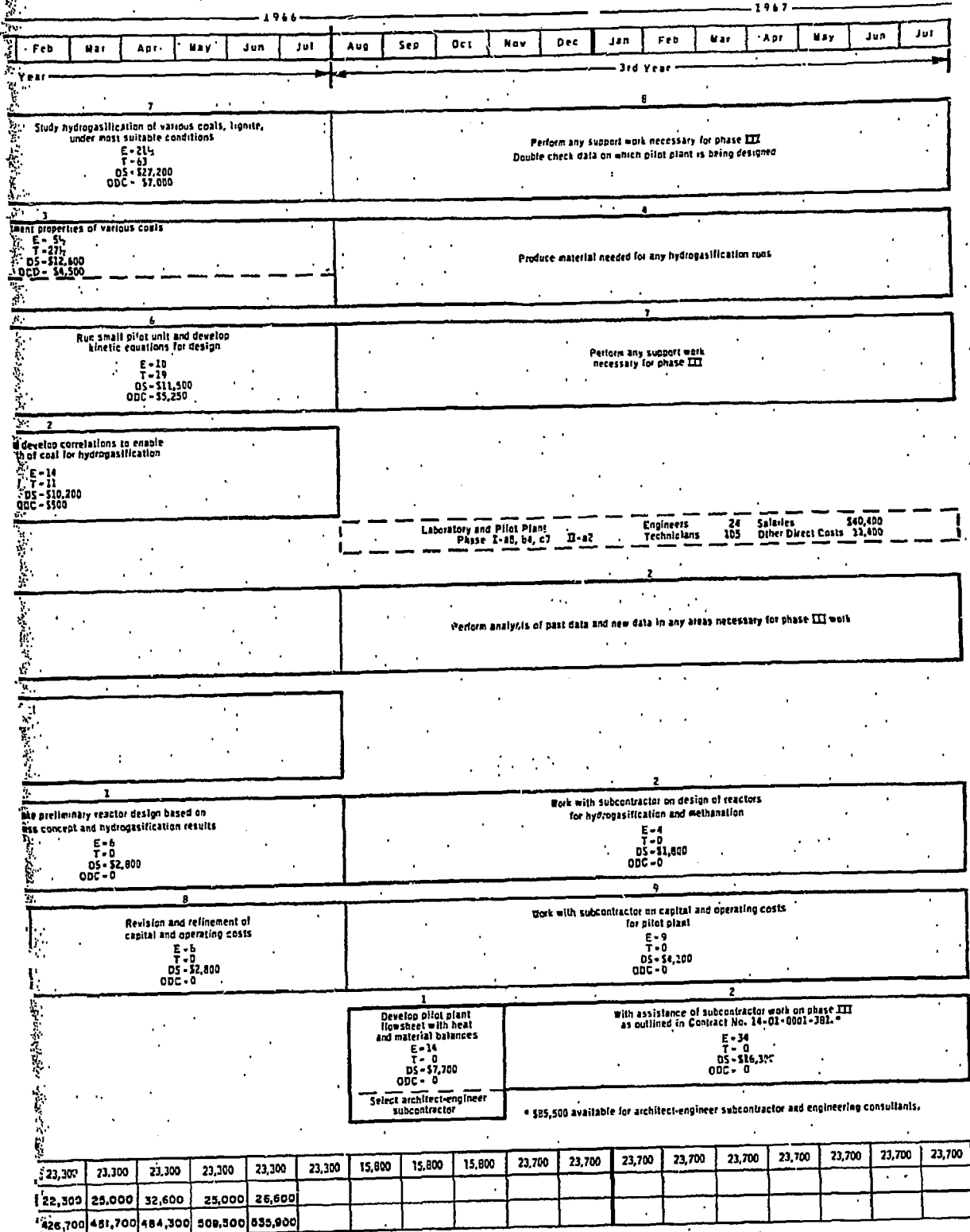
Manpower shown in total man months: E = Engineers
T = Technicians

Costs shown are costs to OCR or one-half of total costs. DS = D
ODC = O

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T HYDROGASIFICATION PROCESS

31 A.G.A. PB-23a:



of total costs. DS - Direct Salaries, \$
ODC - Other Direct Costs, \$

2

IGT-MPR--7/66

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - July 1966

to

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

Hydrogasification

Seven hydrogasification tests (Runs HT-121 to HT-127) were conducted in the balanced-pressure pilot unit. All were conducted with a lightly pre-treated Pittsburgh seam bituminous coal (Ireland mine) which was reacted with a hydrogen-steam feed gas mixture in either a 3-1/2- or 7-ft-deep fluidized bed. Temperatures at the top of the bed and in the free-fall space above the bed were held at 1300°F; the temperature at the bottom of the coal bed was controlled at 1700°F in Run HT-121 and at 1800°F in the other tests. The purpose of these tests was to combine the low-temperature and the high-temperature hydrogasification stages in a single operation. Nominal coal feed rates for the first six tests were 53 lb/hr, the hydrogen/coal ratio 25% of stoichiometric, and the steam concentration 50 mole percent. For Run HT-127 the corresponding nominal feed rates were 74 lb/hr of coal, 25% of the stoichiometric hydrogen/coal ratio, and 30 mole percent steam. Two of these dual-temperature tests (Runs HT-121 and HT-126) were completely successful; one (Run HT-125) was partially successful. The other tests were terminated before steady-state operation was reached because of mechanical difficulties.

Run HT-121 was a successful dual-temperature test conducted with a 7-ft-deep fluidized bed. After initially establishing a 3-1/2-ft coal bed, the coal bed height was increased in stages to 5 ft and finally to 7 ft. The entire test lasted 6 hours, of which nearly 3 hours was with a 7-ft coal bed. Carbon gasification was 34% of the carbon in the coal. A product gas of 568 Btu/SCF (nitrogen-free basis) was produced.

Hydrogasification conditions for Run HT-125 were similar to those for Run HT-121, except that the bottom of the coal bed was controlled at 1800°F to check if additional steam reaction could occur. This test was partially successful in that steady-state operation was established with a 3-1/2-ft fluidized coal bed but not at the higher bed height. Carbon gasified in the 3-1/2-ft coal bed was about 38% while producing a product gas of 534 Btu/SCF (nitrogen-free basis).

Run HT-126 was conducted to check if the bed behavior and temperatures along the entire reactor length would reach steady values at extended duration. The entire test was, therefore, conducted with a 3-1/2-ft coal bed. The test was completely successful, lasting over 6 hours.

Coal Pretreatment

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

Nominal conditions for the tests were:

Run No.	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Avg Bed Temp, °F
FP-73	0.46	783	16.47	772
FP-74	0.56	765	16.19	769
FP-75	0.35	578	21.0	726
FP-76	0.43	778	16.04	736
FP-77	0.40	727	16.44	738
FP-78*	0.20	1000	21.0	775

* Intended run conditions, but not attained.

Bituminous coal crushed and screened to -16+80 mesh from Consolidation Coal's Ireland mine was used as the feed for Runs FP-73, FP-74, and FP-78. The feed for the remaining three runs were chars produced in previous runs that were found to be highly agglomerating. In these runs, the chars were fed through the pretreater again at high solids rates and high oxygen concentration to bring down the volatile matter and make the chars non-agglomerating. The table below compares the feeds to the three runs and the resulting product chars.

Run No.	Avg % Volatiles		Agglomeration	
	in Feed	in Product	Feed	Product
FP-75	27.8	26.2	Fused	Free-Flowing
FP-76	25.2	24.1	Caked	Free-Flowing
FP-77	25.9	24.3	Caked	Free-Flowing

Methanation

Kinetic runs were made with Harshaw Ni-0104 T nickel on kieselguhr at low space velocities and temperatures of 550⁰ to 800⁰F. It was possible to reduce the CO content of the low-CO feed gas (2.8% CO, 11.8% H₂) to about 0.15% at as low as 550⁰F. A maximum dry product gas heating value of 996 Btu/SCF was obtained at 550⁰F.

To study the methanation reaction at high CO concentrations (5-10 %), higher space velocity runs are being made this month.

The catalyst life test unit is in operation. An iron-ammonia synthesis catalyst will be studied first in an attempt to increase higher hydrocarbon production through fixed-bed operation.

Coal Characterization

We are continuing the study of the agglomerating tendency of pretreated coal with the hypothesis that particles having no oxidation skin are the ones that agglomerate. Results of measurements on two samples of Ireland coal, one that caked in the agglomeration test and one that did not, agreed with this hypothesis. A similar test on Broken Arrow coal did not agree, but it appears that this agglomeration test was in error.

Minor modifications are being made on the heat-of-reaction calorimeter to eliminate the effect of heat input to the calorimeter bomb. Some early tests show that the first-phase hydrogasification reaction of the most reactive carbon is definitely exothermic.

IGT consultant, Professor R. H. Essenhigh of Pennsylvania State University, reported on the first combustion tests of the hydrogasifier residue. The char showed excellent burning behavior with a stable flame. The peak wall temperature on the furnace was between 1300° and 1350°C, comparable with the best run on raw Pittsburgh seam bituminous coal. He sees no reason, therefore, why there should be any difficulty with ignition, even under pressure, of this residue in a char producer.

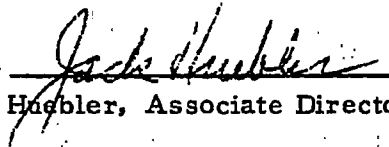
Engineering Economics Studies

Modifications of the cost estimate for production of high-Btu gas by the IGT hydrogasification process are continuing.

Two contractors, Bechtel Corp. and Foster Wheeler Corp., have been asked to submit proposals to conduct preliminary design and cost estimates of the 1-3 ton/hour pilot plant.

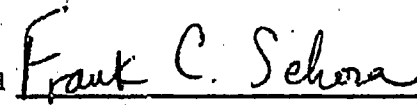
No new inventions were made during the month in the course of this work.

Approved



Jack Haebler, Associate Director

Signed



Frank C. Schora, Manager

PROGRAM FOR DEVELOPMENT OF IGT HY

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

	1964												1965																
	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar									
	1st Year												2nd Year																
PHASE I	a. Hydrogasification	Establish rate-controlling areas of operation E-13 T-36 DS-\$15,800 ODC-\$5,500				Study effect of degree of pretreatment E-8 T-18 DS-\$7,500 ODC-\$2,500				Study effects of variables to establish kinetics of fixed-bed system E-13 T-36 DS-\$15,800 ODC-\$4,000				Study fluidization and establish rates for fluid-bed operation E-10 1/2 T-27 DS-\$12,500 ODC-\$3,000				Study H ₂ O-O ₂ -C System E-3 T-8 DS-\$3,000 ODC-\$1,000				Study particle size effect for both fixed and fluid-bed operation E-9 T-27 DS-\$11,700 ODC-\$2,000				Study hydrogasification under most			
		b. Coal Pretreatment	Modify coal pretreatment unit E-3 T-11 DS-\$4,250 ODC-\$4,000				Study effects of pretreatment variables on char properties E-4 T-22 1/2 DS-\$10,400 ODC-\$4,000								Establish pretreatment properties of various E-5 1/2 T-27 1/2 DS-\$12,400 ODC-\$4,500														
	c. Methanation		Review state of the art, and design lab test unit E-7 T-11 DS-\$3,800 ODC=0				Build and shake down lab unit E-3 T-9 DS-\$4,150 ODC-\$3,500				Lab scale catalyst evaluation E-5 T-12 DS-\$6,350 ODC-\$1,250				Design and construct small pilot unit E-5 T-18 DS-\$8,500 ODC-\$18,500				Study effects of gas composition E-5 T-10 DS-\$6,150 ODC-\$1,000				Run small kinetic						
		d. Coal Characterization	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 tests and develop correlations to estimation of worth of coal for hydrogasification E-14 T-11 DS-\$10,200 ODC-\$500														
	PHASE II		a. Analysis of Experimental Data	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		Develop simple math model of process based on present data E-5 T-0 DS-\$2,300 ODC=0												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 preliminary reactor design process concept and hydrogasification E-6 T-0 DS-\$2,800 ODC=0																											
d. Economic Evaluation		Cost data review and extension E-7 T-0 DS-\$3,200 ODC=0		Develop simple flow sheet E-2 T-0 DS-\$400 ODC=0		Detailed flow sheet, energy and material balances E-3 T-0 DS-\$1,400 ODC=0		Process design of various sections E-4 T-0 DS-\$1,800 ODC=0		Cost estimate of equipment, plant and operating expenses E-3 T-0 DS-\$1,400 ODC=0		Report Preparation E-2 T-0 DS-\$900 ODC=0		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	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32
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	23,300												
Actual		10,000	15,000	23,300	14,500	23,000	21,100	19,600	28,900	23,400	30,200	24,500	22,900	25,800	21,900	28,900	27,800	22,300	23,400	22,500	25,000												
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	427,700	451,700												

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

Costs shown are costs to OCR or one-half of total costs.
DS - Direct Support
ODC - Overhead and Contingency

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GT HYDROGASIFICATION PROCESS

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

1966							1967												
Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	
2nd Year											3rd Year								
7											8								
Study hydrogasification of various coals, lignite, for most suitable conditions E - 23 1/2 T - 83 DS - \$27,200 ODC - \$7,000											Perform any support work necessary for phase III Double check data on which pilot plant is being designed								
3											4								
Investment properties of various coals E - 5 1/2 T - 27 1/2 DS - \$12,600 ODC - \$4,500											Produce material needed for any hydrogasification runs								
6											7								
Run small pilot unit and develop kinetic equations for design E - 10 T - 14 DS - \$21,500 ODC - \$5,250											Perform any support work necessary for phase III								
2																			
and develop correlations to enable worth of coal for hydrogasification E - 14 T - 11 DS - \$10,200 ODC - \$500											Laboratory and Pilot Plant Phase I-a, b, c, 7 II-a, 2 Engineers 24 Salaries \$40,400 Technicians 165 Other Direct Costs 11,400								
											2								
											Perform analysis of past data and new data in any areas necessary for phase III work								
Additional																			
1											2								
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								
8											9								
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								
1											2								
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. 14-01-0001-101. 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	23,700
400	22,300	25,000	32,600	25,000	28,600	27,200													
400	426,700	481,700	484,300	509,300	655,900	563,100													

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

2

IGT-MPP--8/66

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - August 1966

to

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

Hydrogasification

Three hydrogasification tests (Runs HT-128, HT-129, and HT-130) were conducted in the balanced-pressure pilot unit. In the first two of these tests the feed was a lightly pretreated Pittsburgh seam bituminous coal (Ireland mine); HT-130 used a lightly pretreated Ohio No. 6 seam bituminous coal (Broken Arrow mine) as feed. These coals were reacted in a 3-1/2-ft-deep fluidized bed with a hydrogen-steam feed gas mixture containing either 30 or 50 mole percent steam. Temperatures in the free-fall section above the coal bed were held at about 1300°F; the temperature at the bottom of the coal bed was controlled at 1700°F in Runs HT-129 and HT-130, at 1800°F in Run HT-128. Runs HT-128 and HT-130 were conducted at the standard reactor pressure of 1000 psig, Run HT-129 at 1500 psig. These tests are a part of the series investigating hydrogasification in a dual-temperature operation. Nominal coal feed rates were 74 lb/hr in the first two tests, and 53 lb/hr in Run HT-130. The hydrogen/coal ratio in all these tests was 25% of stoichiometric. All the tests were successful and of sufficient duration for evaluation of test conditions.

In Run HT-128, the coal bed was successfully maintained at 1800°F. The relatively high coal feed rate of 74 lb/hr and the reduced steam concentration of 30% in the feed gas helped in keeping this temperature level. The test lasted 5-3/4 hours, and was terminated when the coal feed supply was spent. Of the carbon in the coal, 37% was gasified. A product gas of 564 Btu/SCF (nitrogen-free basis) was produced.

Run HT-129, conducted at 1500 psig, is the first hydrogasification test made in the balanced-pressure pilot unit at a pressure higher than the standard 1000-psig level. The objective of the test was to see if improvements in carbon gasification and in the product gas heating value could be realized at pressures above 1000 psig. The run was ended after 5-3/4 hours when the coal feed supply was used up. Carbon gasification was about 37%. A product gas of 581 Btu/SCF (nitrogen-free basis) was produced.

Run HT-130 with Ohio No. 6 seam bituminous coal was conducted at the standard fluidized-bed dual-temperature run conditions. Earlier in the program this coal was hydrogasified in a moving coal bed at one temperature level (August 1965 Project Status Report). The objective of the test was to evaluate the per-

formance of the Ohio No. 6 seam coal at fluidized-bed conditions. The hydrogasification test was successful, lasting over 4 hours. The run had to be terminated somewhat short of the programmed 5-1/2 hours when a large leak developed in the connector fitting at the top of the reactor.

Coal Pretreatment

Nine pretreatment tests were made in the fluidized-bed pretreater to study minimum pretreatment conditions and to supply the hydrogasification units with nonagglomerating feed char.

Nominal conditions for the tests were:

Run No.	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Avg Bed Temp, °F
FP-78A	0.30	1251	21.0	775
FP-79	0.30	955	21.0	784
FP-80	0.24	1152	21.0	797
FP-81	0.26	1086	21.0	804
FP-82	0.25	1100	21.0	815
FP-82A	0.25	1100	21.0	815
FP-82B	0.25	1100	21.0	815
FP-82C	0.25	1128	21.0	816
FP-83	0.25	1077	21.0	815

Intended run conditions, but not attained.

Bituminous coal, crushed and screened to -16+80 mesh from Consolidation Coal's Ireland mine, was used as the feed for all nine runs. These runs were attempts to increase the capacity of the unit as well as to achieve stable runs with only air as the pretreatment gas. Considerable difficulty was met when trying to operate the unit above 800°F. Control of the reaction can be attained at 800°F with a 15-minute residence time. This appears at present to be the practical limit of pretreater capacity.

Methanation

A study of the methanation reaction catalyzed by Harshaw Ni-0104T nickel catalyst has revealed that, at higher CO concentrations (5-10%):

- Formation of $\text{Ni}(\text{CO})_4$ might occur significantly at temperatures above 550°F, and
- Diffusion of reactants and products into the porous catalyst might influence the observed rate of the methanation reaction.

A comparison will be made of the observed reaction rates for several catalyst particle sizes at higher temperatures (above 550°F), and in the 5-10% CO range.

The catalyst life test unit was used for a methanation run using iron-ammonia synthesis catalyst. The feed gas contained 12.3% CO, 29.5% H₂, 1.2% C₂H₆, and the balance CH₄ and inerts. Two steady-state periods were attained with space velocities of 824 and 226 SCF/cu ft catalyst-hr, 1000 psig, and a maximum bed temperature not exceeding 500°F. With the lower space velocity, 9.1% CO was removed, but heavy hydrocarbon content of the product gas did not exceed 3%. Of this, about 40% was composed of olefins. The heating value was low - about 840 Btu/SCF - due to the product containing about 4% CO₂ and 20% H₂. Similar results were attained with the higher space velocity, but with higher CO and lower C₂⁺ in the product gas. The successful increase in heating value of methanator product gas by use of iron catalyst depends greatly on the follow-up nickel catalyst reactor which must methanate the CO₂ and H₂ without hydrogenolyzing the C₂⁺ paraffins.

Coal Characterization

Connection shields of a better design were installed in the heat-of-reaction calorimeter. This modification reduces the heat loss to a minimum and permits much better reproducibility of experimental results. Work on Ireland mine coal is being continued.

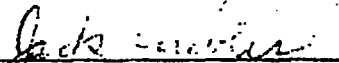
Engineering Economics Studies

A process design and cost estimate for a 250 billion Btu/day pipeline gas plant has been completed. This design differs from the state-of-the-art design in that the hydrogasification step is based on fluidized-bed pilot plant data. The hydrogen is supplied by the steam-iron process. Total capital investment is \$105,500,000 and price of gas, with \$4.00/ton coal, ranges from 50.2¢ to 54¢/million Btu, depending on by-product fuel credits.

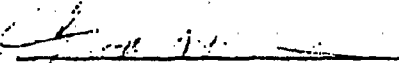
Bechtel Corp. and Foster Wheeler Corp. have submitted proposals, by invitation, for the preliminary design of the hydrogasification portion of the proposed pilot plant. These proposals are now being reviewed.

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

Approved



Signed



Jack Huebler, Associate Director

Frank Schora, Assistant Director

PROGRAM FOR DEVELOPMENT OF IGT F

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

		1964												1965															
		Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar								
		1st Year												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-10 T-27 DS-\$12,500 ODC-\$3,000				5 Study H ₂ O-CO-C System E-3 T-9 DS-\$3,800 ODC-\$1,000				6 Study particle size effect for both fixed and fluid-bed operator E-9 T-27 DS-\$11,300 ODC-\$2,000				7 Study hydrogasification under			
	b. Coal Pretreatment	1 Modify coal pretreatment unit E-3 T-11 DS-\$4,230 ODC-\$6,017				2 Study effects of pretreatment variables on char properties E-4 T-22 DS-\$10,400 ODC-\$4,000								3 Establish pretreatment properties of															
	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-\$6,150 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 Run 88											
	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 correlation estimation of worth of coal for hydrogasification E-14 T-11 DS-\$10,200 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-\$13,800 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 flowsheets updated E-20 T-0 DS-\$9,200 ODC-\$1,500																	
c. Reactor Design Studies	1 Make preliminary reactor design process concept and hydrogasification flowsheet E-14 T-0 DS-\$4,000 ODC-0																												
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		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	1 Make preliminary reactor design process concept and hydrogasification flowsheet E-14 T-0 DS-\$4,000 ODC-0																												

TOTAL		TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)																						
		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	23,300		
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	23,300		
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	28,800	27,800	22,300	23,400	22,300	25,000			
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	428,700	451,700			

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

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

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GT HYDROGASIFICATION PROCESS

11-381 A.G.A. PB-23a

1966							1967											
Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
2nd Year											3rd Year							

<p>Study hydrogasification of various coals, lignite, under most suitable conditions</p> <p>E=21 T=63 DS=\$27,300 ODC=\$7,000</p>	<p>Perform any support work necessary for phase III</p> <p>Double check data on which pilot plant is being designed</p>
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<p>Pre-treatment properties of various coals</p> <p>E=34 T=27 DS=\$12,400 ODC=\$4,300</p>	<p>Produce material needed for any hydrogasification runs</p>
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<p>Run small pilot unit and develop kinetic equations for design</p> <p>E=30 T=19 DS=\$13,500 ODC=\$5,250</p>	<p>Perform any support work necessary for phase III</p>
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<p>and develop correlations to enable of worth of coal for hydrogasification</p> <p>E=14 T=11 DS=\$10,200 ODC=\$500</p>

<p>Laboratory and Pilot Plant Phase I-a, b, c, 7 II-a, 2</p>	<p>Engineers 24 Technicians 105</p>	<p>Salaries 110,400 Other Direct Costs 13,400</p>
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	<p>Perform analysis of past data and new data in any areas necessary for phase III work</p>
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<p>additional</p>

<p>Make preliminary reactor design based on process concept and hydrogasification results</p> <p>E=6 T=0 DS=\$2,800 ODC=0</p>	<p>Work with subcontractor on design of reactors for hydrogasification and methanation</p> <p>E=4 T=0 DS=\$1,800 ODC=0</p>
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<p>Revision and refinement of capital and operating costs</p> <p>E=6 T=0 DS=\$2,800 ODC=0</p>	<p>Work with subcontractor on capital and operating costs for pilot plant</p> <p>E=9 T=0 DS=\$4,100 ODC=0</p>
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<p>Develop pilot plant flowsheet with heat and material balances</p> <p>E=1A T=0 DS=\$7,700 ODC=0</p> <p>Select architect-engineer-subcontractor</p>	<p>With assistance of subcontractor work on phase III as outlined in Contract No. 14-01-0001-381.</p> <p>E=34 T=0 DS=\$16,300 ODC=0</p> <p>\$85,500 available for architect-engineer subcontractor and engineering consultants.</p>
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23,300	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
15,400	22,300	25,000	32,600	25,000	26,600	27,200	22,900												
1,400	426,700	481,700	584,300	309,300	535,900	563,100	586,000												

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



367-MPR--9/66

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - September 1966

to

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

Hydrogasification

Six hydrogasification tests (Runs HT-131, HT-132, HT-133, HT-134, HT-135, and HT-136) were made in the balanced-pressure pilot unit. The first two were conducted with a lightly pretreated Ohio No. 6 seam bituminous coal (Broken Arrow mine). The coal was reacted in a fluidized bed with a hydrogen-steam feed gas mixture containing 30 or 50 mole percent steam. Temperatures in the free-fall section above the coal bed were held at about 1300°F; the temperature at the bottom of the coal bed was controlled at 1800°F in Run HT-131 and at 1700°F in Run HT-132. In the four other tests, an untreated but dried North Dakota lignite was reacted with a hydrogen-steam feed gas mixture containing 50 mole percent steam. In Runs HT-133, HT-134, and HT-136 the lignite was reacted in a fluidized bed; in Run HT-135 it was reacted in a moving bed. Hydrogen-to-lignite feed ratios were 25% of stoichiometric in all the tests except Run HT-132, in which it was 20% of stoichiometric. The two tests with Ohio No. 6 seam coal and the moving-bed test with lignite were completely successful. The three lignite tests conducted at fluidized-bed conditions were terminated before steady-state operation could be established due to plugs in the coal feed tube.

In Run HT-131, Ohio No. 6 seam coal, fed at a nominal rate of 75 lb/hr, was successfully gasified in a 2-ft-deep coal bed. Following 2 hours of steady-state operating, the bed depth was increased to 3-1/2 ft in order to determine the effect of coal bed depth on hydrogasification. One hour after the 3-1/2-ft coal bed was established the coal feed screw jammed when some of the coal agglomerated near the end of the coal feed tube. The operation with a 3-1/2-ft coal bed was too short for a reliable evaluation of the results at that depth.

In Run HT-132, Ohio No. 6 seam coal was gasified in a 3-1/2-ft coal bed when fed at a nominal rate of 66 lb/hr. A steady-state operating period of nearly 1-1/2 hours was obtained before the coal feed screw jammed, due mainly to product gases working up to the feed screw. Two openings in the coal feed tube insert were inadvertently left open allowing product gases to rise to the screw.

Runs HT-133 and HT-134 were the first two attempts to hydrogasify an untreated North Dakota lignite in the balanced-pressure reactor. The lignite reacted in a 3-1/2-ft-deep fluidized bed operated at 1700°F, with a temperature of about 1300°F

in the free-fall section above the solids bed. The feed rate was 53 lb/hr in Run HT-133, and 79 lb/hr in Run HT-134. The feed in both runs contained about 21% moisture. Both tests lasted a little over 2 hours. They were terminated when the feed screw jammed after a series of reactor pressure fluctuations and upsets.

The North Dakota lignite was successfully gasified in Run HT-135 by reacting it in a 3-1/2-ft moving bed with a hydrogen-steam mixture. Lignite and gas feed rates were reduced to about one-half of those in Run HT-134. The lignite feed rate was a nominal 40 lb/hr, and the hydrogen and steam rates were both 265 SCF/hr. The entire test lasted 7-1/4 hours. For the last 1-1/2 hours, the lignite and gas feed rates were doubled, and fluid-bed operation was established. The fluid-bed operating period was characterized by significant pressure fluctuations but these did not affect the screw feeder. The run was terminated when the lignite feed supply was used up.

The fluid-bed hydrogasification of lignite was again tried in Run HT-136. Conditions were similar to those of Run HT-134, except that the lignite feed rate was adjusted to 66 lb/hr to compensate for 5% moisture in the lignite. Within 3 minutes of the start of lignite feed the screw feeder jammed and could not be cleared. This extremely short feed period indicated that the feeder was already partially jammed before the screw was turned on. There were two interruptions during pressurization of the unit to 1000 psig when the pressure had to be reduced to atmospheric from about 50 psig to clear obstructions in the feed gas line. During these times lignite had been suddenly drawn from the feed hopper at a high rate into the coal feed tube where it bridged.

Coal Pretreatment

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

Nominal conditions for the tests were:

Run No.	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Avg Bed Temp, °F
FP-84*	0.25	1000	21.0	800
FP-84A	0.26	952	21.0	801
FP-85	0.43	649	17.3	714
FP-86	0.42	675	21.0	720
FP-87	0.30	904	21.0	778
FP-88	0.30	902	21.0	801
FP-89*	0.30	875	21.0	800
FP-89A	0.30	867	21.0	804

* Intended run conditions, but not attained.

Bituminous coal, crushed and screened to -16+80 mesh, was used as feed for six of the runs. Runs FP-84, FP-84A, and FP-85 used Pittsburgh No. 8 coal from Consolidation Coal's Ireland mine. The remaining runs used an Ohio No. 6 coal from Peabody Coal's Broken Arrow mine. Runs FP-85 and FP-86 used various, previously pretreated char as feed material.

These runs were made mainly to supply the hydrogasifier with nonagglomerating feed char, but different conditions for pretreatment are being investigated on the Ohio coal as time permits. The Ohio No. 6 coal at this time seems to be very similar to the Pittsburgh No. 8 coal in the amount of pretreatment necessary to render it free-flowing.

Methanation

A life study was made using a new lot of Harshaw Ni-0104T nickel catalyst to check variations in the manufacturer's samples. Three weeks of steady operation were achieved at low space velocity, with the highest bed temperature about 1025°C. Carbon monoxide was reduced to less than 0.1% in the product, but data on methanation of CO₂ were not obtained due to a deficiency of hydrogen in the feed gas. All the ethane and heavier hydrocarbons (3.4%) were converted to methane. The catalyst did not appear to deactivate. A similar test is planned for Girdler G-65 nickel catalyst.

Coal Characterization

The heats of reaction of hydrogen and coal were measured at 1300°F and 1000 psia. The coals and chars measured were Ireland mine raw coal, pretreated coal, low-temperature hydrogasifier residue, and high-temperature residue. The samples and results of the runs are being analyzed.

The heats of reactions of hydrogen and coals are now being measured at higher pressures and temperatures.

Engineering Economics Studies

The report on process design and cost estimate of a pipeline gas plant using the steam-iron process for coal gasification was distributed.

We have made some preliminary cost estimates of the cost of pipeline gas with hydrogen derived from a synthesis gas. This gas is generated by gasification of spent char using electrical resistance to provide the energy for gasification.

An invention was made this month pertaining to the operation of the hydrogasifier. In our present concept of the process, the hydrogen/carbon monoxide ratio of the gas going to the catalytic methanator is governed by the amount of steam de-

composed in the hydrogasifier and the amount of external hydrogen fed to the hydrogasifier. A shift reactor is avoided. Under certain conditions it may be difficult to control the H_2/CO ratio at 3.5 - an ideal ratio which enables conversion of essentially all the carbon monoxide and hydrogen to methane. In our present concept, should the ratio tend to be higher, CO_2 is methanated to remove the H_2 . This is somewhat undesirable because it wastes hydrogen. The invention involves the use of electrical heating either by resistance, arc, or induction methods to supply additional heat to the fluidized-bed portion of the hydrogasifier. By supplying this heat, additional steam will be decomposed, which lowers the H_2/CO ratio. This makes it unnecessary to methanate CO_2 to produce a high-Btu gas.

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

	1964												1965					
	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan
	1st Year												2nd Yr					
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-\$1,500 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-10 ^{1/2} T-27 DS-\$12,500 ODC-\$5,000			5 Study H ₂ O-CO-C System E-3 T-9 DS-\$1800 ODC-\$1000		6 Study particle size effect for both fixed- and fluid-bed operation E-9 T-27 DS-\$11,300 ODC-\$2,000			
b. Coal Pretreatment	1 Modify coal pretreatment unit E-3 T-11 DS-\$4,250 ODC-\$1,000			2 Study effects of pretreatment variables on char properties E-4 ^{1/2} T-22 ^{1/2} DS-\$10,400 ODC-\$4,000						3 Establish pretreatment unit 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-\$4,850 ODC-\$3,250			4 Study effects of gas composition E-5 T-10 DS-\$8,150 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 estimation of work					
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												2					
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																		
d. Economic Evaluation	1 Cost data review and extension E-7 T-0		2 Develop simple flow sheet E-2 T-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-\$1,400 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	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
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,
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	28,900	27,800	22,300	23,	
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,	

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

Costs shown are costs to OCR or

HYDROGASIFICATION PROCESS

A.G.A. PB-23a

1966												1967						
Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	
← 3rd Year →																		

Study hydrogasification of various coals, lignite, under most suitable conditions E-314 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
--	--

Properties of various coals E-54 T-274 DS-\$12,400 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
---	--

Correlations to enable capital for hydrogasification E-14 T-11 DS-\$10,200 ODC-\$500
--

Laboratory and Pilot Plant Phase I-a, b, c7 II-a2	Engineers- Technicians	24 103	Salaries Other Direct Costs	\$40,400 11,400
--	---------------------------	-----------	--------------------------------	--------------------

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

Preliminary reactor design based on 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	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
Select architect-engineer subcontractor	* \$85,500 available for architect-engineer subcontractor and engineering consultants.

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	23,700	
300	25,000	32,600	25,000	26,600	27,200	22,900	23,700													
700	481,700	484,300	508,300	539,900	563,100	586,000	608,700													

Total costs. DS - Direct Salaries, \$
 ODC - Other Direct Costs, \$



○ IGT-MPR -- 10/66

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report -- October 1966

to

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

Summary

1. Three hydrogasification runs were attempted. All three were terminated before steady-state operation began. There were mechanical problems with the feed system in two and a steam leak at the base of the reactor in the third. These difficulties have been corrected.
2. Because of gradual failure of thermocouples during 18 months of operation, temperature control was lost in the lower section of the tube, resulting in failure of the tube wall due to overheating. The 4-in.-diam reactor tube was replaced with a new tube equipped with a new set of wall thermocouples.
3. Six fluidized-bed coal pretreatment runs were attempted. Of these, five were successful. These tests were made on No. 5 Block coal from West Virginia to determine minimum pretreatment conditions, and to supply material for hydrogasification tests.
4. In the catalytic methanation program, the reaction rates for Girdler G-65 catalyst are being determined. This work is providing data for the preliminary pilot plant design.
5. The economic study report, "Evaluation of the Effect of By-Product Char Production on the Economics of Pipeline Gas Manufacture," was submitted. This study, which was based on the original state-of-the-art design, indicates that gas cost can be reduced by about 4 cents when by-product char amounts to 35-40% of process coal feed.
6. We are now cooperating with Bechtel Corporation to define the pilot plant flow sheet on which Bechtel's fixed price estimate for the preliminary design work will be based.

Hydrogasification

Three hydrogasification test start-ups were made in the balanced-pressure pilot unit (Runs HT-137, HT-137b, and HT-137c). The objective of these tests was to hydrogasify an untreated North Dakota lignite in a 3-1/2-ft-deep fluidized bed with a hydrogen-steam feed gas mixture containing 50 mole percent steam. Nominal lignite feed rates were 66 lb/hr, and the hydrogen/coal ratio was 25% of the stoichiometric ratio of 34 SCF hydrogen per lb lignite. The temperature in the free-fall section above the lignite bed was held at about 1300°F; the temperature at the bottom of the lignite bed was held near 1700°F. Two of these tests (Runs HT-137 and HT-137b) were terminated soon after start-up because of mechanical difficulties with the lignite screw feeder. In Run HT-137c, steady-state operation was established, but, because the lignite could not be discharged from the bottom of the bed, the test had to be terminated.

About 3 min after the start of lignite feed in Run HT-137, the feed screw jammed. Efforts to clear the screw by reversing, by reducing and stopping gas and steam feeds, and by depressurizing the reactor failed. After shutdown, we discovered that the pressure-equalizing rupture disk between the reactor and the reactor shell was ruptured. This suggested that the screw jammed because of a lignite plug in the feed tube caused by a pressure upset induced by the ruptured disk.

In Run HT-137b, the lignite feed screw jammed again after about 3 min of operation. No lignite plug was found after this test, which indicated that the feed screw jamming was essentially mechanical in nature. This was confirmed by a series of feed tests at atmospheric pressure in which the screw jamming during the tests was duplicated. The screw began to bind in the screw housing soon after the housing filled with solids. The binding appeared to be primarily in a section near the connection between the screw housing and the hopper. To correct this, the diameter of the flights in this section was reduced by 1/8 in. Also, to reduce the possibility of binding at other points, the front edges of the flights of the forward 18 in. of the screw were beveled. After these modifications, the lignite feed mechanism was tested again. No binding occurred during operation with a variety of feed rates.

In Run HT-137c, the lignite bed reached 3-1/2 ft, and the lignite, hydrogen, and steam rates were stabilized as steady-state operation was being established. However, lignite would not discharge from the bottom of the bed, so the test had to be shut down after 2 hr of operation. This difficulty was caused by a leak at the base of the hydrogen-steam feed tube. The leaking steam condensed in the discharge housing, wetting the lignite so it would not flow.

After Run HT-137c, we found that the feed gas tube, the thermocouples, and the gas sample probes were damaged by fusion of the metal at two points. At the lower point, corresponding to the bottom of the furnace, they were fused to the inside wall of the reactor tube. After the damaged components were removed, a pressure test revealed that the reactor tube had also been damaged, so it was also removed from the shell. There was one large (6 x 2 in.) hole and four smaller holes in the wall of the reactor tube. These were formed by fusion of the metal near the bottom of the furnace section. At a level corresponding to the top of the solids feed bed, there were about six small-diameter holes (1/8-1/4 in.). These holes resulted from attack of the metal from the inside of the tube. The fusion of the reactor tube and of the internal components was primarily caused by the loss of temperature control in the bottom zone of the reactor furnace which resulted from failure of the thermocouples in this zone.

A replacement 4-in.-pipe, Type 446 steel reactor tube, and 32 wall thermocouples have been installed. Reassembly of the pilot unit equipment components at the top and the bottom of the reactor is continuing.

Coal Pretreatment

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

Nominal conditions for the tests were:

<u>Run No.</u>	<u>Avg Solids Res Time, hr</u>	<u>Feed Gas Rate, SCF/hr</u>	<u>Oxygen Conc, %</u>	<u>Avg Bed Temp, °F</u>
FP-90	0.31	880	21.0	803
FP-91	0.32	974	21.0	775
FP-92	0.28	1114	21.0	800
FP-93 ^a	0.25	1200	21.0	800
FP-93 ^a	0.25	1181	21.0	801
FP-94	0.40	657	21.0	726

^a Run conditions intended, but not attained.

Bituminous coal crushed and screened to -16+80 mesh was used as feed for five of the runs. Run FP-94 was fed the char made in FP-91. The coal used was a high-volatile bituminous from the No. 5 Block of West Virginia. The coal was obtained from Union Carbide's Hanawha mine.

Runs FP-90 and FP-91 were made to determine the best reactor temperature for this coal. The best temperature again appears to be 800°F. Runs FP-92 and FP-93^a were made to determine

approximately the minimum residence time necessary at 800°F. This appears to be about 16-17 min. Run FP-94 was made to render FP-91's char free-flowing without considering minimum conditions.

The pretreater was also used as a fluidized-bed drier for North Dakota lignite that is being tested in the hydrogasifier. The lignite was predried in batch driers to 21% moisture, and then run through the pretreater at 220°F with hot air as the fluidizing gas. The moisture was reduced to 5%, making the lignite suitable for hydrogasifier feed.

Methanation

Catalyst deactivation in stirred-reactor tests in the past was probably caused by the small amounts of sulfur present in the feed gases. These gases are used for both stirred-reactor and life-study runs. Sulfur removal systems using a ZnO catalyst at 650°F have been installed and tests will begin next month.

Several stirred-reactor runs were made using the Girdler G-65 catalyst. This catalyst, which is less active than Harshaw Ni-OLO4T, would be more practical for the higher (5-13%) CO concentrations now expected in the methanator. The Harshaw catalyst would be better suited for final cleanup (less than 5% CO). Data indicate that the rate of CO methanation is roughly first-order in CO up to about 12% CO for the Girdler catalyst. Additional runs are necessary because of some sulfur poisoning.

Coal Characterization

The measurement of the heat reaction of hydrogen and coals is continuing. The heat capacity of chars is being measured for temperatures from 1000°-1500°F.


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

During the month we have been working with Bechtel Corp. to define the flow sheet for the hydrogasification plant. This flow sheet will be used by Bechtel in establishing its fixed price for the preliminary design work.

Approved



Signed



Jack Huebler, Associate Director Frank Schora, Assistant Director

PROGRAM FOR DEVELOPMENT OF IGT H

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

1984												1985																	
		Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar								
← 1st Year →												← 2nd Year →																	
PHASE I	a. Hydrogasification	1 Establish rate-controlling areas of operation E-13 T-36 DS-\$15,800 ODC-\$9,500				2 Study effect of degree of pretreatment E-6 T-19 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-\$3,000				5 Study H ₂ O-CO-C System E-3 T-9 DS-\$3,800 ODC-\$1,000				6 Study particle size effect for both fixed and fluid-bed operation E-9 T-27 DS-\$11,300 ODC-\$2,800				7 Study hydrogasification under			
	b. Coal Pretreatment	1 Modify coal pretreatment unit: E-3 T-11 DS-\$4,250 ODC-\$4,000				2 Study effects of pretreatment variables on char properties E-44 T-224 DS-\$10,400 ODC-\$4,000								3 Establish pretreatment operation: E-34 T-274 DS-\$12,400 ODC-\$4,900															
	c. Methanation	1 Review state of the art and design lab test unit E-7 T-0 DS-\$1,800 ODC-0				2 Build and shake down lab unit E-3 T-9 DS-\$4,150 ODC-\$3,300				3 Lab scale catalyst evaluation E-5 T-12 DS-\$4,850 ODC-\$1,250				4 Study effects of gas composition E-5 T-10 DS-\$6,150 ODC-\$1,800				5 Design and construct small pilot unit E-5 T-18 DS-\$8,500 ODC-\$28,500				6 Run							
	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-\$6,900 ODC-\$29,500												2 Establish tests and develop correct estimation of worth of coal for hydrogasification E-14 T-11 DS-\$19,200 ODC-\$150															
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	1 Make preliminary 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-\$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-8 DS-\$1,400 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-\$700 ODC-0				7 Revision and updating of flow sheet 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	25,400	30,200	24,500	22,900	25,900	21,900	28,900	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	258,600	282,100	304,000	330,800	358,700	381,000	404,400	428,700			

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

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

DROGASIFICATION PROCESS

PB:23a

1966								1967							
Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
3rd Year															
1 tion of various coals, lignite, suitable conditions E-23 T-63 DS-21,300 ODC-37,000				8 Perform any support work necessary for phase III Double check data on which pilot plant is being designed											
2 coals				4 Produce material needed for any hydrogasification runs											
3 Pilot unit and develop equations for design E-10 T-19 DS-311,900 ODC-37,250				7 Perform any support work necessary for phase III											
4 enable location				5 Laboratory and Pilot Plant Phase I-a8, b4, c7 II-a2 Engineers Technicians 24 105 Salaries Other Direct Costs \$40,400 21,400											
5 Perform analysis of past data and new data in any areas necessary for phase III work				2											
6 Design based on gasification results				2 Work with subcontractor on design of reactors for hydrogasification and methanation E-4 T-0 DS-21,800 ODC-0											
7 Selection and refinement of capital and operating costs				9 Work with subcontractor on capital and operating costs for pilot plant E-9 T-0 DS-34,100 ODC-0											
8 Develop pilot plant flowsheet with heat and material balances E-14 T-0 DS-37,700 ODC-0				2 With assistance of subcontractor work on phase III as outlined in Contract No. 14-01-0001-381. E-34 T-0 DS-116,300 ODC-0											
9 Select architect-engineer subcontractor				* \$85,500 available for architect-engineer subcontractor and engineering consultants.											
23,700	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
12,600	25,000	26,600	27,200	22,900	25,700	19,200									
34,300	308,500	335,900	363,100	386,000	409,700	428,900									

High Salaries, 5 Other Direct Costs, 5

2

IGT-MPR--12/66

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - December 1966

to

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

Summary

1. Seven hydrogasification tests were conducted during the month.
2. The test series for the evaluation of lignite hydrogasification was completed.
3. The test series for the evaluation of West Virginia No. 5 Block seam coal is nearing completion.
4. Five pretreatment runs with Illinois No. 6 coal were attempted to find suitable operating conditions for pretreatment of this coal. The fluidized bed operated successfully for only one run.
5. A life study of the G-65 methanation catalyst was terminated after 10 days because of carbon deposition, presumably due to the loss of temperature control in the catalyst bed.

Hydrogasification

Seven hydrogasification tests were conducted this month in the balanced-pressure pilot unit. Four of these tests (Runs HT-142, HT-143, HT-144, and HT-145) were conducted with North Dakota lignite from the Mercer County District. These tests concluded the basic test series for evaluating the fluid-bed hydrogasification performance of lignite in the pilot unit. The other three tests (Runs HT-146, HT-147, and HT-148) were conducted with a lightly pretreated West Virginia No. 5 Block seam bituminous coal. Last month, hydrogasification of this coal was first attempted in two unsuccessful tests. In both of these tests, the coal feed tube plugged before steady-state operation could be established.

With Run HT-142, the study of the hydrogasification of untreated lignite in a fluid bed was continued. Lignite, at a nominal feed rate of 93 lb/hr, was reacted in a 3-1/2-foot-deep fluidized bed. At these flow conditions the hydrogen/lignite ratio was 25% of the stoichiometric ratio, and the steam concentration in the hydrogen-steam feed gas was 30 mole percent. The reaction was conducted at 1000 psig with the lignite bed temperature controlled to a nominal 1700°F, while the free-fall zone above the bed was regulated to about 1300°F. The test had to be terminated after about 1 hour of lignite feeding when lignite agglomerated at the end of the coal feed tube and the screw feeder jammed. Agglomeration of the lignite appeared to have started in the 4-inch reactor tube near the coal feed tube. The probable cause of lignite agglomeration was the high heat release at the top of the reactor, which was occasioned by the high reactivity of the lignite, the high feed rate, and an initial high hydrogen/lignite ratio before the lignite feed rate was increased to 93 lb/hr.

Run HT-143 was conducted at conditions similar to Run HT-142. To minimize chances for agglomeration, the top 30 inches of the reactor tube were controlled to 1200°F, and, to allow a nearly

constant hydrogen/lignite ratio of 25% of stoichiometric to be maintained at all times, the hydrogen rate was increased in proportion to the lignite rate. Steady-state operation in this test was just being established after 2-1/2 hours of lignite feeding when the coal feed tube plugged as the lignite agglomerated at the end of the tube. Although steady-state operating data were not obtained in this test, the run did establish the validity of the modified start-up procedure for minimizing chances of lignite agglomeration.

In Run HT-144, the lignite was reacted with a hydrogen-steam mixture containing 50 mole percent steam. The hydrogen/lignite ratio was 20% of the stoichiometric. The nominal lignite feed rate was 20% of stoichiometric, the hydrogen feed rate was a nominal 530 SCF/hr, and the steam rate was 25.3 lb/hr. The reactor pressure, temperature, and lignite bed height were similar to those of Run HT-143. The test was completely successful. It was terminated after 5-1/2 hours when the lignite feed charge was used up.

Run HT-145 was conducted at conditions similar to those of Run HT-142. The features of this test were a hydrogen/lignite ratio that was 25% of stoichiometric and a steam concentration of 30 mole percent in the hydrogen-steam feed gas. The run lasted 5-1/4 hours with about 2 hours of steady-state operation. It was shut down when the feed supply was used up. This run concluded the current series of lignite hydrogasification studies in the pilot unit at fluid-bed conditions.

The three tests with pretreated West Virginia bituminous coal were conducted with a 3-1/2-foot fluidized coal bed at 1000 psig. The coal bed temperature was controlled to a nominal 1700°F, while the free-fall zone above the bed was regulated to about 1300°F. In Run HT-146, the coal was fed at a nominal rate of 53 lb/hr and reacted with a hydrogen-steam mixture containing 50 mole percent steam. The hydrogen/coal ratio was 25% of the stoichiometric ratio. The test was completely successful; none of the plugging difficulties exper-

perienced with this coal last month in the initial hydrogasification trials were encountered. The duration of the test was 6 hours.

In Run HT-147, the coal feed rate was a nominal 72.3 lb/hr. The hydrogen/coal ratio was 25% of the stoichiometric ratio, and the steam concentration in the hydrogen-steam feed gas was 30 mole percent. This test was also successful. It lasted 5 hours with 1-1/2 hours at steady-state operation.

In the third test with West Virginia coal, Run HT-148, the hydrogen/coal ratio was reduced to 20% of stoichiometric, while the steam concentration in the hydrogen-steam feed gas was adjusted to 50 mole percent. The coal feed rate was a nominal 64.5 lb/hr. This run was only partially successful as the coal feed screw jammed after 1/2 hour of steady-state operation. Agglomeration of the coal in the feed tube was responsible for the screw's jamming. Total coal feeding time was 3 hours.

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. The pretreater was also again used as a drier for lignite to be used in the hydrogasifier.

Table 1. NOMINAL CONDITIONS FOR THE PRETREATMENT TESTS

<u>Run No.</u>	<u>Avg Solids Res Time, hr</u>	<u>Feed Gas Rate, SCF/hr</u>	<u>Oxygen Conc, %</u>	<u>Avg Bed Temp, °F</u>
FP-101B	0.33	1283	21.0	798
FP-102*	0.30	1150	21.0	775
FP-102A*	0.30	1150	21.0	775
FP-102B*	0.30	1150	21.0	775
FP-102C*	0.30	1150	21.0	775

* 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 for all the pretreatment runs was from the Illinois No. 6 coal bed and was supplied

by Freeman Coal Mining's Crown mine. North Dakota lignite with 35% moisture was dried to 5% moisture using both air and nitrogen as the fluidizing gas. The drying temperature was 215°-230°F with a feed rate of 35-45 lb/hr of wet -10+80 mesh lignite. The pretreatment runs on Illinois coal were attempts to operate at maximum capacity and to overcome problems associated with the high amounts of tars in the scrubbing system. The scrubbing water in the last month has cooled somewhat and causes the tars to plug up the scrubber drain system and the liquid-level controller. This problem can be overcome by heating the scrubbing water to a temperature level similar to that of the summer months: 75°-80°F. Both steam injection and electrical heating of the water were tried, but neither has been completely successful as yet.

The four failures of Run FP-102 were caused by difficulty in sealing a new porous plate into the reactor. The problem was overcome by welding a support ring into the reactor bottom and using leaded gaskets on both the top and bottom of the plate to prevent feed gas from going around it.

Methanation

A life study was made using Girdler G-65 catalyst crushed to -10+20 mesh in the new reactor described last month. Space velocity was 3000 hr⁻¹, and the maximum bed temperature was about 1050°F. CO conversion declined with time on-stream and, after 10 days of running, the run was terminated. We believe the failure was due to the inability to control the feed gas temperature, which was about 835°F when the gas entered the reactor. The feed gas contained 12.5% CO, and carbon deposition occurred, thus fouling the catalyst.

Engineering Economics Studies

Further work has been necessary to complete the plant energy balance in the study of the manufacture of hydrogen for pipeline gas production by the gasification of coal char using

electric resistance to supply the heat for gasification. We are awaiting the costs of the electric generating equipment before completing our investment summary.

Calorimeter

The heat of reaction of the pretreatment of coal is completed for the range of 8-18% pretreatment at 700°F. Experiments are being conducted at 800°F for the same range of pretreatment.

No inventions were made this month under this contract.

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 A.1

		1964												1965								
		Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb		
		1st Year												2nd Year								
		1			2			3			4			5			6					
PHASE I	a. Hydrogasification	Establish rate-controlling areas of operation E-13 T-36 DS-\$15,800 ODC-\$5,500			Study effect of degree of pretreatment E-6 T-18 DS-\$7,500 ODC-\$2,500			Study effects of variables to establish kinetics of fixed-bed system E-13 T-36 DS-\$15,800 ODC-\$4,000			Study fluidization and establish rates for fluid-bed operation E-10 T-27 DS-\$12,500 ODC-\$3,000			MOO-DC System E-3 T-8 DS-\$3800 ODC-\$1000			Study particle size effect for both fixed and fluid-bed operation E-9 T-27 DS-\$11,300 ODC-\$2,000			Study hydro...		
	b. Coal Pretreatment	Modify coal pretreatment unit E-3 T-11 DS-\$4,250 ODC-\$4,000			Study effects of pretreatment variables on char properties E-4 T-22 DS-\$10,400 ODC-\$4,000									Establish pretreatment properties E-5 T-27 DS-\$12,650 ODC-\$4,500								
c. Methanation	c. Methanation	Review state of the art, and design lab test unit E-7 T-0 DS-\$3,800 ODC-0			Build and shake down lab unit E-3 T-9 DS-\$4,150 ODC-\$3,500			Lab scale catalyst evaluation E-5 T-12 DS-\$6,850 ODC-\$1,250			Study effects of gas composition E-5 T-10 DS-\$6,150 ODC-\$1,600			Design and construct small pilot unit E-5 T-18 DS-\$8,500 ODC-\$18,500								
	d. Coal Characterization	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 tests and develop correlation estimation of worth of coal for E-14 T-11 DS-\$10,200 ODC-\$500											
PHASE II	a. Analysis of Experimental Data	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	Develop simple math model of process based on present data E-5 T-0 DS-\$2,300 ODC-0			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-\$9,280 ODC-\$1,500																	
c. Reactor Design Studies	Make preliminary process concept																					
PHASE III	d. Economic Evaluation	Cost data review and extension E-7 T-0 DS-\$3,200 ODC-0		Develop simple flow sheet E-2 T-0 DS-\$900 ODC-0		Detailed flow sheet, energy and material balances E-3 T-0 DS-\$1,400 ODC-0		Process design of various sections E-4 T-0 DS-\$1,800 ODC-0		Cost estimate of equipment, plant and operating expenses E-3 T-0 DS-\$1,400 ODC-0		Report Preparation E-2 T-0 DS-\$900 ODC-0		Revision and updating of flow sheet E-6 T-0 DS-\$2,800 ODC-0								
	a. Preliminary Pilot Plant Design																					

TOTAL		TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE)																		
		23,500	23,500	23,500	23,800	22,400	24,100	24,300	24,700	23,700	23,300	23,800	30,300	29,500	27,800	27,400	28,100	28,100	23,300	23,300
Planned	\$55,400																			
Actual		10,000	15,000	23,300	14,500	23,000	21,100	19,800	28,900	25,400	30,200	24,500	22,900	28,500	21,900	28,900	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

HYDROGASIFICATION PROCESS

A.G.A. PB-23a

1966							1967										
Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
7							8										
Study hydrogasification of various coals, lignite, under most suitable conditions E-21½ T-43 DS-\$27,200 ODC-\$7,000							Perform any support work necessary for phase III Double check data on which pilot plant is being designed										
Correlations of various costs E-5½ T-27½ DS-\$12,400 ODC-\$3,500							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										
Develop correlations to enable design for hydrogasification E-14 T-11 DS-\$10,200 ODC-\$500							Laboratory and Pilot Plant Phase I-a8, b4, c7 II-a2 Engineers Technicians 24 105 Salaries Other Direct Costs \$40,400 11,400										
							Perform analysis of past data and new data in any areas necessary for phase III work										
Preliminary reactor design based on 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							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										
Select architect-engineer subcontractor							* \$85,500 available for architect-engineer subcontractor and engineering consultants.										
3,300	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
3,300	25,000	32,600	25,000	26,600	27,200	22,900	23,700	19,200	21,800	21,400							
20,700	481,700	484,300	509,300	633,900	563,100	686,000	609,700	628,900	650,700	672,100							

Total costs. DS = Direct Salaries, \$
 ODC = Other Direct Costs, \$



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