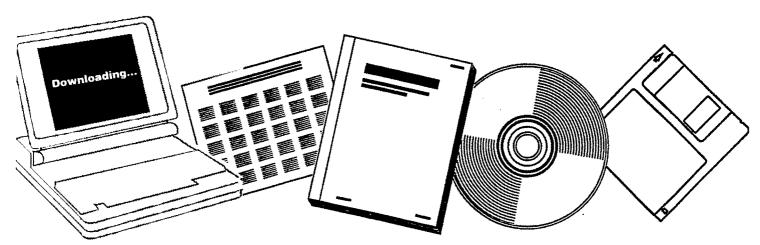




DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS. MONTHLY PROGRESS REPORTS, JANUARY--DECEMBER 1965

INSTITUTE OF GAS TECHNOLOGY, CHICAGO, ILL

1965



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DEVELOPMENT OF 1GT HYDROGASIFICATION PROCESS

Monthly Progress Reports for the Period January - December 1965

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institute of Gds Technology 111 Center Chicago, illinois 60615

Prepared for

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

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



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CONTENTS

Monthly Progress Reports for each month January through December 1965

This report was prepared as an account of work abounced by the United States Government. Neither the United States for the United States Elergy Research and Development Administration, flor any of their contractors, subcontractors, or their employees, makes any warranty, express of implied, or assumes any light liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - January 1965

to

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

Hydrogasification

Rebuilding of the hydrogasification reactor heating furnace was continued. Three of the furnace heating elements have been installed in the reactor shell. Installation of the remaining four heating elements, received from an outside fabricator at the end of the month, was resumed immediately.

To complete the hydrogasification pilot unit modifications outlined in the November 1964 Work Report, fabrication and construction work was continued.

Fabrication of the major assembly components for the elimination of the reduction in cross-section at the reactor bottom and for the screw-feed discharger has been nearly completed. A 4-in.-diameter expansion bellows, to match the diameter of the new reactor tube, has been received from the fabricator. Installation of a coal residue receiver with a larger capacity than the original receiver has been started.

Instrumentation and control valving of the pressure-balancing system between the reactor tube and the outer shell have been installed. Electrical wiring of the system was begun.

Coal Pretreatment

Five coal pretreatment tests were conducted in the continuous-flow fluid pretreater. The purpose of these tests is to define minimum pretreatment conditions for producing nonagglomerating char for the hydrogasifier.

Nominal conditions for these tests are given below:

Run No.	Solids Rate, lb/hr	Avg Solids Residence Time, hr	Gas Rate, SCF/hr	Oxygen,%	Temp, ^C F
7 8	131 86 71	0.45 0.43	800 834	4 21	755 680
9 10 11	50 49	0.67 1.14 0.89	599 603 603	2 <u>1</u> 21	305 700 765

INSTITUTE OF GAS TECHNOLOGY

Oxygen concentrations were increased to 21 percent (air) when it was found that treatment with 4 percent oxygen did not produce a nonagglomerating char. Because the increase in oxygen concentration alone was not sufficient for making the coal nonagglomerating, the coal residence time was increased to about 1 hour. Imboratory tests of the char at conditions simulating those in the hydrogasifier showed the char of Run 10 to be nonagglomerating. Test results of the char produced in Run 11 are not yet available.

Methanation

A meeting was held with Dr. Carberry, consultant on this work, to complete the design details for the reactor and to begin planning the test program. The major pieces of equipment have been designed for the laboratory-scale methanation studies. Suppliers were selected for the purchase of the major pieces of equipment. Designs of the barricading and other facilities, which will be required before the equipment can be operated in the new building, was completed.

Coal Characterization

Dynatech Corp. completed prints of the equipment and instrumentation for the high-temperature, high-pressure heat-of-reaction calorimeter and submitted them for revision and approval. Plans were made for a meeting with them to finalize the design details. The barricades for this unit, which will be located in the new building, were designed.

Work on establishment of a coal petrographic laboratory was continued. United States Steel applied Research Laboratories were visited to obtain information on their methods and apparatus. Delivery of the microscope was completed except for two objectives. Training in maceral recognition and point count analysis was started, and work on mounting and polishing coal (Pittsburgh No. 8 seam and Ohio No. 6 seam) and coal pretreatment products was continued. Shop fabrication of atparatus parts for the reflectance determination was nearly completed. Work on this aspect will be continued when the reflectance standards and test samples are received from Bituminous Coal Research, Inc.

Process Concept Development

The computer model for catalytic cleams methanation was used to develop data to establish probable best areas of operation. It was decided to incorporate into the model tests for probable carbon deposition on the catalyst based on system conditions. This model will enable a better definition of the best conditions for achieving operability of the catalyst.

Economic Evaluation

Work was continued on selecting favorable conditions for the state-of-the-art design. Pipeline gas compositions have been determined for the 60 percent total carbon conversion cases. This series of cases also shows an increase in hydrogen requirements with an increase in the temperature of the high-temperature zone.

The bare cost of the hydrogen, oxygen, and methanation sections has been completed. Cost of the hydrogen and oxygen sections increased with an increase in temperature and total carbon conversion. There is no real cost trend for the methanation section, since several of the gases had to be processed differently to obtain the desired product gas.

Process materials for the gasifier and hydrogasifier have been determined. The coal and steam for the hydrogasifier decrease as the temperature and total carbon conversion increase, but all other materials increase.

No inventions were made during the month of November on this project. All equipment deliveries for the reactor furnace were back on schedule. Heating element wire, delayed aboard an inbound freighter from Sweden by the dock strike, was replaced by a second shipment which was flown in.

Jack Frehler, Associate Director

Frank Schora, Manager

PROGRAM FOR DEVELOPMENT OF IGT

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DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - February 1965

to

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

<u>Hydrogasification</u>

The hydrogasification reactor heating furnace rebuilding was completed with the installation of the remaining four heating elements. All furnace sections were tested for operability and were found to perform normally.

A new 4-in.-diameter reactor tube of type 446 alloy was installed in the furnace, and the ends of the tube welded to the top and bottom reactor shell closures. Reactor tube-wall thermocouples were drawn through the shell wall, sealed in glands, and connected to the appropriate temperature recorder.

Electrical wiring of the differential pressure control system was completed. While failure of the control system is unlikely, rupture disks were installed in the pressure-equalizing line between the reactor tube and the shell as backup protection.

A larger capacity char residue receiver (7.17 cu ft instead of the original 5.1 cu ft) was installed. To discharge char from the reactor into the receiver at smoother and higher rates, a screw discharger was installed in place of the previously used star valve. The coal feed hopper and the coal feeder were reassembled to the top of the reactor. Piping of the hydrogen feed line and steam feed line to the bottom of the reactor was started.

Coal Pretreatment

Four coal pretreatment tests were conducted in the continuous-flow fluid pretreater using Pittsburgh seam bituminous coal from the Ireland mine. These tests are a part of the program for defining minimum pretreatment conditions required to produce a nonagglomerating char for the hydrogasitier.

Nominal conditions for the tests are as follows:

Run No.	Solids Rate, 1b/hr	Avg Solids Residence Time, hr	Gas Rate, SCF/hr	Oxygen Æ	Temp, oF
12	72	1.27	613	2.1	440
13	73	1.00	602	2.2	536 666
13 14	78	1.0	590	9.8	
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Pretreatment gas in these tests was a mixture of nitrogen and air. It was found that with 2% oxygen concentration in the gas, sufficiently high bed temperatures could not be maintained for adequate coal pretreatment. Earlier tests showed that the temperature necessary for pretreatment could be maintained with 21% oxygen (air). To better define oxygen requirements, further tests were conducted with a 10% oxygen concentration. It appeared that adequate temperatures were maintained with this concentration. Imboratory evaluations of the chars of these tests for agglomerating characteristics have not yet been completed.

Methanation

Planning of the test program continued. Design of minor pieces of equipment began. Quotations were obtained for most of the major equipment and instrumentation, and orders were placed for some major pieces of equipment. The materials for the barricading were ordered and received, and construction was begun. Drawings of part of the equipment to be fabricated by IGT were submitted to the machine shop.

Service facilities for the new laboratory area were selected, and their design begun.

Coal Characterization

A meeting was held with Dynatech Corp. to discuss revisions made in the design of the high-temperature, high-pressure heat-of-reaction calorimeter. As mentioned in the Methanation section above, service facilities for the new laboratory area, to be shared by the calorimeter and the methanation equipment, were selected and design was begun.

A review of the literature on coal petrography to establish how it may be applied to coal hydrogasification has largely been completed. A report on this topic has been prepared by Dr. G. H. Cady, consultant on the project.

Methods for determining true density and apparent particle density for coal and chars have been reviewed. This determination is desired for developing reaction kinetics as applied to the period of free-fall. Apparatus and procedures for these determinations will be set up next month.

Reflectance standards and reflectance test sample were received from Bituminous Coal Research, Inc. Our apparatus for this determination will be checked out next month.

Process Concept Development

Work continued during the month on the mathematical model of the cleanup methanation step. An analysis of various operating conditions for the methanator was conducted to investigate potential carbon-depositing side reactions in order to indicate areas of best probable operation from this standpoint.

Economic Evaluation

The total bare costs have been calculated for all the cases being considered for the state-of-the-art design. They vary from \$108,450,000 to \$124,308,000. Although there are many conflicting cost trends, the overall total base cost does vary slightly in a particular pattern.

The cost of the total coal required for all the cases under consideration has been determined. Again, there are conflicting trends in total coal usage. As the coal requirement for the hydrogasifier decreases, the coal required for both hydrogen production and power generation increase. The annual cost varies from \$23,495,000 to \$28,526,500. If char byproduct credit is taken into account, however, the cost varies only from \$22,754,200 to \$24,631,000. Taking coal byproduct credit into account, there is a slight increase in coal cost with an increase in carbon conversion.

All this data will be presented in the February Work Report. Since there is no overall trend in plant cost, the conditions for the state-of-the-art design cannot be established until gas production costs have been calculated.

No inventions were made during February on this project. A complete review of patents in this area of technology is underway so that patentable ideas will be more readily identified.

Jack Huebler, Associate Director

Frank C. Schora, Manager

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Project Work Report

Institute of Gas Technology • 11 T Center • Chicago 60616

Affiliated with Illinois Institute of Technology

Project No. PB-23a OCR Contract No.

February 1965

to American Gas Association - Office of Coal Research

On Pipeline Gas From Coal - Hydrogenation (IGT Hydrogasification Process)

> COAL CHARACTERIZATION H. L. Feldkirchner D. M. Mason ENGINEERING ECONOMICS STUDIES

C. L. Tsaros, Supervisor S. J. Knabel A. T. Talwalkar

HIGH-PRESSURE METHANATION

H. L. Feldkirchner

PILOT PLANT STUDIES

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ABSTRACT*

Engineering Economics

The total bare plant costs and gas production costs have been calculated for the cases being considered for the state-of-the-art design study. The total bare plant costs for a 250 MMCF/day plant vary from \$117,126,000 to \$133,678,000. Although there are many conflicting cost trends, the overall total bare cost tends to vary slightly in a particular pattern. Gas production costs vary from \$0.59 to \$0.66/MMBtu.

Total coal requirements for the cases under consideration have been determined. Again there are many conflicting trends in total coal usage. The annual cost for coal based on a \$5/ton price varies from \$23,725,000 to \$28,526,000. Assuming that byproduct credit, the coal cost varies from \$23,397,000 to \$25,606,000, there being only a slight increase in coal usage with an increase in carbon conversion.

Except for coal pretreatment, the process design will follow the form outlined in Fig. 1. A total carbon conversion of approximately 50% will be required in the hydrogasifier to avoid the production of excess char. An operating temperature of 1700°F will be used in the high-temperature zone. Ten % conversion will be used in the low-temperature zone in the state-of-the-art design.

Pilot Plant Studies

Reconstruction of the hydrogasification pilot unit reactor furnace was completed. A new 4-in.-diameter reactor tube with attached thermocouples was installed.

Electrical wiring of the new reactor-shell differentialpressure control system was completed. Rupture disks were installed in a pressure-equalizing line between the reactor tube and the shell to protect the tube from overpressurization in the event of failure of the differential-pressure control system.

A larger capacity residue receiver and a residence discharge screw assembly were installed. The coal feed hopper and the coal screw feeder were reassembled.

Four coal pretreatment tests were conducted in the continuous-flow fluid pretreater as part of the program for developing minimum pretreatment conditions.

^{*} Abstracts are omitted for those sections whose reports are brief.

WORK IN PROSPECT

Coal Characterization

Calorimetry

Dynatech Corp. will complete the revision of the design of the calorimeter. The new barricading will be completed.

Coal Petrography

Our equipment for determination of reflectance will be checked out. Apparatus and procedures for determination of true and apparent particle density will be set up.

Engineering Economics

Work will begin on the detail process design for the state-of-the-art design.

<u>High-Pressure Methanation</u>

Selection of equipment will be completed and the instrument panels will be fabricated. The new barricading will be completed.

Pilot Plant Studies

The hydrogasification pilot unit will be pressure tested and shakedown operations will be conducted. Hydrogasification tests will be resumed in the pilot unit with hydrogensteam mixtures.

Coal pretreatment tests will be continued in the fluid pretreater to define optimum pretreatment conditions and to produce feed char for the hydrogasification pilot unit.

Coal Characterization

Calorimetry

Dynatech Corp. continued modification of the original calorimeter design and orders were placed for all major pieces of instrumentation.

The materials for barricading were ordered and some were delivered; construction of the barricading was initiated.

Coal Petrography

Reflectance standards and a test sample were received from Bituminous Coal Research, Inc.

True Density and Apparent Particle Density of Coal and Char

Knowledge of these properties of the hydrogasification feed material is required for calculation of free-fall residence times occurring in the experimental gasification studies. Methods of determination are reviewed here. This review was made in preparation for selecting procedures to be used in our determinations.

To determine true density, the displacement of a fluid that will fill all the pores of the substance must be measured. Helium, water, methyl, alcohol, and other fluids have been used for this purpose from time to time. Because the helium molecule is smaller than most other molecules and thus can more easily penetrate pores, and because it has little tendency to be adsorbed, its determination is generally considered to give the true density. However, several investigators that helium is adsorbed at room temperature by high-density carbons in amounts sufficient to cause appreciable error in the density. The effect is attributed to the electrical conductivity of graphitized carbons; it has been shown to be appreciable for carbons with densities of 1.9 g/cc and greater, and to be negligible for a number of carbons with densities less than 1.54 g/cc. Determination at elevated temperature is recommended for high-density carbons. It appears, however, that little error should be encountered from this source in measurements at room temperature on hydrogasification feeds and residues, whose densities are not expected to go much above 1.60 g/cc.

Helium densities can be determined with the Beckman air comparison pycnometer, already available in the Institute's analytical laboratories. Some attention will have to be given to procedures for drying and evacuation of the sample.

Particle density of 100 mesh and coarser can be determined by displacement of mercury. The high surface tension of mercury prevents penetration of small pores. At atmos-

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pheric pressure, pores down to about 14-micron diameter are penetrated. However, the same effect restricts the filling of small volumes formed about points of contact of the particles. For a given volume of solids, the number of these contact points increases rapidly as the average particle size decreases. Thus the pressure of mercury needed to decrease the sum of these contact volumes to a negligible amount increases as the particle size decreases. According to Cartan and Curtis, pressures greater than atmospheric are required for particles smaller than 20 to 40 mesh, while with a pressure of 100 psi the density of 100-mesh particles (of alumina) have been measured successfully. At this pressure, pores of about 2-micron diameter are penetrated. Higher pressures are probably not feasible with our samples because coals and residues are likely to have appreciable porosity in this size range (<2\mu).

Particle density of finer particles can be determined and results from the mercury displacement method on coarse particles checked by means of Ergun's gas flow method. This method is based on measurements of the pressure drop vs. air flow across beds of the powder packed to several different bulk densities. The apparatus is not difficult to construct or run, but requires about 100 grams of a sieve fraction as sample.

REFERENCES CITED

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Engineering Economics Studies

Survey of Pibeline Gas Plant Economics

In order to select a favorable basis for making the stateof-the-art design of a plant that will make pipeline gas from coal,
a preliminary economic survey of operating variables has been completed. The design under study will use a hydrogen-steam feed to
an autothermic hydrogasifier. The study of the effect of total
carbon conversion and temperature level in the hydrogasifier on
pipeline gas costs was made practical by the availability of a
computer program to calculate effluent gas compositions for a
large number of cases. Because of the nature of this survey, detailed designs were not made for each case. Instead, unit costs,
as a function of capacity, were used to determine investment for
a number of sections of the plant such as coal storage, grinding,
gas prepurification, oxygen production, hydrogen production, and
utilities. These unit costs were derived from a previous study of
coal hydrogasification using hydrogen rather than a hydrogen-steam
mixture.

Design Basis

This study is based on the process shown in Fig. 1. It was assumed that pretreatment of the coal fed to the hydrogasifier was not required. In a few cases, a portion of the carbon monoxide had to be shifted because the hydrogen concentration was too low for methanation.

In this study, the cost of the coal was assumed to be \$5/ton. The char credit was based on the equivalent cost of heat from coal at \$5/ton. The gas prices were calculated according to the A.G.A. accounting procedure.

Coal Storage

This section consists of equipment for receiving, storing, and reclaiming incoming coal. No detailed process design was undertaken on this section. The cost was determined from costs from the previous study.

Coal Preparation

In this study, the only preparation considered was grinding of the coal feed to the gasifier and hydrogasifier. Actual pretreatment of the hydrogasifier feed coal was not considered as the study was comparative in nature and free-fall devolitilization was assumed. The coal burned as fuel for steam generation was assumed usable as received, without grinding. No treatment or further grinding of the hydrogasifier char fed to the gasifier, or used as fuel, was considered. The bare cost was estimated from the costs determined in the earlier hydrogasification study.

Hydrogasification

This section consists of feed hoppers, hydrogasifiers, residue hoppers, vent-gas surge drums, vent-gas compressors, and hydrogen steam preheat furnaces. Hoppers and vent-gas equipment were sized directly on the basis of volume of solids handled. Hydrogasifiers were all the same size, with the design consisting of a coal preheat section, free-fall devolatilization zone, and a high-temperature reaction zone. The number of hydrogasifiers was set by the allowable solids throughput in the high-temperature reaction zone. There is no pilot plant data available which permits an estimate of the effect of temperature on solids conversion rate. However, data obtained from H. F. Feldkirchner's tests in a semiflow reactor showed that a higher reaction rate will occur as the gasification temperature rises.2 This correlation showed that specific carbon gasification rate is a function of temperature, percent of carbon gasified, and percent of steam in the feed stream. At the design hydrogasifier conditions used in the current study, the above correlation showed that carbon conversion rates for the 1900° and 2000°F cases are severalfold higher than for the 1700° and 1800°F cases. However, it was found that at hydrogasifier conditions in our study, each pair of temperatures represented similar rate levels.

Rates based on semiflow data do not account for effects of mass transfer that would occur in a countercurrent reactor. These effects would reduce the rate of conversion; to account for them and still show the relative rate advantage of 1900° and 2000°F temperatures in the high-temperature zone, residence times for the 1700° and 1800°F cases are based on 0.5 hour for 50% carbon conversion, with relative rates for 40% and 60% conversions being adjusted in accordance with the semiflow data correlations. For 1900° and 2000°F operation, the rates at corresponding conversion levels were doubled, thus halving the number of hydrogasifiers.

Hydrogasifier calculations were based on steam and process hydrogen entering the reactor at 1200°F. Heat was assumed to be recovered from spent char, with the difference between required and recovered heat being obtained in a separate furnace. The cost of this unit was based on heat transfer duty. Table 1 shows the composition of the hydrogasifier effluent and the pipeline gas.

Gas Prepurification

N S t I T U T E

This section consists of hot carbonate scrubbing and adsorption by activated carbon and iron oxide. Table 2 shows the operating conditions for the hot carbonate process. The target CO₂ concentration remaining in the gas was 2% with variation from 1%-3% depending upon the methanation equilibrium and pipeline gas heating value. The cost for the hot carbonate process was estimated from an article by H. E. Benson. The cost for the activated carbon and iron oxide boxes was calculated from the earlier study. Since the total gas flow rate did not vary significantly in this study, the costs of these items were

considered constant.

<u>Methanation</u>

The method of estimating the cost of the methanation section was described in the January 1965 Work Report. The methanation reactor was sized by equilibrium correlations obtained from the Girdler Company. These correlations are based on data obtained from low-pressure gases with a small hydrogen concentration. The heat of reaction is assumed to be removed by cooling a portion of the effluent and recycling it back to the inlet of the reactor. The cost of the individual components of the section was estimated to obtain a bare cost.

Oxygen Production

This section consists of a complete oxygen plant; the cost for it was based on 1600-ton/day plants. Linde's largest plant to date used 1250 tons/day. Their representative indicated that a 1600-ton/day unit would be reasonable by the 1970's when this plant would be built. In this study, two 1600-ton/day plants were used along with a smaller unit. (For one case, however, three 1600-ton/day plants were used.)

The oxygen costs were determined for two cases according to the A.G.A. accounting procedure. The oxygen plant contributes \$0.06-\$0.10/MBtu to the gas price. The total cost for oxygen is about \$5.70/ton. Approximately 650 hp-hr/ton of oxygen is required.

The bare cost for this section was calculated from Fig. 3 in the January-February 1964 Work Report.

Hydrogen Section

This section will produce the hydrogen to be used in the hydrogasifier. The system used in the January-February 1964 Work Report was also used in this study. Table 3 shows the process data for the hydrogen plant; the bare cost was estimated from Fig. 1 of the January 1965 Work Report.

The cost for the hydrogen produced by this process is about \$0.27/MCF. The cost of hydrogen (including $O_{\rm e}$) constitutes 40%-60% of the gas price.

Offsite Facilities

This section consists of a steam generation plant, an electrical generation plant, a water treatment plant, and cooling water facilities.

Table 4 shows the various utilities required in each

of the sections. The cost estimate for this section was based on the unit costs used in the January-February 1964 Report.

Conclusions

Table 5 shows the materials required for the plant. Figs. 2-4 and Table 6 show the bare cost for the entire plant. The total bare cost increased as the total carbon conversion increased from 40% to 60%. With regard to temperature, the bare cost reached a maximum at 1800°F.

The gas prices are shown as a function of total carbon conversion in Fig. 5. The gas price increases as the total carbon conversion increases from 40% to 60%. Since there was considerable excess char in the 40% cases, no calculations were made to try to obtain an optimum below 40%. At the present time, it is planned to base the state-of-the-art design on a system with little or no excess char.

The 10% carbon conversion in the low-temperature zone is more attractive. In the computer program used for calculating the hydrogasifier operation, the assumption was made that there was no heat produced in this zone. Therefore, not as much steam decomposed so more external hydrogen was required.

At present, 1700°F appears to be the most economical operating temperature. If the system is to operate with little or no excess char, the total carbon conversion would have to be about 50%. If the excess char is not objectionable, the 40% case would be more economical.

PEFERENCES CITED

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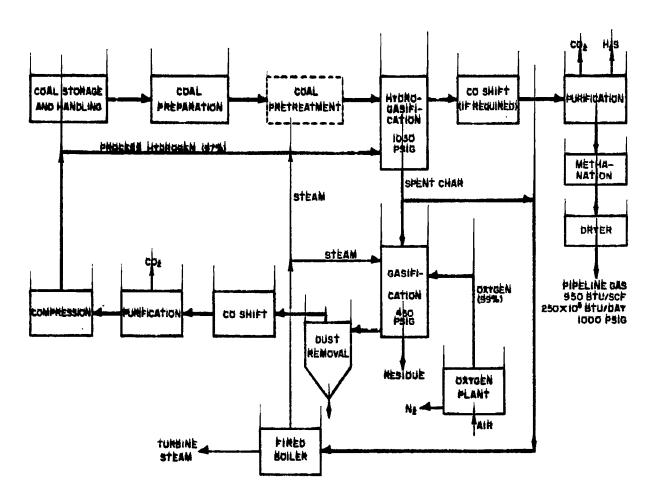


Fig. 1.-PIPELINE GAS BY HYDROGASIFICATION OF COAL

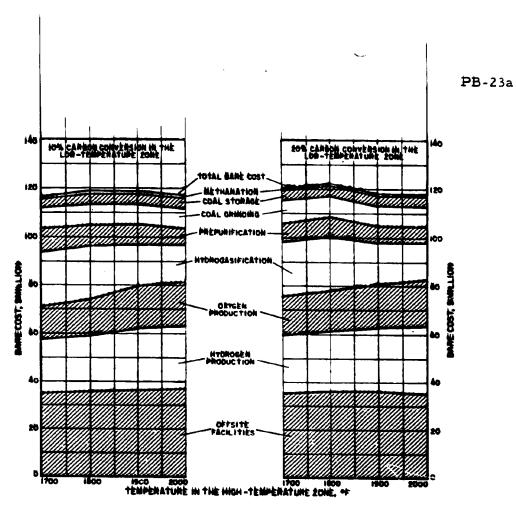


Fig 2.-BARE COST VS TEMPERATURE IN THE HIGH-TEMPERATURE ZONE FOR 40% TOTAL CARBON CONVERSION

-11-

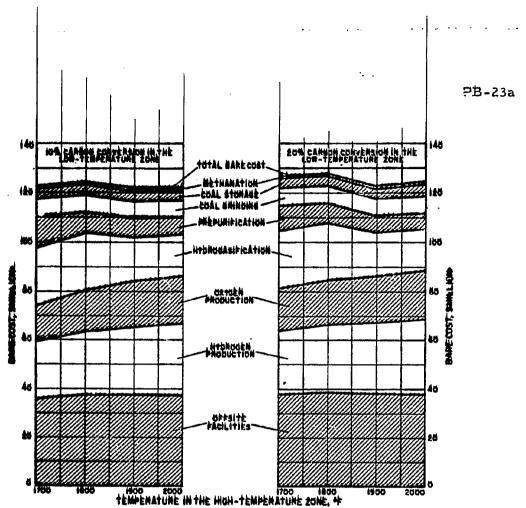


Fig. 3.-BARE COST VS. TEMPERATURE IN THE HIGH-TEMPERATURE ZONE FOR 50% TOTAL CARBON CONVERSION

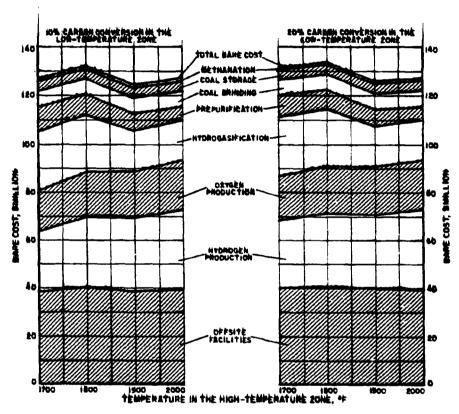


Fig. 4.-BARE COST VS TEMPERATURE IN THE HIGH-TEMPERATURE ZONE FOR 60% TOTAL CARBON CONVERSION

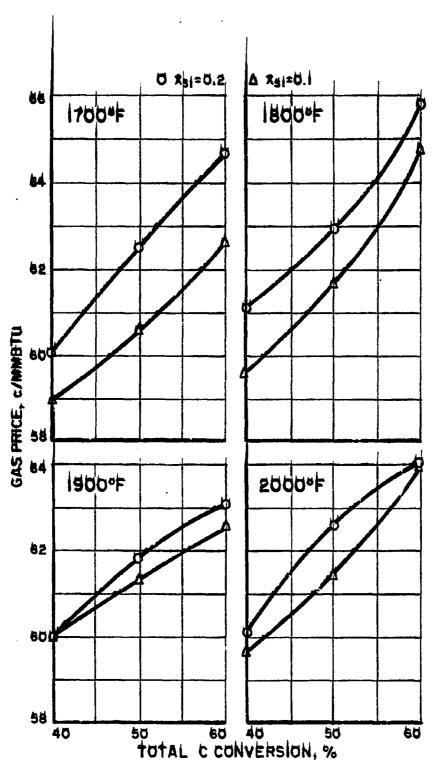


Fig. 5.-CORRELATION OF GAS PRICE WITH TOTAL CARBON CONVERSION AT VARIOUS TEMPERATURES (Temperatures Given Refer to High-Temp Zones)

Table 1.-SUMMARY OF HYDROGASIFIER OPERATION WITH STEAM-HYDROGEN FEEDS AND CALCULATED PIPELINE GAS COMPOSITIONS (Quantities Based on 100-Lb Coal Feed)

Temperature in High-Temperature Zone, OF				1700			_			1800		
Fraction Feed Carbon Converted in Low-Temperature Zone	0.1	0.2	0.1	0.2	0.1	0.5	0.1	0.2	0.1	0.2	C.1	0.2.
Total Fraction Feed Carbon Converted	0-4	0-4	0.5	0.5	0. 5	0.6	0.4	0.4	0.5	0-5	0.6	0.6
Hydrogen/Coel Ratio, # of stoichiometric Moles Hydrogen Feed Moles Steam Feed Feed Steam Decomposed, #	15 1.8775 1.4856 43.3	17 2.1278 1.2874 20.8	19 2.378 2.378 46.6	23 2.879 2.394 29.2	26.0 3.2543 3.8855 42.0	31.0 3.8802 3.8802 30.5	16 2.0027 1.5586 41.0	18 2.2530 1.3444 18.5	2.879 2.539 41.0	26 3.2543 2.4846 25.8	34.0 4.2557 3.6707 42.6	36.0 4.5060 3.5155 33.3
Moles Hydrogasifier Effluent Composition, mole #	4.1242	3.6138	5.4164	5.3446	7.4409	7.4453	4.3314	3.7932	6.0254	5.8045	7.7960	7.4226
GH. H.a CO CO22	45.2 16.8 4.6 11.0	59.3 0.2 (<0.1) 9.9 1.1	\$1.1 17.2 4.5 12.1 0.8	47.1 8.0 1.3 10.1 0.8	35.64 18.13 3.43 11.32	39.68 12.18 1.55 9.15	42.9 19.0 4.7 10.3 0.9	56.5 2.9 0.5 9.0 1.0	37.3 21.8 4.5 10.1 0.7	43-5 12-9 1-8 8-6	35.45 23.81 3.88 8.70 0.57	40.65 16.69 2.07 7.76
Na HaS HaO Total	0.9 1.1 20.4 100.0	28.2 100.0	23.4 100.0	0.9 31.8 100.0	0.59 0.62 30.27 100.00	0.59 9.62 36.23 100.00	1.0 21.2 100.0	1.2 28.9 100.0	0.8 24.8 100.0	0.7 0.8 31.7 100.0	0.58 27.01 100.00	7.76 0.60 0.62 31.61 100.00
Dry Pipeline Gas Composition, mole ⊄ CR.	93.0 3.2	95 .9 0 .3	93.3 3.6	94.3 3.2	89.5 8.8	90.7 7.8	91.9 5.0	96.1 1.4	90.2 6.1	87.8 10.4	83.9 14.8	87.2 11.5
He CO CO Ne	<0.1 2.0 1.7	<0.1 2,0 1.7	<0.1 1.5 1.5	<0.1 0.9 1.5	(<0.1) 0.4 1.3	(<0.1) 0.2 1.3 100.0	<0.1 1.7	<0.1 0.7 1.7 100.0	<0.1 1.3 2.3	<0.1 0.4 1.3	<0.1 1.2 100.0	<0.1 1.2 100.0
Total Pipeline Gas Heating Value, Btu/SCF	952.5	972.5 0.8224	956.4	965.0	934.7	943.4 1.2372	946.3 0.8128	977.9 0.8348	932.8 1.0255	922.3 1.0672	897.7 1.2810	920.3 1.3014
MMBtu/100 lb coal Moles Sydrogen/MMstu	0.7997 2.35	2.59	0.9680 2.46	1.0161 2.84	1.1778 2.763	3.136	2.47	2.70	2.81	3.05	3. 322	3.462

Table 1.-Cont, SUMMARY OF HYDROGASIFIER OPERATION WITH STEAM HYDROGEN FEEDS AND CALCULATED PIPELINE GAS COMPOSITIONS (Quantities Based on 100-Lb Coal Feed)

					•						•	
Temperature in High-Temperature Zone, F			1	900					5	000		
Fraction Feed Carbon Converted in Low-Temperature Zone	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2
Total Fraction Feed Carbon Converted	0.4	0.4	0.5	0.5	0.6	0.6	0.4	0.4	0.5	0.5	0.6	9.6
Hydrogen/Coal Ratio, % of atoichiometric	19.0	20.0	26	28	36.0	38.1	21	22	29	31	42.1	43.1
Moles Hydrogen Feed Moles Steam Feed	2.378 1.1214	2.503 0.8091	3.2543 1.6613	3-5047 1-5526	4.5060 2.1272	4.7563 2.0050	2.6285 0.7432	2.7537	3.6230 1.0760	3.8801 0.9254	5.2570 1.4798	5.3822
Feed Steam Decomposed, % Moles Hydrogasifier Effluent	47.1 4.2716	22.5 3.4882	52.0 5.5264	36.0 5.1169	55.8 6.7867	43.3 6.4166	56.0 4.1389	22.5 3.3680	64	45 4.8403	61.5 6.8118	1.3455 48.2 6.3540
Composition, mole \$									5.2930	-		
Composition, mole \$ CHA. Ha. CO	43.5 25.6 7.3	61.8 7.9 7.8	47.8 27.4	49.4 17.8	40.27 29.54	46.69 22.39	44.9 29.6	64.2 12.8	42.8 31.8	52.5 23.1	40.96 35.23	47.67 28.52 6.48
GO _D	7.3 7.8	1.8 8.2	7.7 8.1	3.7 8.0	7.78 7.23	4.60 7.20	10.2 5.4	3.9 6.3	11.2 5.1	6.4	9.62 4.52	4.93
N≥ HaS	0.9	1.1	0.7 0.9	0.8 0.9	7•23 0•65 0•67	0.69 0.72	0.9 1.1	1.1	0.8 0.9	5.7 0.8 1.0	4.52 0.65 0.66	0.70 0.73
HaO Total	13.8	17.6 100.0	14.4	19.4 100.0	13.86 100.00	$\frac{17.71}{100.00}$	<u>· 7.9</u>	10.4	7.4	10.5	8.36	10.97 100.00
Dry Pipeline Gas	700-0	700.0	100+0	. 100.0	100.00	100.00	100.0	700-0	100-0	700-0	700.00	700.00
Composition, mole #	92.1	94.8	90.9	9 3.2 3.6	90.2 6.9	90.7	90-0	95.0 1.8	93.2 4.0	93.1 3.5	89.4	90.2
CH. He CO	4.1 <0.1	2.7 <0.1	5.5 <0.1	<0.1	6.9 <0.1	7.4 (<0.1)	7.1 <0.1	1.8 <0.1	4.0 <0.1	3. 5 <0.1	7.3 <0.1	7.7 <0.1
CO≥ Na	2.1 1.6	0.8 1.6	2.2 1.3	<u>1:7</u>	1.6 1.2	0.7	1.3 1.5	1.5 1.6	0.9 1.8	2.0	2.0 1.2	0.9
Total.	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	$\frac{1.1}{100.0}$
Pipeline Gas Heating Value, Btu/SCF	945.7	968.2	938.2	955.5	935.2	942.1	934.5	967.6	957.6	954.4	929.3 1.3691	938.6 1.3819
MMBtu/100 lb coal Moles Hydrogen/MMBtu	0.8496 2.80	0.8596 2.91	1.0542 3.09	1.0765 3.26	1.2966 3.475	1.3213 3.600	0.8746 3.01	0.8831 3.12	1.0891 3.33	1.1129 3.48	3.840	3.8 95

Table 2.-OPERATING DATA FOR THE PREPURIFICATION SECTION

raction of Total Carbon Converted					. 4			
emperature in High-Temperature		1700	1	800		1900		000
raction of Carbon Converted in Low-Temperature Zone De Romoved,	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2
moler/100 lb coal	0.4247	0.3047	0.3858	0.3179	0.3012	0.5518	0.1873	0.1922
noles/100 lb coal D₂ + H₂S Removed,	0.0453	0.0453	0.0453	0.0453	0.0453	0.0453	0.0453	0.0453
moles/hr D ₂ + H ₂ S to be Removed,	6, 122	4,433	5,525	4,532	4,259	3,225	2,770	2.803
moles/da. D ₂ + H ₂ S Removed,	146,931	106,396	132,597	108,769	102,208	77.391	56,48 8	67,235
MCF/dey Dp + HrS in Feed, % Dr + HrS in Effluent, €	55.613 12.1 1.0	40,271 11.2 2.0	50,188 11.3 2.0	41,169 10.2 1.0	38,686 8.9 1.0	29,292 9.5 1.0	25.166 5.5 1.0	25,448 7.3 1.0
raction of Total Carbon Converted				·	0.5			
one, of Carbon Converted in		-1700		1800	:	1900		Sc.u
ow-Temperature Zone	0.1	0.2	0.1	n.2	0.1	0.2	0.1	0.2
olem/100 lb coal S Removed,	0.6237	0.4770	0.4923	0.1502	0.4011	0.3009	0.3251	0.238
soles/100 lb coal la + HaS Removed.	0.0470	0.0470	0.0470	0.0470	0.0470	0.0470	0.0470	0.047
soles/hr	8,736	6,637	5,478	4,658	4.286	3,438	3,569	2,67
noles/day	209,664	159,288	131,472	111,792	102,864	82,512	85,656	64,175
MCF/day De + MeS in Feed, ≸ De + HeS in Effluent, %	79,358 13-0 1-0	60,291 10.9 2.0	49,762 10.9 3.0	42,313 9.4 2.0	38,93 4 8.8 2.0	31,231 8.9 3.0	32,421 8.8 2.0	24,290 6.1 1.0
raction of Total Carbon Converted			<u> </u>		0.6			
Zone, OF raction of Carbon Converted in	1	700	18	00	19	···	20	or
cv-Temperature Zone	0.1	0.2	0.1	c.s	0.1	0.5	0.1	0.2
S Removed,	0.7561	0.6003	0.5793	0.4869	0.3643	0. 36 67	0.1900	0.207
oles/100 1b coal be + H-S Removed,	0.0461	0-0#6#	0.0450	0.0459	0.0455	0.0463	0.0455	0.045
holes/hr Dg + HgS to be Removed,	7,095	5,445	5.077	л, 56л	3,452	3 ,25 6	1,789	1,83
noles/day D ₂ + H ₂ S R emoved ,	170,280	130,680	121,848	102,336	62,848	78,144	42,936	44.05
MCF/day Og + H≥S in Feed, ⊈ O⇒ + H≥S in Effluent, ⊈	64,440 11.9 2.0	49,464 9.8 2.0	46,128 9.3 2.0	38,736 6.3 2.0	31,368 7.9 2.0	29,568 7.6 2.0	16,248 5.2 2.0	15, 6 5 5. 2.

Table 3 - OPERATING DATA FOR HYDROGEN PRODUCTION SECTION

-	Fraction of Total Carbon Converted					0.1			
•	Temperature in High-Temperature Zone, OF Fraction of Carbon Converted in	17	00	18	00	130	0		00
•	Fraction of Carbon Converted in Low-Temperature Zone Char Composition, at 5	0.1	0.2	0.1	0.5	0.1	0.2	0.1	0.2
•	C -	0.7998 0.0218	0.7998 0.0218	0.7998 0.0218	0.7998 0.0218	0.7988 0.6218	0.798B 0.0218	0.7 998 0.0218	0.7 998 0.0218
:	ile Oz Na	0.0142	0.0142	0.0152	0.0142 0.0057	0.0142 0.0057	0.0142	0.0142	n. กา น ว
•	\$	0.0057 0.0009	0.0057 0.0009	0.0057	0.0009	0.0003	0.0057	0.0057	0.0057
•	Ash Moles H _P /100 lb Coal	0.1576 1. 6 775	0.1576 2.1278 56.3452	0.1576 2.0027	0.1576 2.2530	0.1576 2.3785	0.1576 2.5033	0.1576 2.6285	0.1576 2.7537 56.3452
	Th Chur/100 1b Coal Hourly Scaleup Factor	55.3152 13,025.8	12.666.2	50.3452 12,815.8 26,405.0	16.3452 12.478.1 28.723.0	56.3452 12,275.2	56.3452 12,118.1	56.3452 11.910.2	55.3452 11.795.6
	Process Hydrugan, coles/hr Total Cher Available, 1b/hr Process Hydrogen, coles/hr	55.3452 13.025.8 25.160.4 733.941	27,727.5 713,679	722, 109	703,081	30,013.3 691,646	31,205.4 682,797	11,910.2 32,207.8 671,002	11,795.6 33,925.7 664,625
•	CO Ha CA	24, 455. 9 125. 8	83.31 26,951.1	79.34 25,665.7	86.90 28,113.2	90.24 29,190.4	95.76 30, 331.6	95.77 51,306.0	101.94 32,975.8 169.6
Pr	CF4 Na	125.8 226.4	138.6 249.5	132.0	114.6 260.3	150.2 270.3	30, 331.6 150.0 280.8	161.0 283.3	169.6
	iĝo	25.2	27.7	237.6 26.4 264.0	28.9 289.0	30.0	11.2	12.2	305.3 33.9 239.3
	COg Char Required, lb/hr Excess Char, lb/hr	25.2 251.6 304,216 429,725	335.255 378,424	319,265 402,044	349.710	%28,755	312.1 377, 707 305, 450	389, 427 281,655	410, 199 254, 426
	Conl Remitred. Th/hr	0	0	0	353, 371	0	U	0	0
e (°	Process Oxygen Required, 15/hr Steam Required, 15/hr	234,246 121,686	258, 146 134, 102	245,834 127,766	269,277 139,884	279, 428 145, 157	290,526 150,923	299,859 155,770	315,853 164,080
	Fraction of Total Caroon Converted					0.5		· · · · · · · · · · · · · · · · · · ·	······································
^	Texporature in High-Temporature Zone, OF Fraction of Carbon Conversed in	17	00		00	130	¢	20	0000
	Lew-Temperature Zone Char Composition wt 5	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2
-4	C Ka	0.77113 0.02523	0.77113 0.02525	0.77113 0.02523	0.77113	0.77113 0.02523 0.01644	0.77113 0.02523 0.01654	0.77113 0.02523 0.01544	0.77113 0.02523 0.01644
_	Ha Om Na	0.01644 0.00485	0.01544 0.00485	0.01644 0.06485	0.01644	0.00465	0.CO485	0.00485	0.00485
111	5	0.00000 0.18235	0.00000 0.15215	0.00000 0.18235	0.00000	0.00000	0.0000 0.16235	0.0000	0.00000
n	Moles Fe/100 1b Conl Th Chom/100 1b Conl	0.00000 0.18235 2.75 48.59755	0.00000 0.15215 2.875 48.65795	2.879 48.69755	0.18275 1.2543 48.6775%	3.2543 48.62155 2.531	3.5C4"	3.6230	3.8801
I	Ash Moles Fg/100 1b Coal 1b Char/100 1b Coal Haurly Scaleup Factor Process Hydrogen, coles/hr	10,751.1 26,727.1 524,039	10,251.5 20,44.6 457,228	10.15	7.740.4	2,551	3.6°6.5 34.696.2 3°1.222	3.6236 46.69755 9.574.5 35.650.4 465.764	3.8801 48.69755 9.360.0 57.263.9 455.809
Z	Total Char Available, 1b/hr Process Hydrogen, moles/hr			494,655	\$2.6"5.6 4"5.52"	35, 052, 4 491, 105			
0	Process Hydrogen, moles/hr CO Ks CHe	79.11 25,589.9	21.23 29,513.4 151.9	20,40 27,244.0 150.4	51, 164.6 165.5	79.45 12,155.1	104.83 33, 313.3	34,652.2	26, 317. 7 26, 317. 7
_	CH⊕ Na H#O	131.6 236.9 26.3	151.8 273.3	270.6	163.5 274.1	165) à	314.0	178.3 320.9	186.87 326.3
_	CO ₂	26.3 263.3	273.3 10.4 10.6 173.649	300.9	274.1 128.5	77.5.5.7.7.5.7.7.7.7.7.7.7.7.7.7.7.7.7.	34.9 459.6	380.5 156.5	376.3 37.4 373.6 455,609
0	Char Required, 1b/hr Excess Char, 1b/hr	253.5 323,366 200,013	373,649 125,5"3	3"0,225 124,430	402.150 "5,151	40", 098 '4, 095	41,953	439,693 27,675	455,869
Φ.	Coal Required, 1b/hr Process Oxygen Required, 1b/hr	5+5,0.4	280.23"	٠ ٨	c	ř	122,003	327, -20	344.518
⊣	Steam Required, lb/hr	129,585	149,460	2***, 669 148, 030	301, 602 160, 854	165.933	171.73	1"5, 4""	103,743

Table 3. Cont. OPERATING DATA FOR HYDROGEN PRODUCTION SECTION

Fraction of Total Carbon Converted Temperature in High-Temperature Zone. OF	17	700	18	100	1°	% ^		1000
Fraction of Carbon Converted in	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.3
Char Composition, Vt \$	0.73085	0.75095	0.73085	0.73085	0.75085	0.73085 0.02912	0.770 0 5	0.7308 0.0298
ř.	0.73085 0.02987 3.01948 0.00371	0.02989	0.02259	0.02955	0.029 8 9 0.01946	0.01948	0.01348	0.0194
Çe Ne	0.00371	0.01348 0.00371	0.00371	c.00371	0.003"1	C.00371	0.00371	0.00 3 7
5	0.00000	0.00000	0.00000 0.21604	6.66 696 6.21684	0.00000	0.50000 0.23508 3.2583	0.00000 0.21604 3.25%3	0.2160
Ash Koles He/100 lb Coal	0.21604	0.21604 3.2543	3,2543	5,254 5 41,1035	3.2545	5.2535	3.25%3	3.254 41.107
Th Char/100 lb Ccal	3.2543 41.1035 8.844	41.1035 8,420	3.2543 41.1055	1,1035	41, 1015	41.1035 24.578.7 324,040	1.1035	*1, 102
Hourly Scaleup Factor	27,610.1	73,615.5 8,450	9, 132 35, 604.6	9,004 5/,105.0	17,234.8	19.578.7	112.15	309,83
Process Kydrogen, moles/hr Total Char Available, lb/hr	* 35,519	346,071	354,254	126, 772	330, 193	384,040	312,433	309,89
Process Hydrogen, molos/hr	84.8	100.22	105.98	111.39	111.68	115.92	123.60	125.4
CO Na Cha	28,781.0	32,671.1 168.1	51.607.7	%,066.1	M. 172.2	37, 1987	11, 244.2 205."	501. 201.
CH♠	148.1 266.5	168.1 302.5	170.0	185.6 353.9	135.1	172.9 147.2 36.6 45.6	370.2 41.1	979.
Ng HaO	23.6	33.6	35.6	37.1	37.8 372.3	28 . 6	411.4	\$1.1
CO ₂	29.6 296.1 %3,512	391.1	35.6 352.1 334,254	371.0 386.9%	330, 143	126,040	312,633	309,02
Char Required, 1b/hr Excess Char, 1b/hr	G	. 0	0	0		• •	151.092.3	
Con! Reguland, 1b/hr	5,481 265,680 147,600	67, 350 237, 533 165, 296	91,746 906,000	125, 009 326, 880	121.175 25.000	1 * 0 . 0 / 1 . 5 3 * * . 10 0	7.0, \$13	190, 100. 190, 95 199, 97
Process Oxygen Required, 1b/hr Steam Required, 1b/hr	147.600	165,296	170,000	171.G00	180,600	185,548	197, 446	199, 97

Table 4.-UTILITY REQUIREMENTS FOR THE PLANT

Fraction of Total Carbon Converted				0.1	.			
Temperature in High- Temperature Zone. F	17	.00	76	300		1900	20	000
Fraction of Carbon Con- verted in Low-Tempera-			-		•			,,,,
ture Ione Power for Coal Handling,	0.1	0.5	0.1	0.2	0.1	0-5	0.1	6.5
hp Found for Hydrogasifier,	֥553	4,427	4,479	4,361	4,290	4.235	4,163	4.123
hp Fower for Propuritiestion,	27,900	27.130	27,450	26,727	25.293	25,956	25.511	25,265
np .	€,234	5.065	5,82 <u>1</u>	5.134	4.945	4.230	3.914	3.93?
Prior for Congress Production, hp	75 .5 58	84.012	79,524	87.480	91, 197	94,059	97.645	102,348
Power for Hydrogen Con- pressor, hp Power for Fluor Process,	18,727	20,638	19,653	21.527	22,339	23,226	23.972	25.251
hp Total Fower, hp Steam for Power.	26,731 150,713	20,458 170,730	28,053 164,980	30,728 175.957	31,887 180,951	33, 153 164, 869	34.218 169.425	36,043
lb/hr at 525 pala	1.237,130	1.297.300	1,269,800	1,339,570	1,369,510	1.398.690	1,424.250	1,442.310
Steam to Gasifier, ib/hr at 525 pula Steam to 2nd Shift.	121,686	134, 102	127,706	139,884	145.157	150,923	155.770	164.080
15/km at 525 pola Total 525 pola Steam,	151,503	175, 191	169, 502	185,874	192,880	200,542	266,283	518. C5#
15/hm	1,520,509	1,609,593	1.567.198	1,665,328	1.707.547	1,750,155	1.787.003	1,624,414
Steam to Hydrogesifier, lb/hr at 1025 poin	349,635	293,780	359.860	302.393	247.987	176.640	159,487	96.093
Font Reculred for 1025 pain Steam, MCStu/hr	338.70	285.47	349.68	293.84	240.97	177.64	15 ⁴ .08	93.38
Hout Required for 525 gain Steam, Michight	1,494.66	1,582.03	1.540.47	1.636.76	1.675.18	1,719.97	1.756.12	1,792.78
Heat for H. Prehent,	52.00	50.00	50.40	55.20	52.50	<u>40.00</u>	7.48	7.00
Total Hone Required, Wildenship	1,885.36	1,925.50	1,959-55	1,995.80	1,971-65	1,940.61	1.918.58	1.893.16
Heat From Excess Char,	4,422.50	3,894.74	4.146.07	3.535.90	3.383.54	3.144.10	2.898.70	2,508,60
Excess Heat From Re- mining Char, Midte/hre Heat Required From Addi-	2,537.14	1,968.24	2,186.52	1.543.10	1,413.89	1.203.49	980.21	705.44
tional Coal, MeBtu/hr Coal Required, ton/hr	0 0	0	0	0	0	ç	0	0
Total Fower Comerated,	10,787	9,492	10,300	9.495	9,235	8,465	8,079	8,060
Process Water to Coal Handling, MM lb/hr	1.156	1.124	1.137	1.107	1.089	1.075	1.057	7 n47
Process Water to Pre- purification, MM 1b/hr	0.860	0.732	0.888	0.757	0.839	0.676	0.789	r 6 3 6
Process Water to 1st Shift, MM 1b/br	0.523	0.575	0.549	0.601	ი.624	0.649	0.570	0.705
Process Water to Scrubber, MM lb/hr	0.110	0.132	0.725	0.137	0.142	0.148	0.153	0.161
Total Process Water. MM lb/nr	2.658	2.564	2.699	2.602	2.694	2.548	2.669	2.549
Boiler Feed Water, MM	5,024	2.820	2.962	2.862	2.963	2.803	2.936	2.804
Total Process and Boiler Feed Water,	E 500	O	- 650	- 4-09				
MM lb/hr Total Process and	5.582	5.384	5.668	5.464	5.657	5.351	5.605	5-353
Boiler Feed Water, gpm Cooling Water for Coal	11,164	10,768	11,336	10,928	11,314	10,702	11,210	10,706
Fandling, gpm Cooling Water for Hydro-	34,850	33,900	34,350	33,400	32,800	32,450	31,900	31,600
Gesiller, gpm Cooling Water for Prepuri-	37,100	35,600	36,500	35,500	35,000	34,500	33,800	33.550
fication, gpm Cooling Water for Methana-	18,500	13,390	16,680	13,690	12,860	9,740	380 و8	8,460
tion, spm Cooling Water for Oxygen	10,080	0	10,080		10,080	5,550	•	10,080
Production, gpm Cooling Water for Hydrogen	118,800	130,800	124,640	135,520	141,670	147,300	152,030	160,140
Production, gpm Total Process Cooling	54,260	26,740	25,460		28,440	30,090	31,050	32,710
Water, spm Cooling Water for Power gpm Total Cooling Water, spm	243,590 419,310 662,900	240,430 <u>431,300</u> 671,730	247,710 426,760 674,470	250,450 443,560 694,010	261,850 450,690 712,540	259,630 456,850 716,460	267,280 462,500 729,780	275,540 460,180 730,720

^{*} Based on 80% fuel efficiency

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Table 4 -Cont. UTILITY RECUIREMENTS FOR THE PLANT

ion of Total Carbon				0.5-				
rature in High-	1-	e	1E	_	10		20	юс
rature Zone, of on of Carbon Con-	17	·	10		13			···
Lov-Tempera-	0.1	0.2	0.1	c.5	c.1	0.2	0.1	0.2
il Handling,	3,761	3,583	3,550	3,411	3,453	3,362	3.343	3,25-
gasifier,	21,321	20,684	20,693	19,864	20, 129	19,712	19,484	19,058
ification,	8,041	6,590	5.788	5,221	±,300	¥, 5 :8	£, £68	3,249
Production,	40,531	46,745	45,313	50, 703	52,507	52,566	55.572	55,958
n Cons-	19,595	22,600	22,393	24,723	24,523	25, 369	26,535	27,810
rocess,	27,970 121,819	32,250	130,701	137,851	35, 127 120, 759	37.068	37,876 137,278	39,595
		132,652				143,075		129,655
&	1,218,190							
la ft,	129,583	149,450	148,090	150,854	162,637	171,735	175,477	183,743
ia team,	159, 191	195,130	193.354	210.015	212,605	225,222	229,103	240,110
sifier, sia	1,516,964							
or 1025	461,028	442,155	464,640	436,918	295,71	270,667	185,410	156,050
tu/hr r 525	447.99	429.65	451.50	424.56	287.36	263.01	180.17	151.54
tu/hr mat,	1,496.06	1,648.19	1,625.73	1,725.37	1,758.46	1,813.79	-	1,908.67
ed,	179.00	200.00	200.50	187.00	159.00	145.00	131.90	130.60
char,	2,123.05	2,277.84	2,277.73	2,336.93	-	2,221.80		2,191.11
n Ne-	2,043.46		1,270.88	747.54	756.76	¥27.78	275.54	0
#Btu/hr* rom Add1- #Btu/hr	70 50	0 995.22	1,006.65	7 580 30	1,448.08	0 1,794.02	0	0
on/hr*	79.59 3.67	45.94	46.48	1,589.39 73.37	66.84	85.66	87.10	2,191.11 101.14
Coal	11,802	10,173	9, 338	8,632	9,353	7,760	7,811	7,133
b/hr o Pre-	0.955	0.910	0.901	0.866	0.877	0.859	0.849	0.834
MM lb/hr o lst	0.932	0.877	0.979	0.907	0.874	0.792	0.810	0.725
	0.547	C.631	0.626	0.6 79	0.688	0.725	0.741	0.777
/hr iter,	· <u>0.125</u>	0.749	0.143	c.155	0.157	0.155	0.169	0.177
er, MM	2.559	2.562	2.649	2.607	2.596	2.541	2.569	2.513
nd	3,069	3.083	<u>2.98</u> 7	2.960	5.810	2.831	2.566	2.850
iter,	5 .628	5.645	5.636	5.567	5.445	5 .372	5.435	5.363
nd ter, gju	11,256	11,290			_	-		
or Comil	28,800	26,450			•	-		-
or Hydro-	26,400		-			a= a-	••	
ar Prepuri-	26,380							
for Methana-	10,080		-		• -	-	· ·	
for Chygen on for Hydrogen	123,190							_ •
ZC.	25,390							
ooling or Power gpa	240,240	249,572	247,602	258,136	262,452		-	
Water, gim	\$07,01\$ 647,254	682,481	673,183	701,107	709,010	726,146	271,197 465,543 736,740	753,622

^{*} Based on 80% fuel efficiency

Table 4 -Cont. UTILITY REQUIREMENTS FOR THE PLANT

Fraction of Total Carbon Conversed					e .					
Temperature in High- Temperature Zone, of		700		900			Cons			
Fraction of Carbon Con-		(U()			I	9¢c		.00c		
verted in Lov-Tempera- ture Zone	0.1	0.2	0.1	c.2	0.1	c.2	c.1	0.2		
Power for Coal Handling,	3,110	3,178	3, 163	3,234	3.231	3.255	3.291	3.299		
Power for Hydrogasifier, hp	17,096	16,277	15.720	15,473	15,527	75.250	14,703			
Power for Prepurification,	6,907	5,766	5,511	4,929	4,387	4.254	3,237			
fower for Oxygen Production, hp	56,129	25,620	99,039	106,527	105,221	111.405	107.115			
over for Hydrogen Com- pressor, hp over for Fluor Process,	22,039	25,018	25,501	27,517	27,714	28.714	30.518			
D	31,458	75,710	37,827	36,421	30,550	40.0F*	43.705	44,745		
al Power, hp	156,739	182,569	187,761		195,539	203,6=5	202,555	305.234		
hr at 525 psia pe to Gamifier,	1,247,390	1,355,690	1,397,510	1,452,210	1,446,390	1,598,550	1,556,680	1,574,330		
m at 525 psia a to 2nd Shift,	147,600	165,296	170,000	181,600	180,600	165,648	197,490	199,975		
at 525 psia 25 psia Steam,	100,205	215,010	<u>228,815</u>	236,460	239,287	247.025	<u> 264, 365</u>	268,241		
hdrogasifier,	_	1,736,996	1,796,425	1,872,270	1,866,277	1,932,023	2,018,535	2,042,546		
1025 pein ired for 1025	619,138	588,601	537,693	506,935	307,815	284,786	202,777	182,731		
n, Mistu/hr red for 525	601.63	571.96	522.49	492.60	299-11	276.73	197.04	177.56		
s, Militu/hit Preheat,	1,563.44	1,713.06	1,771.67	1,846.47	1,640.56	1,905.40	1,990.72	2,014.40		
Required,	310.10	335.00	322.52	302.00	213.40	195.00	216.50	214.00		
Excess Char,	2,475.17	2,620.02	2,616.68	2,641.07	2.353.07	2,377.13	2,404.26	2,405.96		
From Ne-	0	0	0	0	o	0	0	o		
har, Mibbu/hr	0	0	0	0	0	О	C	c		
nd, Mebtu/hr	2,475.17 114.25	2,620.02 120.99	2,616.68 120.78	2,611.07 121.91	2,353.07 108.62	2,377.13 109.78	2,404.26 110.98	2,405.96		
Generated, er to Coal	10,017	8,944	8,674	8,183	7,618	7,499	6,528	5,569		
MM 1b/hr er to Pre- lon, MM 1b/hr	0.785	0.747	0.722	0.716	0.713	0.700	0.675	0.669		
lon, MM 1b/hr	1.053	1.003	1-014	0.951	0.871	0.809	0.829	0.766		
1b/hr	0.616	0.699	0.740	0.771	0.774	0.802	0.855	0.868		
er to M 1b/hr IS water,	9,140	0.150	0.159	0.176	9.177	0.183	0.195	0,198		
Water, 1#	2.594	2.608	2.645	2.608	2.535	2.494	2.554	2.501		
5 and	2,253	2.859	2,409	2.869	2.789	2.743	2.809	2.751		
Water,	5.447	5-477	5.554	5,477	5.324	5-237	5.363	5.252		
a water, gon	10,894	10, 954	11,108	10,954	10,648	10,474	10,726	10,504		
er for Coll	23,650	22,550	21,750	21,400	21,500	21,100	20,400	20,200		
er for Hydro- gpa	18,400	17,500	15,900	16,600	16,650	16,350	15,800	15,600		
iter for Frejuri-	21,429	16,445	15,334	12,878	10,426	9,834	5,403	5,545		
CAR YOR MATRIALS	10,080	10,080	10,080	10,080	10,080	10,080	10,080	10,080		
iter for Caygen as, gits tter for Hydrogen	134,130	150,850	155,140	165,730	164,820	174,510	182,730	185,030		
	28,550	12.412	34, 330	35.780	35,900	37,200	39,670	40.250		
for Power gim	236,239 197,037	249,837	253,534 439,648	262,468 451,265	259,376 449,765		274,083	275,705		
g Water, gin	633,276	125,738 575,575	693,182	451,265 713,733	709, 141	269,074 461,405 730,479	477,799 7 51, 882	482, 990 759, 695		
					-		•			

^{*} Based on 80% fuel efficiency

Table 5 MATERIALS FOR HYDROGASIFIER AND GASIFIER (Quantities Based on 1 Hr Operation at 250 Billion Bin Day)

Tomporatura in High-Temperatura		-1700		tsc=		1900		-2005		-1"00		-1800
Zine, "F Fraction of Carbon Converted in		1100								-1.00		
Low-Temperature Zone	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2
cal to Hydrogasifier, ton	651.29	633.31	640.79	523.30	613.76	505.30	545.41	580.78	5 45.01	512.50	50".68	488.04
cal to Gasifier, ton	0	0	c	c	C	o	0	o	٥	C	c	J
ctal Processed Coal, ton	651.29	633.31	640.79	623.30	513.76	505.46	595.51	580,78	5,48.0;	512.58	507.88	486.C4
cal for Prwer, ton	o	0	c	O	c	c	r	•	* 6 *	45.94	46,48	13.51
stal Coal, ten	651.29	653.31	640.73	523.90	613.76	€05.90	595.51	REAL OF	541.60	558.52	554. 1€	561.41
recessed Hydrogen to Hydro- gasifier, moles	25,160.4	27,727.5	26,405.0	28,923.0	30,013.3	31,205.4	12,207.8	33, 225.7	26, 527.1	10,764.0	10,000.4	12,679.6
rccessed Oxygen to Gasifier,	117.12	129.07	155.95	134.64	139.71	145.26	(49, 95	157 - 27	121.40	140.12	138.85	150.801
team to Hydrogasifier, Ib	348,635	293,780	359,666	302, 593	247,327	175,540	159, 487	95, 6 31	461,028	*42,155	húk, úhc	436,918
team to Casifier, 1b	121,686	134, 102	127,706	137,894	145,157	150, 123	155,770	164,080	129,586	149,460	146,090	160, 85¢

Fraction of Total Carbon Conver	rted -		-0.5										
Temporature in High-Temperature Zono, F		1700		-5000		-1700		-1600		-1900			
Practice of Carbon Converted in Low-Temperature Zone	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2	0.1	0.2	
Coal to Hydrogasifior, ton	494.05	483.62	478.22	468.00	442.20	421.00	*0 6.60	40C.20	401.60	394.20	360.30	376.90	
Coal to Gasiflet, ton	0	0	0	1.77	2.74	33.58	15.87	62.50	60.69	70.03	90.55	95.05	
Total Processed Coal, ton	494.05	483.82	478.22	469.77	144.04	454.58	452,47	492.70	462.29	464.23	470.85	471.05	
dral for Payer, ton	65.24	82.66	8:.10	101.14	114.25	120.39	120.78	121.91	108.62	109. 'F	110.08	111.06	
T tel Coel, ton	550.30	566.48	555.32	570.31	559-17	5-5.5-	573125	594.51	570.91	5"4,01	561.03	583.01	
Processed Hydrogen to Hydro- gesifier, soles	33,682.4	34,890.2	35,650.4	37,263.9	23,610.:	13,612.2	15,604.6	37, 105.0	37,034.8	.0.578.9	41,156.0	11.730.0	
Processed Oxygon to Gasifier, ton	154.66	161.00	164.51	172.26	132.28	148.77	153.00	163,24	162.54	172.08	180.21	182.48	
Steam to Hydrogasifier, 1b	235,741	270,667	165,410	150,050	619, 138	589,501	537,633	506,955	307.815	284.786	202,777	145,731	
Steam to Casifier, 1b	162,837	171,735	175,477	133,743	147,600	165,295	170,000	181,600	180,600	185,648	197.490	199,975	

Table 6. SUMMARY OF COST DATA

Praction of Total Carbon Converted					-0.4			
Temperature in High-Temperature Zone, OF Fraction of Carbon Converted in	1700) 	190	00	190	0	50	00
Coal Storage, \$ million Coal Granding, \$ million Hydrogasification, \$ million Prepurification, \$ million Shift, \$ million Methanation, \$ million Oxygen Production, \$ million Hydrogen Production, \$ million Offsite Facilities, \$ million Total Bare Cost, \$ million Coal Cost, \$ million/yr Char Gradit, \$ million/yr Char Gradit, \$ million/yr Cas Price, \$/YMBtu	0.1 4.622 8.520 9.721 0.783 13.745 22.700 34.228 11.229 28.5265 5.129 0.58938	0.2 4.549 8.357 22.720 8.454 0 0.401 16.256 24.100 35.105 119.942 27.7320 3.980 0.60052	0.1 4.580 8.335 22.450 8.578 0.905 15.260 35.200 35.504 118.578 28.6666 4.421	0.2 4.505 8.280 22.500 8.270 0.684 16.778 24.700 26.272 122.129 27.228 3.116 0.61110	0.1 4.461 8.173 17.500 8.189 0.997 17.511 25.700 76.216 118.747 26.6827 2.855 0.59994	0.2 4.424 8.098 17.450 6.807 0.682 17.908 26.500 26.032 17.501 2.500 2.433 0.59856	0.1 8.380 8.013 19.510 6.844 6.060 1.052 18.238 27.200 35.829 117.125 26.0833 1.962 0.59605	0.2 4.357 7.948 15.590 6.735 0.050 0.600 18.699 28.400 35.088 117.677 25.8324 0.66085
Fraction of Total Carbon Converted Temperature in High-Temperature Zone, CF	1790		1800)	-0. 5 -1900		2000)
Fraction of Carbon Converted in Low-Temperatury Zone Coal Storage, \$ million Coal Grinding, \$ million Hydrogasification, \$ million Prepurification, \$ million Shift, \$ million Methanation, \$ million Coxygen Production, \$ million Coxygen Production, \$ million Offsite Facilities, \$ million Total Bare Cost, \$ million Coal Cost, \$ million/yr Char Credit, \$ million/yr Gas Price, \$ Million/yr Gas Price, \$ Million/yr	0.1 4.125 7.152 23.500 12.391 0.786 15.066 23.200 25.789 122.107 23.725 0.60530	0.2 4.133 7.2550 9.821 0.654 17.577 25.603 185.541 23.670	0.1 4.115 7.159 23.250 8.374 0.895 17.181 25.100 124.091 23.491 23.495 0.61652	0.2 4.141 6.952 23.300 8.019 0.627 18.228 27.568 12.155 23.737 0.62926	7.1 4.131 7.022 17.770 7.609 0.060 1.080 18.899 27.800 37.314 121.655 23.676	0.2 4.159 6.924 17.720 6.625 0.820 19.167 29.160 37.883 122.394 23.907 0.61799	0.1 4.128 6.867 16.771 0.060 1.250 19.428 29.600 37.190 122.244 23.614	0.2 4.700 6.784 16.684 0.848 20.088 30.800 37.666 124.180 24.031 0.62533

Practice of Total Custon Converted	0.6												
Temperature in High-Temperature			1800		1900)							
Prestion of Content Converted in Low-Puspendary Town Coal Storage & mailion	0.1 4.139	0.2 4.207	0.1 4.194 6.450	4.2 76	0.1 4.202	0.2 4.186 6.457	0.1 4.215 6.444	0. 3 4. 32 0					
Cool Granting & stillor Hydrogendfloation & stillor	6.516 24.6 5 0	6.501 29.700 8.808	23.600	6.472 23.650	6.477 16.820	16.770	16.600	4.920 6.436 16.520					
Propertionality, & million- Shift, & million- Nothemation, & million-	10.045 0 0.900	9.600 9.600	8.519 0 0.970	7.890 0 0.700	6.9 62 0 1.180	6.799 0.970	5. 797 0 1. 7 00	5.353 0 1.000					
Oxyger Production, \$ million Refrance Production, \$ million	16.7 8 4	18.238 28.200	0.970 18.635 29.500	19.428 30.600	19.428 30.730	20.088 31.600 39.085	20.617 33.300 30.218	20.617 37.800					
Office Partition, 5 million Total Base Cost, 5 million Coal Cost, 5 million/yr	126.675 24.497	39, 964 131.218 25,207	40,158 132.006 25.109	133.678 25.606	124.194 25.006	125.955 25.129	127.057	127.255 25.536					
Char Credit, & million/gr Gas Price, \$/88800	a.62 602	0.64659	0.64764	0.65787	0.62507	0.63141	0.63844	0.6 7948					

Table 6.-Cont, SUMMARY OF COST DATA

High-Pressure Methanation

Imboratory Methanation Study

Quotations were received for most major equipment; selecting and ordering major equipment and other equipment having a long delivery time was continued.

A survey was made of ways to continuously analyze the feed and product gases for carbon monoxide and carbon dioxide. Because of the 0.10 mole percent limit set for synthetic pipeline gas, special analytical techniques are required for analyzing exit gases. In addition, a dual-range analyzer is required to operate accurately at composition ranges of 0-2 mole percent and 0-20 mole percent. For these reasons, a continuous process monitoring-type infrared analyzer was decided upon; the appropriate manufacturers will be contacted and quotations obtained.

Pilot Plant Studies

The objective of this study is to investigate the hydrogasification of suitably treated coals in a continuous-flow pilot unit in order to develop data for the design of an integrated pilot plant. In the present phase of hydrogasification studies, tests are being conducted in a high-temperature, balanced-pressure pilot unit. Gas-solids contacting is achieved in a 4-in.-diameter reactor tube, the hydrogen or hydrogen-steam mixture flowing upward and the coal flowing downward as either a moving or fluid bed, or in free fall.

Hydrogasification

Rebuilding of the hydrogasification pilot unit reactor heating furnace was completed with the installation of the remaining four heating elements. The entire furnace was tested for operation to a temperature of 600°F.

A new 4-in.-diameter reactor tube, type 446 alloy, and an expansion bellows was installed in the reactor shell. Before installation, the reactor tube was wrapped with a 1/2-in.-thick layer of Fiberfrax insulation so that temperature profiles could be recognized. The lower 31 inches of the reactor was left uninsulated to improve heat transfer to the tube in this zone where the heat loss is high, and final preheating of the steam and hydrogen feed is accomplished.

Reactor tube-wall thermocouples were drawn through the shell and sealed in glands against pressure. Six of the 31 reactor tube thermocouples were postioned on the outside of the Fiberfrax insulation at approximately 31-inch intervals, and at nearly the midpoints of each of the top six heating zones. Temperatures sensed by these thermocouples, and those sensed by thermocouples on the tube wall at corresponding levels will be used to determine

-26-

temperature gradients across the insulation to establish direction of heat flow. Location of the reactor tube thermocouples is shown in Fig. 1.

Electrical wiring of the seven solenoid valves for the new reactor-shell differential-pressure control system was completed. Rupture disks were installed in a pressure-equalizing line between the reactor tube and the shell. These disks are designed to rupture and equalize the pressure between the reactor tube and the shell in the extreme case of failure of the differential-pressure control system.

The modified pressure vessel, which will serve as a temporary residue receiver, was installed in the pilot unit. This receiver has a volume of 7.7 cu ft compared to 5.1 cu ft in the original receiver. The reactor discharge screw housing, the discharge screw, drive shaft, and the variable speed drive were installed. A feed calibration of the screw was made, correlating weight of coal discharged with screw rpm.

Piping the hydrogen and steam feed lines to the bottom of the reactor was started.

The coal feed hopper and the coal screw feeder were reassembled to the top of the reactor. Reconnection of the six gas-sampling probes was started.

Coal Pretreatment

INSTITUTE

Four coal pretreatment tests were conducted in the continuous-flow fluidized bed pretreater. These tests are a part of the program for defining minimum pretreatment conditions for producing nonagglomerating char for the hydrogasification pilot unit.

Nominal conditions for these tests are given below:

Run No.	Solids Rate, _lb/hr	Avg Solids Residence	Gas Rate, SCF/hr	Oxygen,	Temp,
FF-12	72	1.27	613	2.1	440
FP-13	73	1.0	602	2.2	536
FP-14	78	1.0	590	9.8	666
FP-15	74	1.0	600	10	7 0 8

It was found that with a 2% oxygen concentration in the pretreatment gas, sufficiently high bed temperatures for adequate coal pretreatment could not be maintained in test Runs FP-12 and FP-13. In earlier tests, it was shown that sufficiently high bed temperatures for pretreatment could be maintained with 21% oxygen (air). To investigate oxygen concentrations with respect to oxidation rate,

8 4

1 E C H N O L O B T

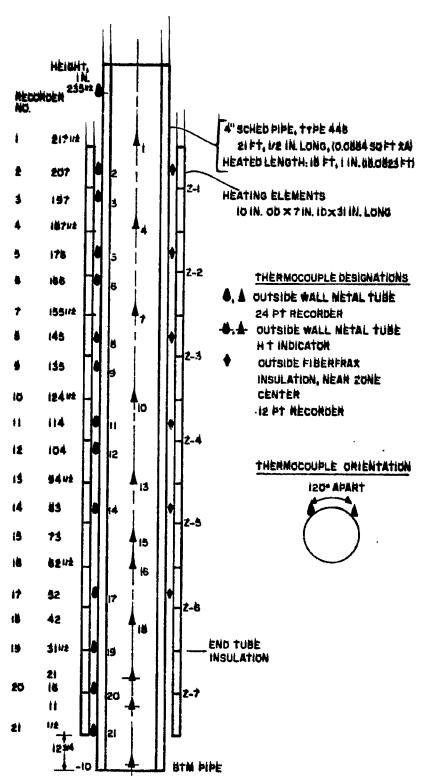


Fig. i -BALANCED-PRESSURE REACTOR THERMOCOUPLE LOCATIONS

further tests (Runs FP-14 and FP-15) were conducted with a 10% oxygen concentration. Bed temperatures in these tests were maintained at a level adequate for pretreatment. Chars produced in these tests have not yet been evaluated for agglomeration characteristics. A more detailed data presentation of these tests will be made when other analytical results are completed.

Operating conditions and results of four coal pretreatment tests conducted last month are presented in Table 1. Chemical and screen analyses of the feeds and residues of these tests are given in Table 2.

Pittsburgh seam bituminous coal obtained from Consolidation Coal Company was pretreated in these tests. In Run FP-7 the coal was from Montour 4 mine; in the other tests the coal was from the Ireland mine.

When it was found that an oxygen concentration of 4% and coal residence times of 27 to 40 minutes were not sufficient for adequate pretreatment, the oxygen concentration was increased to 21% in the pretreatment gas; the average coal residence time was increased to about 1 hr by increasing the bed height to 5 ft and by decreasing the coal feed rate to 50 lb/hr.

Laboratory evaluations of the chars produced in these tests (procedure described in the January 1965 Work Report) showed that only the char of Run FP-11 may have been sufficiently treated to prevent agglomeration when hydrogasified. As shown in Table 2, there was a significant reduction in the volatile matter and in the hydrogen content of the char produced, compared to the feed content. The reactor residue analyses do not truly reflect the pretreatment operating conditions because these residues remained in the reactor during the cooldown period for about 18 hours.

Table 1.-OPERATING RESULTS OF THE PRETREATMENT OF MONTOUR 4 BITUMINOUS COLL IN CONTINOUS-FLOW PILOT UNIT

Run No. Sieve Size, USS Pretreatment Gas	PP-7 10/40 Nitrogen	FP-8 -10 Air	FP-9 -10 A1#	FP-10 -10 Air
Purge Gas Duration of Test, hr Steady-State Operating	+ Air Nitrogen 2	Air 2 - 1/2	Air 2	A1r 2-3/4
Feriod, min	40-113	99-159	51-123	•
OPERATING CONDITIONS				
Standpipe OD, in. Height, ft Bed Inventory, lb Reactor Pressure, psig Bed Temperature, oF	3 3 58.90 1.50	3 37.49 3.40	3 47,41 1,25	3 5 56.34 7.25
Bottom 1/2 ft 1/2 ft 1-1/2 ft 2 ft 2-1/2 ft	656 777 773 777 773	833 560 510 844	239 239 246 266 370	700 700 700 700 705
5 ft Average Coal Rate (dry), lb/hr Air Rate, SCF/hr N'trocen Rate, SCF/hr	781 756 131.4 167.9 632.5	543 678 86.4 833.6	462 304 71.2 598.8	705 702 49.6 602.8
Oxygen Concentration, mole % Nitrogen Purge Rate, SCF/hr	∓.4 3.0	21.0	sj.0	51.0
Oxygen Concentration, mole % Nitrogen Purge Rate, SCF/hr Air/Coal Feed Ratio, SCF/hb Air/Coal Bed Ratio, SCF/hr-lb Coal Bed Pressure Differential, in. Hg Coal Space Velocity, lb/cu ft-hr Coal Residence Time, min Pretreatment Gas Residence Time, min	1.278 2.851 1.88 86.31 26.9 0.0570	9.64 22.24 1.84 58.10 25.0 0.0585	8.41 12.63 1.80 47.86 40.0 0.1086	12.15 10.70 2.28 20.84 68.2 0.1565
Superficial Pretreatment Gas Velocity, ft/sec OPERATING RESULTS	0.878	0.852	0.460	0.511
Product Gas Rate, SCF/hr Residue Char (dry), wt \$ dry coal Knockout Drum Residue, wt \$ dry coal	771.2 96.0 0.3	952•8 92•5 0•1	606.0 88.8 0.6	601.2 76.8 5.8
Condenser and Filter Residue, wt % dry coal Total Residue Char, wt % dry coal Water and Other Condensates,	95.9	0.5 93.1	0.8 90.2	2.1 84.7 2.1 (Llauid)
Water and Other Condensates, wt % dry coal Overall Material Balance, % Carbon Balance, %	96.6 100.0	100.7 93.1	96.8 90.2	1.8 (Tar) 94.86 85.8d
PRODUCT GAS PROPERTIES				No Gas
Ges Composition, mole % N2 CO CO2 H2 Ar	94.0 0.99 0.99 1.99 1.89	84.1 0.7 14.5 	79.5 1.5 1.3 16.8	Samples
Total	99-99	100.04	100.03	
Heating Value, Btu/SCF ^b Nitrogen and Argon-Free Heating Value, Btu/SCF Specific Gravity, Air = 1.00	8.9 17.4 0.965	0.0 0.0 0.997	4.7 24.2 1.005	
Laboratory Agglomeration Test of Residue Evaluation 1350°F	***	Fused	Fused	Pertially
1700°F	Fused	Fused		Caked

<sup>a. Carbon in liquids not included
b. Gross, gas saturated at 60°F, 30 in. Hg pressure. SCF - dry gas volume in SCF at 60°F, 30 in. Hg pressure
c. Feed and product gases not included
d. Carbon in liquids, tar and product gas not included</sup>

Table 2 - CHEMICAL ANALYSES OF PRETREATMENT COAL FEED AND PRODUCT CHAR

	Run No. Sample Proximate Analysis, vt #	Food	Resotor Residue	Residue Receiver	Feed	PP-8 Reactor Residue	Residue Receiver	Feed	Reactor Residue	Residue Recelyer	Feed	Reactor Residue	Residue Receiver
	Moisture Volatile Matter Fixed Carbon Ash	1.0 74.0 48.8 16.2	0.3 32.0 45.1 21.0	0.7 34.3 52.6 12.4	0.9 34.3 50.5 14.3	0.3 35.7 52.8 11.2	0.5 34.1 50.5 14.9	0.7 31.8 51.9 15.6	0.1 31.6 48.1 20.2	0.3 35.6 49.7 14.4	0.4 33.1 51.5 15.0	0.1 22.7 62.0 15.2	1.1 24.2 56.6 16.1
	Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
2	Ultimate Analysis (dry), wt Carbon Hydrogen Nitrogen* Oxygen* Sifur* Aeh	67.5 4.50 — — — — 16.37	62.5	70.5 4.94 12.51	69.8 5.15 14.47	71.6 4.95	68.7 4.86 15.02	67.9 4.85	63.2	68.9 4.80 14.39	68.2 4.76 15.05	69.6 3.86 15.17	67.6 3.67 16.27
	Screen Analysis, USS, wt \$ +10 +12 +16 +20 +30 +40 +100 -100 Total	0.0 0.1 2.4 10.5 21.5 24.8 34.0 6.6	0.0 0.3 4.6 19.8 29.7 23.7 20.9 1.0	0.0 0.0 1.8 9.0 17.7 22.9 35.9 12.7	0.0 0.0 1.8 11.9 18.3 17.9 33.2 16.9	0.0 0.0 0.3 3.7 9.3 12.6 53.5 20.6	0.0 0.0 0.9 5.3 10.5 13.2 37.7 32.4	0.0 0.0 1.9 8.5 10.7 11.1 36.4 31.4	0.0 0.0 1.5 7.7 14.6 16.6 42.6 17.0	0.0 0.0 1.2 6.0 11.9 13.6 34.3 33.0	0.0 0.0 1.2 5.8 11.0 12.2 37.5 32.3	1.3 2.9 8.9 15.3 18.0 14.4 27.0 12.2	1.2 0.8 2.1 3.6 5.4 5.3 15.0 66.4

[·] Not analyzed

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - March 1965

to

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

Hydrogasification

Reassembly of the hydrogasification pilot unit was completed. Hydrogen and steam feed piping to the bottom of the reactor was installed along with gas-sampling probes for sampling gases at different levels inside the reactor. For sensing coal bed temperatures, three thermocouples were installed inside the reactor. The pilot unit was pressure tested and readied for shakedown tests.

Calibration and setting of the radiation-type coal bed-level sensor by a representative of Nuclear-Chicago, supplier of the level gage, is in progress. The use of a radiation gage for sensing solids in a small-diameter tube with a relatively large-diameter outer shell and insulation represents a novel application of the gage. The amplification unit for detecting the coal bed level has required extremely fine adjustments for sensitivity. To improve the sensitivity level, changes in the amplifier circuitry are being made.

Three hydrogasification runs were made in the pilot unit when the reassembly of the unit was completed (Runs HT-53, HT-54, and HT-55). These runs were made with the low-temperature bituminous coal char used in previous tests, and with hydrogensteam feed mixtures. The turpose of these runs was to shake down the rebuilt reactor, and to check results of the last run made in the pilot unit prior to rupture of the reactor tube.

Coal Pretreatment

Four coal pretreatment tests were conducted in the continuous-flow fluidized bed pretreater using Pittsburgh seam coal from the Ireland mine. The objective of these tests was to continue investigation for defining minimum pretreatment conditions for producing nonagglomerating char for the pilot unit hydrogasifier.

illerities as a as technology

Nominal conditions for the tests are as follows:

Run No.	Solids Rate, 1b/hr	Avg Solids Residence Time, hr	Gas Rate, SCF/hr	Oxygen,	Temp. of
FP-16	73	1.34	567	5.1	658 600
FP-17 FP-18	89 73	1.11 1.39	580 567	2.7 2.9	629 587
FP-19	68	0.77	554	9.7	706

A mixture of nitrogen and air was used as the pretreatment gas. It was found that with 3%-5% oxygen concentration in the feed gas, temperatures above 600°F could be maintained. Laboratory evaluations of the chars produced in these tests showed that the char of Run HT-16 was fused, that of Run HT-17 lightly fused, and that of Run HT-18 was free flowing, when heated to 1400°F at atmospheric pressure in a hydrogen atmosphere. Evaluation of the char of Run HT-19 has not yet been completed. Based on the above results there does not seem to be as yet a correlation between pretreatment conditions and laboratory test results. The reason may be because of fluctuation in composition of feed material. Further analysis on char and feed compositions should show this.

About a year ago, the same laboratory evaluation for agglomeration was made on four chars pretreated at 1000 psig in another reactor. These chars were found to be free flowing when criginally tested at 1050° F. They were also found to be free flowing in this test at 1400° F.

Methanation

Work continued on planning the test program and on design and ordering of major pieces of equipment. Construction work on the barricading was given top priority so that the laboratory area will be ready when the equipment arrives.

Design of service facilities for the new laboratory area was continued.

Coal Characterization

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Dynatech Corp. continued redesigning the high-temperature, high-pressure heat-of-reaction calorimeter. As was mentioned in the Methanation section above, work on construction of the barricading and design of the service facilities for the new laboratory area was continued.

Apparatus for determination of the reflectance of coal maceruls was checked out this month. Light filters for the microscope and a constant-voltage transformer for the microscope lamp were received. The constant-voltage transformer in the photometer

0 1

was found to be defective and a replacement has been requested. Tests with a borrowed constant-voltage transformer were made to "iron out" procedure and apparatus problems. On the basis of the observations made so far, it appears that the apparatus will be satisfactory.

Apparatus for determination of particle density by mercury displacement was built except for shop fabrication of a pressure vessel. Precision-bore glass tubing for determination of particle density by a gas flow method was ordered; this will be used on particle sizes smaller than about 80 mesh.

Process Concept Development

Work has involved developing a mathematical model describing the state-of-the-art of coal partial oxidation or suspension gasification processes. This work to aid the economic study is in preparation for estimating operating parameters when using spent char from the hydrogasifier as feed for these processes.

Economic Evaluation

In preparation for design of the hydrogen section of the pipeline gas plant, a study is being made of synthesis gas production by suspension gasification of coal. Contacts with Texaco Corp. and Babcock and Wilcox Co. will be made for state-of-the-art information. These two companies have developed pressurized gasification processes (300-400 psig). Gasification at atmospheric pressure would not be economical for our process because of the need to compress hydrogen to 1000 psig. A study of basic gasification reactions is being made.

Fluidized-bed gasification will also be considered. Spent char from hydrogasification would be a good feed for this process since there would probably be little tendency toward agglomeration. The fluidized gasification process has some advantages over suspension gasification in the operation at nonslagging conditions, possibly giving higher yields per unit of reactor volume. However, carbon gasification efficiencies would probably be lower for fluidized operations.

No inventions were made during the month of March; however, we are now reviewing our work to establish any patentable aspects of the process as it is presently envisioned.

Jack Huebler, Associate Director

Trank Schora, Manager

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DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - April 1965

to

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

Hydrogasification

Analytical results have been completed for Run HT-53 - the first test made last month in the rebuilt hydrogasification reactor. A low-temperature bituminous coal char was hydrogasified in this run at 1000 psig and an average reactor temperature of 1485°F, with a hydrogen-steam mixture containing 36 mole percent steam and a hydrogen/char ratio 41 percent of stoichiometric. Results show that 52% of the carbon was gasified, and 41% of the steam was reacted.

Five hydrogasification tests (Runs HT-56, HT-56b, HT-56c, HT-57, and HT-57b) were attempted in the pilot unit to gasify a lightly treated Pittsburgh seam (Ireland mine) bituminous coal. These tests are being run to establish the minimum amount of pretreatment required for satisfactory operation in the 4-in. reactor. The pretreated coal was prepared in the pilot plant pretreater in Run FP-15 (February 1965 Report) and was shown to be nonagglomerating based on our laboratory test.

Three of these tests lasted only up to 7 minutes, as difficulties were encountered in feeding the coal. The coal feed tube eventually became plugged at the screw. To correct this problem, the feed tube was enlarged from 0.4 in. to 1.0 in. ID, and was shortened to keep the discharge end somewhat cooler. After these changes, two hydrogasification tests were run for 34 and 27 minutes. These tests were terminated when the coal eventually bridged in the 4-in. reactor tube, and the coal level built up to choke the coal feed tube. Product gas samples were taken during these tests; a partial evaluation will be made when analytical results of the tests are completed.

Coal Pretreatment

Two coal pretreatment tests were conducted in the continuous fluidized-bed pretreater with a Pittsburgh seam coal from the Ireland mine. These tests continue the study for defining minimum pretreatment conditions necessary for producing a nonagglomerating char for the pilot unit hydrogasifier.

INSTITUTE OF BAS TECHNOLOG

Nominal conditions for these tests are as follows:

Run No.	Solids Rate,	Avg Solids Residence Time, hr	Gas Rate, SCF/hr	Oxygen,	Temp,
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Pretreatment gas was a mixture of nitrogen and air. These tests were designed to increase the degree of pretreatment over that in earlier tests by using a higher temperature and an exygen concentration of 10%. Laboratory evaluations of the chars for agglomerating characteristics have not yet been completed. A laboratory evaluation showed the char produced in Run FP-19 (March 1965 Report) at 706°F to be nonagglomerating.

The pilot plant swing hammer mill, which pulverizes coal for the pretreater, has been modified to produce a smaller fraction of fines. The speed of the mill was reduced and the screen removed.

Methanation

With construction of the barricading nearly completed, work was begun on the instrument panels. Nearly all the major pieces of instrumentation were received.

Design of service facilities for the new laboratory area was completed.

Coal Characterization

4 1 1 1 1 1 1

The high-temperature, high-pressure heat-of-reaction calorimeter redesign was completed by Dynatech Corp. Barricading for the calorimeter was completed.

Construction and assembly of apparatus for determination of particle density by mercury displacement was completed. Determinations on the coarser sieve fractions (100 mesh and larger) of feeds and residues from several free-fall runs were started.

Development of procedures for determination of reflectance of coal macerals was continued. Determination on an area of only 2.8-micron diameter appears feasible with the addition of a recorder or output meter having somewhat greater sensitivity than that which the instrument now provides. This will make it possible to read reflectance on vitrinite and other macerals in attrival coal grains that have only small areas of uniform reflectance.

Process Concept Development

During the month integration of presently developed kinetic equations into the process model was begun, the purpose of which is directed to improve the accuracy of the model in predicting process operation.

Economic Evaluation

Texaco has been contacted about their gasification process for making hydrogen. They indicated that it may be possible to operate at 1000 psig.

A study is under way to determine the effect of the hydrogasifier feed composition on the external hydrogen required.

An economical evaluation of the various methods of controlling the temperature for methanation is also in progress.

No inventions were made during the month. The work to establish patentable aspects of the process is continuing.

Jack Highler, Associate Director

Frank Schora, Manager

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DEVELOPMENT OF 19TH HYDROGASIFICATION PROCESS

Progress Report - May 1965

to

Office of Coal Research Contract No. 14-91-0001-381

Hydrogasification

Seven hydrogasification tests (Runs HT-58, HT-59, HT-60, HT-61, HT-61b, HT-61c, and HT-62) were attempted in the balanced-pressure pilot unit to gasify lightly treated Pittsburgh seam bituminous coal. Coal from the Montour 4 mine was used in Run HT-58; the other tests used coal from the Ireland mine. The Montour 4 coal was pretreated at 1000-psig pressure in tests conducted in the early part of 1964. The Ireland mine coal was pretreated in the fluidized-bed pilot plant pretreater at atmospheric pressure (Runs FP-22, FP-23, and FP-24). These pretreated coals were non-agglomerating according to laboratory test.

Two of the hydrogasification tests were completely successful, one with coal char in free-fall, and one with a moving char bed. In these tests, no operational difficulties were experienced in feeding the coal char or in discharging the residue. Three of the tests were only partially successful. After falling through the heated reactor tube, the coal bridged in the lower end of the reactor tube below the furnace and could not be discharged. The bridging probably resulted from steam condensation rather than from stickiness of the coal char. Two of the tests had to be terminated shortly after they were started due to difficulties in the operation of the reactor-shell differential control system. The trouble was traced to a pneumatically operated control valve that was sticking. The faulty valve was replaced. Evaluation of the successful hydrogasification tests will be made when analytical results of the tests are completed.

Coal Pretreatment

Three coal pretreatment tests were conducted in the fluidized-bed pretreater with a Pittsburgh seam bituminous coal from the Ireland mine. The primary objective here was to produce a nonagglomerating char for the pilot unit hydrogasifier and, at the same time, study pretreatment conditions.

Nominal conditions for these tests are as follows:

Run No.	Solids Rate, 1b/hr	Avg Solids Residence Time, hr	Gas Rate, SCF/hr	Oxygen,	Temp,
FP-23 FP-24	<i>3</i> 5	1.50	597	9.6	764
	34	1.50 1.89	830	9.2	738
FP-25	37	1.86	760	9.8	749

A mixture of nitrogen and air was used for pretreatment. The solids rate was reduced to half of that of previous tests in order to increase the coal residence time and thus increase the degree of pretreatment. Laboratory evaluations at 1400°F show the chars produced to be nonagglomerating. The chars of Runs FP-23 and FP-24 were used as feed to the pilot unit hydrogasifier.

Fluidization tests were conducted in a bench-scale glass tube to study the fluidization characteristics of 10 to 40 mesh and -10 mesh coal, and to define more accurately minimum fluidization rates. These tests are to provide information for improved operation of the pilot plant pretreater.

Methanation

The methanation reactor test program was formulated in a meeting with Dr. James Carberry, consultant to the Institute on this work. The barricade and instrument panel structures were completed for the test program. The reactor and most of the instrumentation were received.

Cosl Characterization

Dynatech Corp. continued construction of the heat-of-reactor calorimeter. The drop calorimeter has been essentially completed; the instrument console is being constructed.

Characteristics required of a recorder to be used with the reflectance apparatus in the coal petrographic work were determined. Feeds and products of pretreatment Runs FP-4A and FP-8 were analyzed petrographically. No significant difference between feed and product was evident in either run. Small differences between the two feeds, one of Montour 4 coal and the other Ireland mine coal, were found. Particle densities of two sieve fractions of the feed and residue of a free-fall hydrogasification run were determined.

Process Concept Development

The work on integrating the kinetic equations for the two stages of hydrogasification into the process model is continuing. Although the kinetic equations describing the hydrogen char and steam-char reactions will undergo modification as the operation of

-2-

the pilot unit continues, integration of the kinetics into the process model will provide significant insight into the variation in equipment size with various operating parameters.

Economic Evaluation

During the past month the study to determine the effect of hydrogasifier feed composition on the required amount of external hydrogen has been completed. A study has now been undertaken to determine the total carbon conversion that will eliminate the production of excess spent char.

Determinations have been started on the design criterion for the pretreatment section to be used in the state-of-the-art design.

Gasification requirements for hydrogen production from spent char have been discussed with the Texaco Development Corp. Char partial combustion appears feasible. Texaco has given us tentative synthesis composition and reactant requirements. We have calculated synthesis gas costs as affected by oxygen/carbon and steam/carbon ratios.

No inventions were made during the month. Some problems on delivery of the special heat-of-reaction calorimeter are anticipated. Dynatech Corporation informs us that, due to problems in nickel plating one of the large enclosures, the delivery date of July 1 might not be met. We plan to keep in close touch with Dynatech to expedite this item.

Jack Ruebler, Associate Director

Frank Schora, Manager

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DEVELOPMENT OF IGT HYDROGASIFICATION PROCESSION RESEARCH Progress Report - June 1965

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Office of Coal Research
Contract No. 14-01-0001-381 DEPT OF THE INTERIOR

Hydrogasification

Seven hydrogasification tests (Runs HT-63 to HT-69) were conducted in the balanced-pressure pilot unit to study the gasification of lightly pretreated Pittsburgh seam bituminous coal from the Ireland mine. The coal was pretreated in the fluidized-bed pilot plant pretreater with nitrogen and air at 750°F (Runs FP-25, 27, 28, and 29). When tested in the laboratory at conditions simulating hydrogasification operation, these pretreated coals were found to be nonagglomerating.

The partially hydrogasified residue from one of the tests conducted at 1300°F was used as a feed for a following hydrogasification test designed to simulate the high-temperature reaction zone of a two-stage hydrogasification process. The test was conducted at a nominal temperature of 1700°F with a hydrogen-steam feed gas containing 50 mole percent steam. Approximately 20% of the carbon in the already partially hydrogasified feed was gasified. A product gas having a heating value of 414 Btu/SCF on a nitrogen-free basis was produced.

In four other tests, the low-temperature reaction zone of a two-stage hydrogasification process was simulated. Lightly pretreated Ireland mine bituminous coal was reacted at 1300°F with feed gas of the following composition: hydrogen, 45 mole percent; methane, 25 mole percent; and steam, 30 mole percent. This composition approximates that of a gas leaving the high-temperature zone. Two of these tests lasted only 30 minutes, as coal caked at the top of the reactor tube and plugged it. The feed coal for these tests, pretreated in Run FP-27, apparently did not have its agglomerating characteristics entirely destroyed in pretreatment. In the other two tests, pretreated coal from Runs FP-28 and FP-29 was used. Both tests were completely successful, lasting 8 hours. Bed-level control was ideal, and the coal did not cake. To develop data for studying reaction rates within the coal bed, product gases were sampled in these two tests at levels of 5, 18, and 42 inches below the top of the 7-ft bed.

The partially hydrogasified residue from the two successful, simulated low-temperature tests was hydrogasified in another test simulating the high-temperature zone of a two-stage operation. The coal feed rate was reduced in proportion to the

fraction of coal already converted to approximate the rate at which coal would be entering the high-temperature zone. The coal was reacted at 1700°F with a hydrogen-steam feed gas containing 50 mole percent steam. Approximately 20% of the remaining carbon in the partially hydrogasified feed was converted in this second-stage test.

Components have been machined for adapting the hydrogasification reactor to fluid-bed operation. These include a feed gas distributor and a sectional support tube for positioning the gas distributor at different levels within the reactor. A reactor head assembly was machined to accommodate an internal cyclone which will be necessary for gas-solids separation in fluid-bed operation. The head assembly also has accommodations for a coal feed tube, product gas off-take tube, gas sampling probes, and thermocouples.

Fabrication of larger capacity coal feed hopper and residue receiver vessels for the hydrogasification pilot plant is proceeding in the IGT shop. Major welding of the residue receiver has been completed. Hydrogasification tests at higher coal feed rates and of longer duration will be possible when these larger vessels are installed.

Coal Pretreatment

Four pretreatment tests were made in the pilot unit fluidized-bed pretreater using a Pittsburgh seam bituminous coal from Consolidation Coal Co.'s Ireland mine. The objectives of these tests were to produce a nonagglomerating char for the pilot hydrogasification unit and to study minimum pretreatment requirements and conditions.

Nominal conditions for these runs are as follows:

Run No.	Solids Rate, lb/hr	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc, %	Bed Temp,
FP-26 FP-27 FP-28 FP-29	51 51 54 54	1.73 1.85 1.61	987 1325 537 609	9.1 9.8 9.9 9.9	731 719 744 756

An air and nitrogen mixture was used as the feed gas. The coal feed particle size for Runs FP-26 and FP-27 was -10 + 40 mesh. Fluidization was erratic with this particle size distribution even at substantially increased feed gas rates. Because of plugging in the reactor the computed average solids residence time for Run FP-27 was unreliable. The coal feed particle size for

Runs FP-28 and FP-29 was changed to -20 mesh and -15 + 80 mesh, respectively. The standpipe for these runs was increased from 5 ft to 7 ft to prevent solids residence time from decreasing due to greater bed expansion of the smaller sized particles. There were no operational difficulties during either of these tests. Laboratory agglomeration tests showed FP-26 char to be fused and FP-27 char to be lightly caked, whereas FP-28 and FP-29 chars were nonagglomerating and were successfully run in the pilot unit hydrogasifier.

Coal Characterization

The product from pretreatment Run FP-22, which was used successfully as feed for hydrogasification, was examined petrographically. Substantial modification of the coal, with appearance of vesicles, disappearance of eximite, increase in the reflectance throughout the particle, and appearance of a skin of still higher reflectance, was observed of many but not all particles.

A comparison of our reflectance determination with that at Bituminous Coal Research, Inc. was made on two coal samples. Reasonably good agreement, within 0.02% on a high-volatile coal and 0.04% on a low-volatile coal, was obtained.

Apparatus is being constructed for determination of particle density by the Ergun gas flow method.

Dynatech Corp. continued construction of the heat-of-reaction calorimeter. The drop calorimeter and the instrument console have been completed.

Methanation

Instruments for the methanation reactor were installed and are being tested. Water and air supplies and drain facilities were installed in the barricade.

During the month of June, no inventions were made in the course of this work. To ensure maximum efficiency in the pilot plant work, the pilot plant program will be suspended for the first two weeks in July for vacation purposes.

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Approved			Signed .	it is the
Jack Huebler,	Assocluta	Director	Frank C.	Schore, Manager

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DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS

Progress Report - July 1965

to

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

Hydrogasification

Two hydrogasification tests (Runs HT-70 and HT-71) were conducted in the balanced-pressure pilot unit to study the gasification of lightly pretreated Pittsburgh seam bit maintenance coal from the Ireland wine. The coal was pretreated in the fluidized-bed pilot plant pretreater with nitrogen and air at 750°F (Runs FP-28, FP-29, and FP-31). These pretreated coals were found to be nonagglomerating when tested in the laboratory at conditions simulating hydrogasification operation.

Both hydrogasification tests were conducted at similar conditions to simulate the upper, low-temperature reaction zone of a two-stage hydrogasification process, and to produce a sufficient quantity of partially hydrogasified coal to use as feed for a high-temperature hydrogasification test. The lightly pretreated bituminous coal was reacted at 1300°F with a feed gas containing \$\frac{1}{2}\$5 mole percent hydrogen, 25 mole percent methane, and 30 mole percent steam. This gas is typical of one that would be leaving the lower, high-temperature hydrogasification zone. The two tests were completely successful; each lasted about 6 hours and hydrogasified about 150 lb of coal. Product gases also were sampled in these tests at levels of \$\xi\$, and \$42\$ inches below the top of the 7-ft coal bed to develop data for studying reaction rates within the coal bed.

Installation of a feed gas distributor inside the hydrogasification reactor was started. The gas distributor is a 3-inch, porous, stainless steel disk that will also set as a coal bed support plate to keep the bottom of the coal bed well within the heated portion of the reactor tube. Installation of the gas distributor is necessary for fluid-bed hydrogasification operations.

To facilitate the handling and conveying of high-moisture-content coals after being crushed, a used, rotary-type dryer was purchased. The gas-fired dryer has a 12-ft-long double shell, the inner shell having a 17-inch ID. It will be installed out-doors for drying coals used in pretreatment and hydrogasification tests before they are ground in the hammer mill.

Coal Pretreatment

NSTITUTE

Four pretreatment tests were conducted in the fluidizedbed pretreatment pilot unit. A Pittsburgh seam bituminous coal from Consclidated Coal Company's Ireland mine was used as the feed in the first two runs (FP-30 and FP-31). The purpose of these tests was to continue providing a nonagglomerating char for the pilot hydrogasification unit. Laboratory agglomeration tests showed the chars to be free-flowing, and both were successfully tested in the hydrogasification unit.

In the latter two tests (FP-32 and FP-33), the study of the Broken Arrow mine coal was initiated. It is a high-volatile bituminous coal like the Pittsburgh seam coal but of a slightly higher rank. The coal was acquired from the Ohio No. 6 seam (Middle Kittanning) and is one of the seven coals selected to be evaluated in the hydrogasification project. The purpose here is to study minimum pretreatment conditions and to supply the pilot hydrogasification unit with nonagglomerating char.

A typical proximate analysis of the Broken Arrow coal is as follows:

Moisture, 😤	Volatiles, 5	Fixed Carpon, %	Ash. %	Heating Value, Btu/lb
4.0	39.0	51.6	5.4	12,400

Nominal conditions for the four tests are as follows:

Run Mo.	Solids Rate, lb/hr	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc. 5	Bed Temp,
FP-50	19.5	5.23	598	10.1	756
FP-51	18.2	5.80	584		750
FP-52	28.4	2.56	5 89	10.5	702
FP-55	28.2	2.01	630	10.2	749

An air and nitrogen mixture was used as the pretreatment gas. Coal feed particle size was -16, +80 mesh for the four runs. Run FP-30 was terminated when solids plugged the cyclone, thus restricting the gas flow from the reactor. The remaining three tests operated successfully and were voluntar of terminated.

Laboratory agglomeration tests showed the char from Pun FP-32 to be caked, whereas char from Run FP-33 was free-flowing. These results illustrate the effect an increase in bed temperature has on the severity of pretreatment.

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Ccal Characterization

Problems of mounting pretreated coal, which consists largely of relatively fragile hollow spheroids, were investigated. An apparatus and procedure for mounting the coal without particle breakage by attrition or by application of high pressure was devised. A low-power oil immersion objective was received with which photographs of whole particles so mounted were made.

The behavior of minoral constituents of the Ireland and during pretreatment was investigated. The coal contains about 3 volume percent of shale particles unassociated with al: these tend to accumulate in the reactor, as shown by a 12 volume percent content found, by petrographic examination, in the reactor residue from Run FP-31. Coal particles high in pyrite also concentrate in the reactor. About half of the pyrite in the Run FP-31 reactor residue had reacted with oxygen to form a product tentatively identified as magnetite.

Dynatech Corp. continued construction of the heat-of-reaction calorimeter. Most of the parts have been completed and are being assembled. The water, drain, air, and vent facilities have been installed in the barricade area.

Methanation

The inserts for the methanation reactor were fabricated and installed. Instruments were installed and tested.

State-of-the-Art Design

INSTITUTE

During June and July, the following was accomplished:

The pretreatment, hydrogasification, and methanation sections were designed. Drawings were given to Chicago Bridge & Iron Company for estimating the cost of vessels for these sections.

Discussions were held with engineers of The Lummus Co., the firm recommended to us by Texaco. They have made an estimate of capital and operating costs for producing the process hydrogen by partial exidation of spent hydrogesification char.

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

Approved Associate Director Fr

Frank C. Schore, Menager

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OFFICE OF COAL RESEARCH

DEVELOPMENT OF TOT HYDROGASTFICATION PROCESS

Progress Report - August 1965

969 SET 28 FR 3 FG

Office of Coal Research Contract No. 14-01-0001-381 BEFT OF THE INTERIOR

Everopseification

Three hydrogasification tests (Runs HT-72, HT-73, and HT-74) were conducted in the balanced-pressure bilot unit to study the gasification of lightly pretreated bituminous coal. Pittsburgh seam bituminous coal from the Ireland mine, partially hydrogasified in Runs HT-70 and HT-71 (July 1965 Progress Report), was the feed in the first two tests. Feed in the Hird test was a pretreated Onio No. 6 seam bituminous from the Broken Arrow mine. This coal, supplied by Peabody Coal Company, was pretreated with nitrogen and air at 750°F in the IGT fluidized-bed pilot plant pretreater (Runs FP-32, FP-33, and FP-34). When tested in the laboratory at conditions simulating hydrogasification operation, the pretreated coal was found to be nonagglomerating.

Runs HT-72 and HT-73 were conducted to similate the lower, high-temperature reaction zone of a two-stage hydrogasi-fication process. Partially hydrogasified coal was reacted at 1850 F in Run HT-72 with a hydrogen-steam mixture containing 1850°F in Run HT-72 with a hydrogen-steam mixture containing 40 mole percent steam, and a hydrogen/coal ratio 37% of the stoichiometric ratio. In Run HT-73 partially hydrogensified coal was reacted at 2000°F with a hydrogen-steam mixture containing 20 mole percent steam, and a hydrogen/coal ratio 42% of stoichiometric. Both tests were conducted at coal feed rates of 15 lb/hr in a 7-ft moving bed. The bottom of the coal bed was located at about the midpoint of the reactor tube, the coal resting on a gas distributor disk. The two tests were successful, one lasting 5-1/2 hours, the other 4-1/2 hours. To develop data for studying reaction rates within the coal bed, product gases were sampled in these tests at levels of 5, 18, and 42 inches below the top of the 7-ft coal bed.

Run HT-74 was the first hydrogasification test in which a pretreated this No. 6 seam high-volatile bituminous coal was processed. The test was designed to simulate the upper, low-temperature reaction zone of a two-stage hydrogasification process, and to produce partially hydrogasified coal for a subsequent high-temperature hydrogasification test. The coal was reacted at 1300°F with a feed gas containing 45 mole percent hydrogan, 25 mole percent methans, and 30 mole percent steam. The test had to be terminated after about 2 hours and before steady-state operation was reached because of difficulties in discharging the coal.

) N S t i t u t k

Small pieces of caked coal lodged in the annulus between the reactor walls and the gas distributor disk, which kept coal from flowing.

The feed gas distributor described in the July 1965 Progress Report was installed in the hydrogasification reactor tube. This distributor will enable more uniform feed gas distribution for fluid-bed operations, as well as move the bottom of the coal up into the midpoint of the reactor tube, which will bermit better temperature control. Operability of the gas distributor system was demonstrated in the three tests cited above.

Coal Pretreatment

Six pretreatment tests were conducted in the fluidizedbed pretreatment pilot unit. The feed was Broken Arrow mine bituminous coal from the Ohio No. 6 seam (Middle Kittanning). The objective of these tests was to study minimum pretreatment conditions and to supply the pilot hydrogasification unit with nonagglomerating char.

Nominal conditions for the six runs are as follows:

thin No.	Solids Rate, 1b/hr	Avg Solids Res.	Feed Gas Rate, SCF/hr	Oxygen Conc.%	Bed Temp,
#P-34 FP-34A	29.9	1.92 1.82	591 590	5.5	737 744
FP-35A	49.7	1,31	654 644	21.0	747 749
FP-35B FP-35C	* 56.3	* 1.94	570 574	21.0 21.0	650 634

* Not calculated because of erratic coal feeding caused by reactor plus

In the first two tests, an air and nitrogen mixture was used as the pretreatment gas. Run FP-34 had to be terminated before steady-state conditions were attained, due to solids plugging the cyclone in the product gas offtake line. Run FP-34A (made under the same conditions as FP-34) was terminated after 4 hours when the cyclone began to plug. However the test was long enough to obtain a residue char representative of steady-state conditions. Imboratory agglomeration tests showed the char to be free-flowing.

For the FP-35 series of tests, air was used as the pretreatment gas. These tests were made to investigate the oxygen breakthrough, if any, due to the higher oxygen concentration in the feed gas, under similar bed temperatures, gas and coal feed rates, and solids residence times. Considerable difficulty was encountered in trying to control the bed temperatures in the range desired (700°-750°F), due to the increased exothermic reaction contributed by the higher oxygen/coal feed ratio. Runs FP-35, 35A, and 35B were terminated before steady-state conditions were reached, due to erratic bed temperatures and subsequent plugging of the reactor. Investigation of the reactor following these runs showed the bottom portion to be plugged with a solid coke-like formation. From these attempts it was found that the exothermic reaction would continue even with the external heat supply to the reactor turned off at 600°-650°F. During Run FP-35C, the heatup rate to the reactor was reduced in an effort to prevent plugging. By discontinuing the external heat, the bed temperatures were controlled in the 650°F range without upsetting the operation. However, analytical analysis of the residue char and mass spectroscopic analysis of the product gas gave evidence of very slight pretreatment in this temperature range. Also, laboratory agglomeration tests of the residue char showed that it was agglomerating.

In view of the results of these tests it was decided to continue pretreatment studies with air and nitrogen mixtures as the feed gas in order to investigate minimum pretreatment requirements and to prepare nonagglemerating char for the pilot hydrogasification unit.

A newly designed cyclone, which will facilitate cleaning, is being fabricated in the IGT shop to alleviate the problem of solids plugging.

Methanation

Mixing tests with standard gases were carried out and calibration curves for the CO and CO2 analyzers were constructed. Preparation for feed gas storage and transfer was completed. The feed gases are being prepared by The Matheson Company, Inc.

The purpose of the first series of tests to be conducted in the reactor will be to establish which combinations of feed rates and basket speeds will provide perfect mixing.

Coal Characterization

N S t i t U t k

Dynatech Corp. is nearing completion of the heat-of-reaction calorimeter; it will undergo shakedown operation in September.

Reflectance standards for determination of reflectance of chars and hydrogasification residues are being investigated. These products have higher reflectances than coal and the standards developed for use on coal.

Engineering Economics Studies

Process design and cost estimates for the state-of-theart pipeline-gas-from-coal plant continued. Designs for the hydrogen and prepurification sections were completed; those for utilities and process heat recovery were partially completed. All tables for material balances and the overall flow diagram of the block flow process, with gas stream compositions, have been finalized. Drawings of other sections are in various stages of preparation.

Three patent disclosures were made during this period. The first concerns improvement in the fluidized-bed pretreatment operation. To reduce the residence time and the degree of pretreatment, while retaining the desired heat transfer characteristics of a fluidized bed, it is suggested that a two- (or more) stage fluidized bed be considered in which both stages are at the pretreatment temperature and pretreatment gas is introduced to each. Alternatively, a fluidized bed with tapered walls should reduce axial mixing of solids and reduce back mixing.

The second disclosure pertains to pretreatment of coal in a slurry form. By pumping the coal as a slurry, costly lock hoppers for solids introduction to the high-pressure gasifier can be eliminated. Preliminary experiments have shown that a free-flowing coal can be obtained by pretreating it at 1000 psig, 500°F with oxygen in a water slurry.

The third disclosure deals with a device for dewatering the coal slurry prior to its introduction into the gasifier. Reducing the amount of water added with the coal would reduce both the quenching effect on the gasifier effluent and the loss of high-availability heat. The dewatering unit consists typically of a conical section that prevents the dewatered coal from bridging. This coal can then be fed, for example, by a screw fander into the desifier. feeder into the gasifier.

As a result of an acute illness of our code welder, some delay has been encountered in modifications for converting the hydrogasifier reactor for fluidized-bed operation. Attempts were made to obtain outside help but welders with the desired capabilities are in short supply. Our welder will return to work in September. Because of the delay, we have been undertaking moving-bed runs with Ohio No. 6 coal rather than going to fluidized operation. Since Ohio No. 6 coal is to be investigated as a hydrogasifier feed, no time has been lost in the overall program.

Author Signed Approved Jack Hueber, Associate Director

PROGRAM FOR DEVELOPMENT OF 19t OCR: Contract No. 14-01-0001-381 PHÁSE I L call Probabilities ek: ka Product material requires for nydrogasification rups d. Coli Chiristenia MASE # A. Antigates or Benfariableste basis AND IN SEC. Æ: a. Designates Datas Dides Manife. TOTAL COSTS TO OUR CHICLUMES ON THE HHA 21.50 21.50 29,380 27,880 拉爾 拉鍋 拉糖 拉鄉 34,166 24,386 34,786 21,786 10,000 15,000 25,000 16,500 25,000 21,000 15,000 25,000 25,000 15

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SFFICE OF COAL RESEARCH

DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS RECEIVED

Progress Report - September 1965

1965 GOT 25 AT 10 45

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

DEPT OF THE INTERIOR

Hydrogasification

Study of the hydrogasification of lightly pretreated bituminous coal in the balanced-pressure pilot unit continued with six tests (Runs HT-75, HT-76, HT-77, HT-78, HT-79, and HT-80). Ohio No. 6 seam bituminous coal from the Broken Arrow mine, pretreated in Runs FP-32, FP-33, and FP-34, was the feed in the first three tests. Feed for Run HT-78 was a Pittsburgh seam bituminous coal from the Ireland mine, pretreated in Run FP-37. The partially hydrogasified residue from Run HT-78 was used as the feed for Runs HT-79 and HT-80.

Run HT-75 was designed to simulate the upper, low-temperature reaction zone of a two-stage hydrogasification process, and to produce partially hydrogasified coal for a subsequent high-temperature hydrogasification test. Pretreated Chio No. 6 seam coal was reacted at 1300°F with a feed gas containing 45 mole percent hydrogen, 25 mole percent methane, and 50 mole percent steam. The test ended before steady-state operation was reached when the coal in the reactor could not be discharged past the gas distributor disk. The annulus between the reactor walls and the gas distributor disk, through which the coal must flow, became clogged with flaked pieces of caked coal. This caked coal formed on the upper walls of the reactor tube in the previous high-temperature runs and flaked off during Run HT-75.

Runs HT-76 and HT-77 were conducted at the same conditions as HT-75, and used the same coal feed. Because of the difficulties in discharging coal past the gas distributor disk in Runs HT-74 and HT-75, the disk was not used in Runs HT-76 and HT-77. The feed gas was fed through a previously used 3/8-inch gas feed tube that extends 16-1/2 inches above the bottom of the lower furnace section. Both runs were successful. Run HT-76 lasted 4-1/2 hours and was terminated because of a gas leak that developed at the screw feeder packing gland. Run HT-77 lasted about 5 hours, and was terminated when the coal feed was exhausted. The partially hydrogasified residues from these tests will be combined for use as a feed in a later high-temperature test.

In Run HT-78, lightly pretreated Pittsburgh seam coal. was hydrogasified at 1300°F with a feed gas of the same molar composition as in Runs HT-76 and HT-77. To develop hydrogasification data at low hydrogen/coal stoichiometric ratios, the test

was conducted at a hydrogen/coal ratio 28% of stoichiometric. Over 125 lb of coal was processed in this test in 7 hours.

The partially hydrogasified residue of Run HT-78 was the feed for Run HT-79. Conducted at 1700°F, this test was designed to simulate the lower, high-temperature zone of a two-stage hydrogasification process. Feed gas was a hydrogen-steam containing 50 mole percent steam, fed through a gas distributor disk positioned half-way up the reactor tube. The disk also supported the bottom of the coal bed. The coal bed height was 5-1/2 feet so that operating results could be compared with similar tests conducted in a 7-foot bed. The run was terminated after 50 minutes when a plug formed in the product gas line upstream of the filter. Solids lodged in the line following an uncontrollable gas surge in the reactor shortly after steam was introduced to the reactor.

Run HT-80, a repeat of HT-79, was conducted at similar conditions, using the same feed. The test was successful, and was terminated after 4-1/2 hours when the coal feed supply was exhausted.

Fabrication of the increased-capacity coal feed hopper was resumed upon the return of our code welder. Welding of the hopper and reactor head assembly are nearly completed. X-rays of the welds and stress relieving of the hopper have been scheduled.

Coal Pretreatment

NSTITUTE

Three pretreatment tests were conducted in the fluidizedbed pretreatment pilot unit. The objective of the tests was to study minimum pretreatment conditions and to supply the hydrogasificiation pilot unit with nonagglomerating char.

Nominal conditions for the three runs are as follows:

Run No.	Solids Rate, lb/hr	Avg Solids Res	Feed Gas Rate, SCF/hr	Oxygen Bed	i Temp,
FP-36 FP-37	55•4 45•0	1.07 1.26	596 605	9.6	722 744
FP-38	58.3	1.18	609	9.8	723

The feed for Runs FP-36 and FP-38 was a -16, +80 mesh Broken Arrow mine bituminous coal from the Ohio No. 6 seam (Middle Kittanning). An air-nitrogen mixture was used as the pretreatment gas. Both tests were operationally successful and were terminated voluntarily. Imboratory agglomeration tests of the residue char from FP-36 showed that it was lightly caked, indicating that pretreatment was not severe enough to ensure a free-flowing feed for the hydrogasification pilot unit. However, since

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TECHNOLOGY

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the char was only lightly caked, minimum pretreatment conditions were approached. FP-38 was run under similar conditions to compare results of the residue char with that of FP-36. Laboratory agglomeration tests showed that the char was also caked. Therefore, the conditions of the two tests were inadequate for producing a free-flowing char.

Test FP-37 was run with Ireland mine bituminous corl from the Pittsburgh seam. Coal feed particle size was -16, +80 mesh. The test was conducted to supply the hydrogasification pilot unit with Ireland mine char for testing in the modified reactor. An air-nitrogen mixture was the pretreatment gas; the oxygen concentration was 9.6%. The run was successful and was terminated when sufficient char for the hydrogasification tests was obtained. Imboratory agglomeration tests showed that the char was free-flowing; this was verified by a successful run in the hydrogasification unit.

The new cyclone for the pretreatment pilot unit has been received and is in the process of being installed in the product gas offtake line.

Coal Characterization

The heat-of-reaction calorimeter is being calibrated and undergoing acceptance tests at 1500 psig and 1500°F at the Dynatech Corp. facilities in Cambridge, Mass. The calibration and testing procedure includes drop heater, alumina, and nedecane tests. The drop calorimeter is being calibrated with alumina.

The carbon monoxide alarm was installed in the laboratory. The combustible gas detector is presently being installed.

Use of strontium titanate as a reflectance standard for hydrogasification residue samples is being investigated in the petrography program. Large single crystals of this material are obtainable commercially. Petrographic examination of residues from both high- and low-temperature hydrogasification runs was started.

Flowmeters in the apparatus for determination of particle density by the Ergun gas flow method were calibrated. Determination of the particle density of sieve fractions of the feed to free-fall hydrogasification Run No. HT-60 was started.

Methanation

INSTITUTE

The continuous-flow, stirred-tank catalytic reactor was tested to establish the ranges of flow rates and catalyst basket speeds for which "perfect mixing" of the gas phase is realized.

Calibrations were prepared for the various measuring devices. Catalysts are being obtained from several suppliers in various formulations and sizes. The test program has been finalized and methanation tests are now being initiated.

State-of-the-Art Design and Evaluation

The state-of-the-art design and evaluation was completed and sent for final editing and printing.

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

Jack Huelder, Associate Director

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Frank C. Schora, Manager

PROGRAM FOR DEVELOPMENT OF IG OCR: Contract No. 14-01-0001-38 4.. Dec 110 Dec PHASE I Establish rash-controlling areas of operation E - 13 7 - 36 DS - \$15,800 ODC - \$5,500 A. Hotelas de Arion E - 9 7 - 27 05 - 511 300 000 - 52,000 CS - \$12,500 CDC - \$3,000 B. Call Patholipson Produce material requires for hydrogasitication runs E - 5 T - 12 DS - 14,850 ODC - 51,250 E - 7 T - 8 DS - \$3,800 OOC - 8 Establish that and d E 13 T - 8 DS - \$8,400 ODC - \$20,500 PRASE II Analyze and corrèlate data from hydrogas-fication, coal prévidanders, and méticangtion dans, and recommend conditions for additional bass. Dévideo à mêtic deuteurs to use in diactor design MODELS OF ESSENTIABLES DAVIS E - 23 7 - 34,289 00C - \$1,560 c. Réfeter Désign Studiés d. Econodic Entlution E-4 05-32.000 00C-6 PRASE III Proliminary Print Plant Driven TOTAL COSTS TO OCR (INCLUDES OVERHEAD AND FEE) 855,44D 23,50 22.40 21,780 23,300 23,000 27,600 27,460 26,160 26,160 23.300 10,000 15,000 23,300 14,500 23,000 21,100 19,800 28,800 23,400

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DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS DEFICE OF COAL RESEARCH

Progress Report - October 1965

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Office of Coal Research Contract II. 14-01-0001-381

DEPT OF THE INTERIOR

Hydrogasification

INSTITUTE

Four hydrogasification tests (Runs HT-81, HT-82, HT-83, and HT-84) were conducted in the balanced-pressure pilot unit in a continuation of the study of the hydrogasification of lightly pretreated bituminous coals. Partially hydrogasified Ohio No. 6 seam bituminous coal (Broken Arrow mine) from Runs HT-76 and HT-77 was the feed in Run HT-81. Pittsburgh seam bituminous coal (Ireland mine), pretreated in Run FP-37, was the feed in Runs HT-82 and HT-83; that pretreated in Run FP-43 was the feed in Run HT-84.

Run HT-81 simulated the lower, high-temperature reaction zone of a two-stage hydrogasification process. Onio No. 6 seam coal, already partially hydrogasified at 1300°F, was reacted at 1700°F with a hydrogen-steam feed gas mixture containing 50 mole percent steam. The coal was reacted in a 3-1/2-ft bed, the bottom of which was located at a gas distributor disk half-way up the reactor tube. Results of this test will be compared with those of tests conducted in a 7-ft bed to determine the effect of coal bed depth. Run HT-81 lasted 5-1/2 hours, terminating when the coal feed was spent. About 35% of the carbon in the feed was gasified.

Runs HT-82 and HT-83 were conducted at similar conditions to simulate the upper, low-temperature reaction zone of a two-stage hydrogasification process. Lightly pretreated Pitts-burgh seam coal was reacted at 1300°F in a 7-ft bed with a feed cas containing 36 mole percent hydrogen, 30 mole percent methane, and 34 mole percent steam. To obtain additional hydrogasification data at low hydrogen/coal stoichiometric ratios, the tests were conducted at a hydrogen/coal ratio 12% of stoichiometric. But HT-82 was terminated after 2-1/2 hours and before steady-state operation was reached, when coal would not discharge from the reactor. An internal leak in the bottom connection of the 3/8-inch feed gas tube allowed steam to condense in the relatively cool section above the discharge screw. The condensation wetted the coal and kept it from flowing down to the discharge screw. After the leaking connection was repaired, Run HT-83 was conducted successfully. The test lasted 5-1/2 hours and was shut down voluntarily when the coal feed supply was exhausted. Residue from this run will be used as feed for a later high-temperature test.

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Run HT-84 was another low-temperature hydrogasification test with a lightly pretreated Pittsburgh seam coal. The coal was reacted at 1500°F in a 7-ft bed with a feed gas containing 45 mole percent hydrogen, 15 mole percent methane, and 30 mole percent steam. The feed gas hydrogen concentration was higher than in Run HT-85 (56 mole percent), however, the hydrogen/coal ratio was still low at 15% of stoichiometric. The test lasted 5-1/2 hours, including a 5-hour steady-state period. The test was terminated when a gas leak developed at the screw feeder packing gland which could not be repaired under test conditions.

A larger capacity residue receiver, recently fabricated in the IGT shop, was installed in the hydrogasif cation pilot unit. The receiver has a capacity of 14.7 cubic feet for holding the larger quantities of residues which will be produced when fluid-bed tests with high coal feed rates are initiated. Since the installation required moderate modification of the support framework and the counterbalancing system, the pilot unit was shut down for nearly 2 weeks.

Additional outdoor feed gas storage capacity - 16 high-pressure gas cylinders - has been received. These cylinders are mounted horizontally in four banks adjacent to the pilot plant. Each cylinder is 21 feet long with an internal volume of 14,171 cubic inches.

The recently fabricated 17.5-cubic foot capacity coal feed hopper was X-rayed to determine the soundness of the welds, and then stress relieved. This hopper will be installed when fabrication of the modified reactor top is completed.

Coal Pretreatment

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Four pretreatment tests were conducted in the fluidizedbed pretreatment pilot unit to study minimum pretreatment conditions and to supply the hydrogasification pilot unit with nonagglomerating char.

Mominal conditions for the four runs were as follows:

Run No.	Solids Rate, lb/hr	Avg Solids Res Time, hr	Feed Gas Rate, SCF/hr	Oxygen Conc.%	Bed Temp,
FP-59 FP-40 FP-41	46.7 41.7 64.3	1.64 1.64 0.80	66 <u>5</u> 58 <u>5</u> 602	9.3 9.1 9.5	739 711 733
FP-42	65.4	0.9 !	589	9.5	712

The feed for Runs FP-39 and FP-40 was -16, +80 mesh Broken Arrow mine bituminous coal. An air-nitrogen mixture was the pretreatment gas. The tests were conducted to provide the pilot hydrogasification unit with free-flowing Broken Arrow mine char. Both runs were made under conditions that had produced

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nonagglomerating char in previous experiments. Run FP-39 was operationally successful and was terminated when the coal feed supply was depleted. Laboratory agglomeration tests showed the char to be free-flowing. During Run FP-40 the desired bed temperature (750°F) could not be maintained due to erratic bed movement and subsequent plugging of the reactor. The average bed temperature during the test was 711°F. Agglomeration tests showed the char to be partially caked.

Run FP-41 was made with -16, +80 mesh Ireland mine bituminous coal as the feed. An air-nitrogen mixture was the pretreatment gas. The objective of the test was to simulate the first stage of a two-stage pretreatment process.

The coal residence time and crygen/coal feed ratio were one-half that required to produce free-flowing char in previous Ireland mine coal pretreatment tests. Run FP-41 was terminated after 2-1/2 hours when the internal cyclone plugged and the back pressure in the reactor caused the coal in the bed to flow through the cyclone dip leg to the knockout drum. FP-42, run under the same conditions as FP-41, was successful and was terminated when enough char was produced for use as feed in the succeeding second-stage test. Agglomeration tests showed the char to be caked.

The second-stage test will be conducted with FP-42 char as feed, other operating conditions remaining the same. The combined coal residence times and oxygen/coal feed ratios of the two tests will equal that of previously conducted one-stage pretreatment tests. The primary interest in this series of tests is to study the effect of two-stage pretreatment on the volatile content of the resultant char.

Coal Characterization

. N S T I T U T E

Dynatech Corp. continues to have difficulties with the heat-of-reaction calorimeter. The internal high-temperature leads residual short to the heater shields. The trouble has been traced to residual organic materials in the insulation which, under high vacuum, enter the vapor phase and crack to form carbon at the heater lead junctions. To correct this situation, Dynatech is prefiring the insulation to destrey the organic matter and installing shielded leads. Any further carbon deposition would therefore only create a short from lead shield to heater shield, which are both grounded. We believe this will solve the problem. After reassembly and performance of satisfactory n-decame hydrogenation tests, the unit will be shipped.

Reactor residue samples from hydrogasification of pretreated Ireland mine coal were examined petrographically. Sigmificant increase in the reflectance of coal samples was observed with the degree of gasification, changing from about 1% for pre-

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treated coal to 2.5%-5.5% for low-temperature hydrogasification residue, and to 4%-6% for high-temperature hydrogasification residue. Progress of gasification was shown by the partial disappearance of the interior vesicle wall pattern. The exterior walls appeared to have reacted less than the interior; this may be the result of low reactivity of the skin formed during pretreatment. Considerable variation among particles in degree of tasification was noted.

Particle densities were determined on each of two sieve fractions of the feed and residue from free-fall hydrogasi-fication Run HT-50.

Methanation

Pulse tests were continued in an effort to determine the mixing characteristics of the reactor at conditions of synthesis. Several preliminary methanation runs were carried out to determine the range of operation. High conversions to methane were found for the three feed mixtures with no carbon deposition at temperatures from 700° to 800°F and at a reactor pressure of 1000 psig. Girdler G-65 catalyst was used.

Engineering Economics

Evaluation of the effect of reduced carbon conversion in the hydrogasification step on the overall process economics is proceeding. Reduced carbon conversion produces an excess of spent hydrogasification char. With the state-of-the-art design as a basis — at 55% carbon conversion — estimates are being made of the cost of pipeline gas production with carbon conversions in the hydrogasifier from 45% to 20%.

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

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Jack Huebler, Associate Director

Frank C. Schora, Manager

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DEVELOPMENT OF IGT HYDROGASIFICATION PROCESS RESEARCH

Progress Report - November 1965

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

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Hydrogasification

Three hydrogasification tests (Runs HT-85, HT-86, and HT-87) were conducted in the balanced-pressure pilot unit in a continuation of the study of the hydrogasification of lightly pretreated bituminous coals. Lightly pretreated Pittsburgh seam bituminous coal from the Ireland mine was the feed in Run HT-85; partially hydrogasified Pittsburgh seam bituminous coal from Runs HT-82 and HT-83 was the feed in Run HT-86; and residue from Runs HT-84 and HT-85 was the feed in Run HT-87.

Run HT-85 was a low-temperature test in which pretreated Pittsburgh seam coal was reacted in a 7-foot coal bed at 1300°F with a feed gas containing 45 mole percent hydrogen, 25 mole percent methane, and 30 mole percent nitrogen. Steam, normally used in hydrogasification tests, was not used. This test was performed to determine if steam has any effect on hydrogasification results when present as a diluent in the low-temperature runs. Although there is only very little, or no net reaction of steam with carbon at low temperatures, the role of steam may not be that of a completely inert gas, since it is also a product of the reaction of feed hydrogen and the oxygen in the coal. After 6-1/4 hours of trouble-free operation, the test was terminated when the coal feed supply was used up. The partially hydrogasified residue from this test was used as a feed in a later high-temperature test.

Partially hydrogasified Pittsburgh seam coal (residue of Runs HT-82 and HT-83) was used in Run HT-86 at a nominal bed temperature of 1700°F with a mixture of hydrogen and steam. The steam concentration in the feed gas was 35 mole percent, and the hydrogen/coal ratio was 28% of stoichiometric. This high-temperature test was conducted in a 3-1/2-foot coal bed and lasted 4-1/3 hours. The test was terminated when the coal feed was consumed.

Run HT-87 was also a high-temperature hydrogasification test with partially hydrogasified Pittsburgh seam bituminous coal as the feed. Residue from hydrogasification Runs HT-84 and HT-85 was made to react at 1700°F in a 5-1/2-fcot bed with a mixture of hydrogen and steam. The steam concentration in the feed gas was 30 mole percent, and the hydrogen/coal ratio was about 28% of stoichiometric. The test was successful, and was terminated after 7 hours of operation.

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After the completion of Run HT-87, further testing in the pilot unit was delayed until necessary equipment modifications for fluid-bed operation could be completed. These modifications allow hydrogasification operation at gas throughputs three to four times the previous rates used, with proportional increases in coal feed rates.

The present coal feed hopper was removed from the pilot unit and the recently fabricated 17.5-cubic-foot-capacity pressure vessel installed. This installation required modification of the supporting framework and servicing deckwork. Coal will be fed from this pressurized hopper to the reactor by a machined 1-7/8-inch-diameter feed screw, which replaces the original 7/8-inch-diameter screw.

A high-capacity Hills-McCanna water feed pump was installed. This pump will feed water at operating pressure to the steam generator. Piping of water lines to and from the pump is nearing completion.

U-bend return heat-exchange tubes were fabricated to handle the increased steam generating and hydrogen preheat loads associated with the higher feed gas rates expected in fluid-bed operation. The heat exchange tubes were installed in a gas-fired furnace. Piping of the tubes to the unit is nearly completed.

A large-capacity Rockwell gas meter was installed. It has a capacity up to 3000 CF/hr, and will meter product gases leaving the hydrogasification unit.

Manifolding and piping of the recently installed battery of 16 high-pressure gas storage cylinders was completed. This involved welding of short line sections and fittings. Hydrogen and hydrogen-methane feed gas mixtures will be stored in these cylinders at pressures up to 3500 psig.

Coal Pretreatment

Three pretreatment tests were conducted in the fluidized-bed pretreatment pilot unit. The objective of the tests was to study minimum pretreatment conditions and to supply the pilot hydrogasification unit with nonagglomerating char.

Nominal conditions for the three runs are as follows:

Run No.	Solids Rate, lb/hr	Avg Solids Res	Feed Gas Rate, SCF/hr	Oxygen Conc.%	Bed Temp,
FP-43	36.5	1.56	608	9.6	742
FP-44	48.7	1.13	600	9.6	733
FP-45	49.6	1.30	611	9.1	728

Run FP-43 was conducted using partially pretreated char from Run FP-42 as the feed material. Run conditions were set so that the combined oxygen/coal feed ratios and residence times of tests FP-42 and FP-43 would approximate those of previously conducted one-stage pretreatment tests. The run was operationally successful and was terminated when the feed supply was depleted.

The purpose of this series of tests was to observe the effect of two-stage pretreatment (as discussed in the October 1965 Report) on the volatile content of the resultant char. Production of a high-volatile content (approx 27%-28%) nonagglomerating char was of particular interest. Leboratory agglomeration tests showed the char to be free-flowing but the volatile content (24.0%) was similar to that of previously conducted one-stage tests.

Runs FP-44 and FP-45 were conducted primarily to supply the pilot hydrogasification unit with suitable feed. A -16, +80 mesh Ireland mine coal was used as feed with an air-nitrogen mixture as the pretreatment gas.

Both tests were operationally successful. Agglomeration test results of the FP-44 char showed some of the smaller particles to be lightly taked, whereas all the char from FP-45 was lightly taked. The reason for this was the decrease in residence time in the conditions of the two tests. Although the runs were primarily conducted to produce a free-flowing char for hydrogasification tests, we were still attempting to lower the minimum requirements for pretreatment. The two chars will be stored for possible use in the fluidized-bed hydrogasification test series.

A Schutte-Koerting venturi gas scrubber has been installed in the product gas line for further cleaning of the product gas to prevent the carry-over of mist to the gas meter.

Coal Characterization

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Testing of the heat-of-reaction calorimeter is continuing at Dynatech Corp. Leaks were found in a welded portion of the calorimeter bomb. The calorimeter was disassembled, rewelded, and examined by X-ray. The heater leads on top of the calorimeter shield again burned out. Arcing from the leads to the shield was suspected. This arcing apparently occurs because of off-gassing from the insulation within the heater shield during chamber evacuation. Under the low pressures in this system, the gases ionize, which melts the heater leads. The heating elements are being modified to eliminate this problem.

Particle density was determined on additional sieve fractions for the feed and residue from free-fall hydrogasification Run HT-60. True density by helium displacement was also determined on these samples and on the two sieve fractions whose

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particle densities were determined last month. Porosity calculated from these measurements ranged from 56% to 59% on the feed and 67% to 73% on the residue.

A gem-quality diamond was obtained for use as a reflectance standard in the high reflectance range. A strontium titanete single-crystal boule has been ordered for the same purpose.

Arrangements have been made for an electron microprobe examination on a cross-sectioned sample of residue from hydrogasification. This should provide information on the inherent ash of individual residue particles.

Means for improving our photomicrographic equipment is being investigated.

Methanation

Additional mixing tests were conducted to assure that complete mixing is being achieved. These mixing tests were carried out by the step input technique at various reactor shaft rotation speeds and different feed rates. The results indicate complete mixing.

Kinetic studies were performed with three different feed gases. Table 1 shows the conditions of these studies at flow rates from 5 cu ft/hr to 30 cu ft/hr.

Table 1.-METHANATION STUDIES WITH GIRDLER G-65 CATALYST

Fee	d Gas	Compos	<u>itions</u>	55	Pressure,	Temp,	No. of
CO	H2	CO _e	CH4	Total	psig	c _E	Steady-State Tests
7.2				100.00	1000	700	3
				100.00	1000	700	5
2.6	13.25	1.95	82.20	100.00	1000	700	8
2.6	15.25	1.95	82.20	100.00	1000	800	3

Data obtained from these tests are being analyzed.

Engineering Economics

Evaluation of the effect of the degree of carbon conversion in the hydrogasifier on the economics of pipeline gas production is continuing. The state-of-the-art design was based on a carbon conversion of 53%. Using this design as a basis, six cases are being evaluated in the range of 20% to 45% carbon conversion.

To date, process stream quantities and compositions have been determined for all cases. Equipment for coal grinding and pretreatment, with the exception of conveyors, has been sized and costs estimated. Prepurification vessels and methanation reactors have been estimated; costs for oxygen and hydrogen production have been determined; and most of the power and steam requirements have been calculated.

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

Approved

ack Huebler, Associate Director

Signed

Frank C. Schora. Manager

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DEVELOPMENT OF IGT HYDROGASIFICATION PROCESSOFFICE OF FOAL RESEARCH
Progress Report - December 1965

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

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<u>Hydrogasification</u>

The first fluid-bed hydrogasification test (Run HT-86) was conducted in the balanced-pressure pilot unit following completion of necessary equipment modifications for fluid-bed operation. Lightly pretreated Pittsburgh seam bituminous coal from the Treland mine was reacted in a 3-1/2-foot fluid bed at 1000°F with a feed gas containing 45 mole percent hydrogen, 25 mile percent methane, and 30 mole percent steam. In this low-temperature run, the coal feed rate was 65 lb/hr, and the hydrogen/coal ratio was 250 of the stoichiometric ratio. The test was terminated after less than 1 hour, and before steamy-state operation was reached, when difficulties were encountered in discharging coal from the reactor. Compaction of the reacted coal at the annulus between the feed gas distributing disk and the reactor wall was the apparent cause of this difficulty. To remedy this situation, the feed gas distributing disk will be reduced from 3-1/2 to 3 inches in diameter, which will increase the cross-section of the annulus. Although the run was too short to yield any operational data, it did serve to check the operability of the new equipment components.

Before conducting Run HT-00, the necessary equipment modifications for fluid-bed operation were made. These consist of a 1-7/6-inch-diameter coal feed sorew, feed sorew housing, variable-speed drive, a reactor head assembly with an internal cyclone, gas-sampling probes, internal coal-bed thermocouples, and peripheral equipment to handle the higher gas flows.

The modified installation underwent shakedown tests, including:

- Operation and calibration of the real freed shrow at coeffectes up to 100 lb/hr.
- Operation of the water feed pump and steam pendrator at rates up to 50 lb/hr.
- Calibration of the Feed-pas cristics mater of 1200 SOF/Er against a new gas meter.
- Nitrogen flow test through the pilot entitlet 5000 SOF/hr to test capacity of the feed and product gas lines and the capability of the unit back-pressure regulator to function at high gas rates.

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 Pitrogen-steps New Last Dirough the pilet unit to check capacity of the cas Hatributing disk and off-ma condenser at hydromal New Pates of 1140 SCF in and 25 lb/hr steps.

Ocal Pretreutment

Four pretristment teaths were conducted in the fluidizedtion proceediment place unit to dud, minimum pretreatment conditions and to supply the pilou hydrogasification unit with nonautomorating char.

Mominal conditions for the four tests are as follows:

Run IIc.	Solida Rate,	Avg Splide Res	Feed Gas Rate, SOF/hr	Oxygen Conc. :	Bed Temp,
FF-46	45.3	1.10	596	1.7	743
FF-47	48.0	1.51	2 978	3.0	741
당면_46	50.7	1.75	> őin	#.5	738
378-43	91.0	1.27	√e2	9.8	750

A -W, and mean inclaim mine bibining is at was used as feed in Run FP-WG, with an air-nitrogen mixture as the pre-treatment gas. The test was conducted to provide the pilet hydrogasifier with nonagglomerating feed. The run was operationally successful, and laboratory agglomeration test results showed the char to be free-flowing.

Treland sine oftunious coal of -1/4 in. was the feed material for Run FP-47, and an air-vitropen minture was the pre-treatment cas. The test was conducted to investigate the operability of larger sized coal particles in the pretreatment pilotunit. The test was successful and laboratory anglomoration tests showed the char to be lightly caked.

A West Virginia bituminous coal from the No. 5 Block deam was used as the feed for Runs FP-48 and FP-49. The challs the trird of seven quals selected for abuse under the compact project. A problem analysis of the coal is:

<u> </u>	Nejte	<u> </u>	irlay - Augmont, I	Antimother, Bu/D
i • I	<u></u>	**	5 .5	18.000

An alse time consists was used as the pretreatment one for bits runs will the scal feed particle size was -10. Will mesh. Both tests were conducted under similar conditions except for the extent escentration of the pretreatment was. How FF-10 was rade with 5.00 cayeen concentration; FP-40 was tested with 5.00 cayeen. Important applement ion tests showed both means to be partially caked, indicating that minimum pretreatment conditions were not attained. Further tests will be

conducted to define minimum pretreatment conditions for the West Virginia coal.

Coal Characterization

Dynatech Corp. continued testing of the heat-of-reaction calorimeter. During a 4-day test, all heaters functioned properly at 1500°F. The insulation was repacked to reduce heat leaks. However, the 0-ring failed at 1500°F and 1500 psia, due presumably to lack of sufficient vent holes. The calorimeter was reassembled with new 0-rings and calibration has begun.

Calibration of the drop calorimeter was completed. The results are satisfactory.

Ccal Petrography

Preparation of samples for a systematic examination of the coal at different stages of the hydrogasification process was started. Feed and product from pretreatment Run FP-51 and feed and residue from high-temperature hydrogasification Run HT-72 have been chosen to constitute such a series for Ireland mine coal. Mounting and polishing of selected sieve fractions of these samples has been started. Helium density and porosity will also be determined on some of these sieve fractions.

Improved sample polishing and photographic techniques were developed. Polishing of pretreated coal and hydrogasification residues is difficult because of the voids in these particles. A much better polish has been obtained by reimpregnating the mounted sample surface after rough grinding with epoxy resinunder vacuum. Photomicrographic technique was reviewed with a consultant to obtain better acutance, or sharpness, and better exhibition of slight differences in sample reflectance. The latter will allow illustration of the remnants of pretreatment skin found in hydrogasification residues.

A sample of residue from a free-fall hydrogasification run (HT-59), in which raw (not pretreated) coal had been fed, was prepared for petrographic examination. Observations on this sample will be reported next month, particularly on absence of the effect of the pretreatment skin on reactivity.

A prism has been cut from a strontium titanate singlecrystal boule for use as a high-reflectance standard. Polishing of the surfaces is not yet completed.

Methanation

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Methanation studies continued with Girdler G-65 and Harshaw Ni-0104T nickel catalysts. For comparable space velocities the Harshaw catalyst gave conversions equal to those

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obtained with the Girdler catalyst operating at approximately a 100° F higher temperature. The lower limit of activity for Ni-0104T is about 450° F, compared to 600° F for G-65. The activity of the Ni-0104T was more steady during its time onstream than was the G-65 catalyst.

Catalyst screening tests are continuing and will include studies of a nickel catalyst from Catalyst & Chemicals, Inc., and a 0.5% ruthenium on alumina catalyst from Engelhard Industries, Inc.

Engineering Economics

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Evaluation of the effect of the degree of carbon conversion in the hydrogasifier on the economics of pipeline gas production is largely completed. Results will be presented in a memorandum to A.G.A.-OCR.

Approved _______ Signed _______ Signed _______ Frank Schora, Manager

PROGRAM FOR DEVELOPMENT OF 1GT

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

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T HYDROGASIFICATION PROCESS

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Liff of total costs. DS - Errect Salaries, \$ ODC - Other Direct Custs, \$

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