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U.S. DEPARTMENT OF ENERGY
Division of Coal Conversion

QUARTERLY REPORT

October - December 1977

DOE / ET - 0026 / 4

Publication Date: May 1978

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FOR THE

COAL

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COAL LIQUEFACTION

QUARTERLY REPORT OCTOBER-DECEMBER 1977

U.S. DEPARTMENT OF ENERGY

Division of Coal Conversion

Washington, D.C. 20545

Publication Date: May 1978

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FOREWORD

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EXECUTIVE SUMMARY

The United States has more energy available in coal than in petroleum, natural gas, oil shale, and tar sands combined. Nationwide energy shortages, together with the availability of abundant coal reserves, make commercial production of synthetic fuels from coal vital to the Nation's total supply of clean energy. In response to this need, the Department of Energy (DOE) is conducting a research and development program to provide technology that will permit rapid commercialization of processes for converting coal to synthetic liquid and gaseous fuels and for improved direct combustion of coal. These fuels must be storable and suitable for power generation, transportation, and residential and industrial uses.

DOE's program for the conversion of coal to liquid fuels was begun by two of DOE's predecessor agencies: Office of Coal Research (OCR) in 1962, and Bureau of Mines, U.S. Department of the Interior, in the 1930's. Current work is aimed at improved process configurations for both catalytic and non-catalytic processes to provide more attractive processing economics and lower capital investment. The advantage of coal liquefaction is that the entire range of liquid products, including heavy boiler fuel, distillate fuel oil, gasoline, jet fuel, and diesel oil, can be produced from coal by varying the type of process and operating conditions used in the process. Furthermore, coal-derived liquids have the potential for use as chemical feedstocks.

To provide efficient and practical means of utilizing coal resources, DOE is sponsoring the development of several conversion processes that are currently in the pilot plant stage. The development of the solvent refined coal process is being carried out in pilot plants at Fort Lewis, Washington and Wilsonville, Alabama. Responsibility for the operation of the *Solvent-Refined Coal (SRC) Process* pilot plant at Ft. Lewis, Washington is assigned to Pittsburg and Midway Coal Mining Company. During the quarter, two material balance runs were completed with Illinois No. 6 coal, and testing began with Pittsburgh seam coal from Consol's Blacksville No. 2 mine. The Merriam Laboratory continued supporting studies on the Pittsburgh coal with nine SRC II process variable runs completed.

DOE, together with the Electric Power Research Institute (EPRI), has contracted with Southern Services Company, Inc. for the *Operation of a Solvent-Refined Coal Pilot Plant* at Wilsonville, Alabama. Indiana coal from the old Ben No. 1 mine continued to be processed during the quarter.

Ashland Synthetic Fuels, Inc. (ASFI) is responsible for management of the construction of the *H-Coal Project* pilot plant. Construction was 30 percent complete by the end of the quarter. Hydrocarbon Research, Inc. (HRI) successfully completed a 30-day PDU run with Illinois No. 6 coal and also a 30-day run with Wyodak coal.

Using engineering and design technology similar to that practiced in the petroleum industry, the *Donor Solvent Liquefaction Process* is being developed by Exxon Research and Engineering Company. Studies continued during the quarter with Illinois No. 6 coal in the one-ton-per-day Coal Liquefaction Pilot Plant. Solvent quality studies continued in the 50 pound-per-day Recycle Coal Liquefaction Unit on Wyoming coal.

Cities Service Research and Development Company is conducting sub-scale studies as the first phase of the *Commercial Scale Ebullated-Bed Hydroprocessing of Solvent Refined Coal Extract*. On the basis of screening runs completed during the quarter, a commercially available denitrogenation/desulfurization catalyst was selected as the candidate for a 30-day catalyst aging run that began in December.

Fluor Engineers and Constructors, Inc. is responsible for reactivating the DOE-owned *Cresap Test Facility* in Cresap, West Virginia. Four extraction operation runs were conducted during the quarter.

Construction of the 10-ton-per-day process development unit (PDU) to test the *Synthoil Process* is being managed by Foster-Wheeler Corporation. Associated laboratory research was conducted by the Pittsburgh Energy Research Center (PERC). During the quarter, a high quality product which was low in sulfur and liquid at room temperature was produced for 1440 hours of continuous uninterrupted operation of the 1/2 ton-per-day unit. The run indicated that the process could be used to produce liquid fuel from nearly all bituminous coals.

The *Clean Coke Process* is under development by USS Engineers and Consultants, Inc., a subsidiary of United States Steel Corporation. The results of laboratory tests and data obtained from the process development unit (PDU) will be used for possible scale-up. The carbonization PDU program for West Kentucky coal was completed during the quarter, and operation with Pittsburgh seam coal started. Preparation and testing of binder materials and formcoke specimens continued.

The Rocketdyne Division of Rockwell International is conducting a program to develop a technology which will permit the *Liquefaction of Coal by Direct Hydrogenation* in an entrained-flow reactor. Process variables studied

in 20 tests conducted in the one-ton-per-hour reactor during the quarter included residence time, reactor hydrogen requirements, and hydrogen injection temperature.

Studies are being conducted by the Occidental Research Corporation to evaluate the commercial potential of the company's *Flash Pyrolysis Coal Liquefaction Process*. Eight runs were conducted on the pyrolysis reactor during the quarter. Progress was made in reducing circulating char upsets which have hindered operations.

Research to improve solid-liquid separation is being conducted by Johns-Manville Sales Corporation in their *Filtration Process and Equipment Studies*. The new test leaf system to allow "in situ" operation for future runs was completed, installed, and partially evaluated during October. No filtration rate data was obtained during the quarter as the system was moved and revised for purpose of improving safety conditions.

Dow Chemical Company has conducted studies dealing with the production of *Chemicals from Coal* using the liquids from four coal liquefaction processes (COED, Synthoil, H-Coal, and SRC) in standard petro-chemical processing experiments. The Final Report based on the HRI H-Coal product was published in October.

Oak Ridge National Laboratory is providing *R&D Engineering Services* dealing with *Hydrocarbonization of Coal*. Hydrocarbonization is a type of coal liquefaction process which combines high temperature, high pressure, and fluidized-bed operation using hydrogen-rich gas for fluidization to enhance product yields. One hydrocarbonization run using Illinois No. 6 coal pretreated with aqueous sodium carbonate at 300°C was completed during the quarter.

Conoco Coal Development Company, with the participation of Shell Development Company is developing the *Zinc Halide Hydrocracking Process*, a technique designed to produce clean liquid and gaseous fuels from coal or coal extract. The hydrocracking section of the one-ton-per-day Process Development Unit (PDU) was completed during the quarter. The continuous bench-scale hydrocracker program was completed as was an economic analysis of the cost of secondary zinc recovery.

Exxon Research and Engineering is studying the potential for *Fluid Coking of Coal Liquefaction Residues* to recover distillate liquids. Analyses of products and interpretation of data from each of the Continuous Stirred Coking Unit runs continued throughout the quarter. A fourth coal liquefaction residue sample, an H-Coal vacuum bottoms, was received for analytical characterization.

To facilitate the possible desirable upgrading of primary coal liquids using technology in the petroleum industry, UOP, Inc. is conducting a study of *Upgrading Coal Liquids*. Second-stage hydrocracking of H-Coal gas oil to fuel oil was concluded during the quarter. A series flow second-stage hydrocracking of high nitrogen H-Coal liquid to gasoline was also conducted. Studies began on Exxon Donor Solvent process liquid.

Texaco, Inc. is conducting a study of the *Gasification of Residual Coal Liquefaction Products*. Material from several coal liquefaction pilot plants will be evaluated to determine their suitability as feedstock for the proprietary Texaco partial oxidation process. A type I evaluation to determine the chemical composition and physical properties of a sample of SRC ash concentrate from Kerr-McGee Corporation was completed during the quarter.

The *Production of Clean Industrial and Transportation Fuels From Coal* is being investigated by the Lummus Company using a multi-stage ebullated-bed catalytic reactor developed through the firm's activities with a bench-scale pilot plant. Operations during the quarter were directed toward the preparation of equilibrium recycle solvent.

I. SOLVENT-REFINED COAL (SRC) PROCESS

THE PITTSBURG & MIDWAY COAL MINING
COMPANY

MERRIAM, KANSAS

Plant Site: Tacoma, Washington

Contract No.: EX-76-C-01-496

Contract Funding: \$88,759,707
(100% DOE)

INTRODUCTION

The SRC process is being developed by Pittsburg & Midway Coal Mining Company (P&M), a subsidiary of Gulf Oil Corporation, under the sponsorship of DOE. The specific objectives of the contract are to (1) continue obtaining technical and economic data from the pilot plant to validate scale-up to commercial production and (2) provide large samples of solvent-refined coal and by-products from the process for market development studies. Figure I-1 provides a schedule for the development of the process.

PROCESS DESCRIPTION

The SRC process converts high-sulfur, high-ash coals to low-sulfur, low-ash liquid fuel. Figure I-2 shows a schematic of the process. The coal is first pulverized and mixed with a coal-derived solvent in a slurry mix tank. The slurry is combined with hydrogen and is then pumped through a fired preheater and passed into a dissolver. In this unit, the coal is hydrogenated and thereby depolymerized, leading to an overall decrease in product molecular weight and dissolution of the coal. The solvent is also hydrocracked in the dissolver unit, yielding lower molecular weight hydrocarbons ranging from light oil to methane. Yet another reaction occurring in the dissolver

is the hydrogenation of the organic sulfur in the coal, thus producing hydrogen sulfide.

From the dissolver, the mixture passes to a separator where the gases are separated from the slurry of undissolved solids and coal solution. The raw gas is sent to a hydrogen recovery and gas desulfurization unit. The recovered hydrogen is then recycled and combined with the slurry coming from the slurry mix tank. Hydrocarbon gases are released and recovered and the hydrogen sulfide is converted to elemental sulfur.

The slurry of undissolved solids and the coal solution is then separated in a filtration unit. In a commercial-scale process, the solids would be sent to a gasifier-converter where they would react with supplemental coal, steam, and oxygen to produce hydrogen for use in the process. The coal solution passes to the solvent recovery unit; and the final liquid product, solvent-refined coal, is produced. This material has a solidification point of 350°F to 400°F and a heating value of approximately 16,000 Btu per pound.

HISTORY OF THE PROJECT

The SRC project was begun in 1962 when Spencer Chemical Company was awarded a research contract by the Office of Coal Research (OCR, subsequently a part of ERDA, now DOE) to study the technical feasibility of a

LEGEND: △ BEGIN MILESTONE
▽ COMPLETE MILESTONE

Figure I-1. SOLVENT-REFINED COAL PROGRAM SCHEDULE

Figure I-2. SRC PROCESS SCHEMATIC

coal de-ashing process (now called the SRC process). In 1965, the process was successfully demonstrated in a 50-pound-per-hour continuous-flow unit, and the work on the contract was therefore completed. During the term of the contract, Gulf Oil Corporation acquired Spencer Chemical Company. After reorganization, the contract was assigned to the research department of P&M.

To develop the SRC process further, a contract was awarded to P&M to design, construct, and operate a pilot plant that would be capable of processing 50 tons of coal per day. In 1969, Stearns-Roger Corporation completed the design for the pilot plant, but funds to begin construction were not available until late 1971. In June 1972, OCR extended its contract with P&M to construct and operate the pilot plant. Rust Engineering Company began constructing the pilot plant in July 1972 at Ft. Lewis, Washington, near Tacoma. The pilot plant became fully operational in October 1974.

Tests runs during 1975 studied the effects of different coal feed rates and dissolver temperatures. A 3,000-ton run was begun late in 1975 for production of specification SRC for use in combustion testing by Southern Services Company's Georgia Power Company (see Section III) and was completed in December of 1976.

After modification of filtration instrumentation following the production run, filtration tests were conducted. The plant was then shut down for a major maintenance turnaround and SRC II (slurry recycle) piping tie-ins. Plans had been completed during the year for modifications of the pilot plant for SRC II testing.

The laboratory unit has been run in both the distillate solvent mode and slurry recycle mode. The variables investigated included filtration rate, solvent recycle rate, slurry residence time, concentration of coal in the slurry, and temperature. Tests were also run to simulate operating conditions at the pilot plant. A series of experimental runs were designed to develop the SRC II process. The effect of process variables in SRC II operation was investigated.

In addition to laboratory experiments conducted at the P&M Merriam, Kansas laboratory, other research supporting the development of the SRC process included:

- Formulation of an interagency agreement between EPA and ERDA to conduct environmental studies concerning the removal of sulfur and nitrogen compounds from the coal.
- Experiments by Oklahoma State University to test the efficiency of commercial catalysts in removing sulfur and nitrogen from coal liquids, and to determine the rate of decay of hydrogenation catalyst activity.

- Tests at Washington State University to determine the trace element content of the feed coal, process, product and residue streams in the SRC process.

PROGRESS DURING OCTOBER-DECEMBER 1977

Summary

During the quarter, all operations were in the SRC II mode. In October, two material balance runs were completed with Illinois No. 6 coal from Peabody's River King Mine. During the remainder of the quarter, the feed was switched to Pittsburgh seam coal from Consol's Blacksville No. 2 mine and four material balances runs were completed.

Nine SRC II runs were made on Blacksville Mine No. 2 coal at the Merriam Laboratory. The effect of varying dissolver temperature and residence time was studied as was the effect of increased hydrogen partial pressure.

The EPRI Filter "C" was set in place and the bridge crane installed above the filter in November. At the end of December, the overall project was 66 percent complete.

Pilot Plant

During 28 days of operation in October, 755 tons of coal were processed. Plugging problems were experienced in the coal feed and eductor mixing systems and in the vacuum flash systems.

Typical distillation and other analytical data for the SRC II liquid products from Illinois coal were:

SRC II Liquids — Illinois Coal			
	Naphtha	Middle Distillate	Heavy Distillate
Naphtha (IBP-350° F), Vol. %	83	1	0
Middle Distillate (350°-550° F), Vol. %	17	94	1
Heavy Distillate (550°-850° F), Vol. %	0	5	99
Specific Gravity, 60/60° F	0.818	0.964	1.084
Viscosity at 100° F, cSt	0.67	3.09	66.37
Pour Point, °F	—	-52	32
Flash Point, °F	—	161	—

MAF coal conversion averaged 94.47 percent during October. Two material balance runs were completed and the resultant yields on an MAF basis were:

Yields, Wt % of Moisture-Free Coal

Component	Run 77SR-10	Run 77SR-11
H ₂	-5.1	-3.1
C ₁	3.1	4.0
C ₂	2.9	3.3
C ₃	2.4	2.5
C ₄	1.0	1.2
CO	-0.5	0.2
CO ₂	1.8	1.9
H ₂ S	2.6	2.8
H ₂ O plus losses	19.1	9.8
Naphtha (IBP-350°F)	10.7	6.9
Middle Distillate (350-550°F)	22.2	17.4
Heavy Distillate (550-850°F)	-2.0	9.6
SRC*	25.0	27.6
IOM	4.4	4.4
Ash	12.4	11.5
Total	100.0	100.0

*Average SRC Fusion Point: °F 224 307

Due to a high degree of liquefaction obtained in run 77SR-10, the residual SRC production was low while the coal feed ash concentration was high, resulting in a very heavy solids loading in the vacuum flash system. To maintain a solids concentration of less than 45 percent in the vacuum bottoms, the vacuum flash severity was decreased. The old, oversized vacuum flash system was in use during the run. The new vacuum flash system was in use for run 77SR-11, making it possible to produce a vacuum bottoms product with a higher fusion point.

Coal feed was switched to Pittsburgh seam coal from the Blacksville No. 2 mine in November. To facilitate coal handling, two steam-heated dryers were installed, the pulverizer inlet chute was modified, and the primary crusher was recommissioned to break-up the large lumps. Typical distillation and other analytical data for the SRC II liquid products from the Pittsburgh Seam coal were:

SRC II Liquids — Pittsburgh Seam Coal

	Naphtha	Middle Distillate	Heavy Distillate
Naphtha (IPB-350°F), Vol. %	87	1	0
Middle Distillate (350-550°F), Vol. %	13	99	0
Heavy Distillate (550-850°F), Vol. %	0	0	100
Specific Gravity, 60/60°F	0.829	0.985	1.087
Viscosity @ 100°F, cSt	—	4.03	49.23
Pour Point, °F	—	-36	20
Flash Point, °F	—	178	—

The metering of slurry flow rates continued to be a problem in November. The material balance runs were made at lower coal concentrations and shorter residence times than planned, consequently.

Four material balance runs were completed on the Pittsburgh seam coal during the quarter and the resultant yields on an MAF basis were:

Yield, Weight Percent of Moisture-Free Coal

Component	Run 77SR-12	Run 77SR-13	Run 77SR-14	Run 77SR-15
H ₂	-4.2	-4.7	-4.5	-3.6
C ₁	9.2	7.8	7.7	5.7
C ₂	4.7	4.8	4.8	3.5
C ₃	3.6	4.0	4.0	3.0
C ₄	1.5	1.8	1.8	1.6
CO	0.7	-0.1	-0.2	0.1
CO ₂	1.5	1.5	1.5	1.2
H ₂ S	2.2	1.7	2.2	2.0
NH ₃	0.5	0.5	0.6	0.4
Water	4.1	6.7	4.5	4.3
IOM	12.2	10.1	10.1	8.8
Naphtha	5.9	3.7	11.2	3.9
Middle Distillate	17.9	26.7	25.9	22.3
Heavy Distillate	5.8	2.6	3.0	12.6
SRC	34.4	32.9	27.4	34.2
Fusion Point, °F	387	329	316	330
% Solids in Vacuum Bottoms	43.2	42.7	46.7	39.4

Run 77SR-12 was made at a dissolver temperature of approximately 860°F and pressure of 1900 psig. The yields from this run indicated that SRC and insoluble organic matter (IOM) yields were higher than would be expected at similar conditions with western Kentucky or Illinois No. 6 coals. In addition, the distillate yield was lower with the Pittsburgh coal, and the gas yield was higher.

In run 77SR-13, an attempt was made to achieve more desirable product yields by increasing the system pressure from 1900 to 2000 psig. The dissolver temperature remained at 860°F. Results showed that the increased pressure increased the distillate yield and decreased gas and IOM yield.

Run 77SR-14 was designed to study the effect of higher dissolver temperature (870°F) on the Pittsburgh coal yield structure. In general, the higher temperatures

resulted in slightly higher distillate yield, lower SRC yield, and higher IOM yield. In addition, the higher temperatures produced more hydrocarbon gas with correspondingly higher hydrogen consumption.

The accuracy of the results of Run 77SR-15 are questionable due to the short run duration of 38.4 hours, the higher concentration of vacuum bottoms in the feed slurry, and unusually large amounts of flushing and solvent transfers during the run.

Laboratory — Merriam, Kansas

Nine supporting laboratory runs were made with Pittsburgh seam coal from the Consol's Blacksville Mine No. 2 in the Merriam Laboratory. Problems continued with the slurry pump; however, it is expected that these problems are unique to small scale equipment and were not expected to be significant in large scale equipment.

Three SRC II runs were completed in November. The results indicated that with increasing dissolver temperature, the light hydrocarbon gas yield increased, the insoluble organic yield increased, SRC yield decreased, and total distillate oil yield decreased. Increase in residence time produced increased light gas yield, increased total distillate oil yield, and decreased SRC yield. No significant effect of residence time on IOM was observed.

In December, four SRC II runs were made with the Blacksville coal to determine the effect of increased hydrogen partial pressure. Results showed increased hydrogen partial pressure significantly improved process yields and operability. Mechanical operability improved substantially at both the 2200 and 2500 psig levels compared to that at the base 1900 psig level. No pump head changes were required during the higher pressure runs, but when the pressure was lowered to 1900 psig, pump check fouling again became a recurrent problem.

SOUTHERN COMPANY SERVICES, INC.

BIRMINGHAM, ALABAMA

Plant Site: Wilsonville, Alabama
Contract No.: EX-76-C-01-2270
Contract Funding: \$8,729,892
 DOE: \$5,741,892
 Industry: \$2,988,000

INTRODUCTION

The operation of the solvent-refined coal (SRC) pilot plant at Wilsonville is jointly sponsored by DOE and the Electric Power Research Institute (EPRI). The schedule for the project is shown in Figure I-3. The objectives of the project are to:

- Correlate the results of operation at the Wilsonville SRC pilot plant with those from the Ft. Lewis, Washington SRC pilot plant.
- Test several coals of various ranks and locations to determine the effects of process variables on product yield and quality.
- Evaluate SRC process improvements.
- Evaluate improvements in equipment and operations.

PROCESS DESCRIPTION

The SRC process converts high-sulfur, high-ash coals to a low sulfur, low ash solid fuel. A schematic of the process is shown in Figure I-2. The pulverized coal is mixed with two to three parts process-derived solvent. Hydrogen is then added to the coal-solvent slurry. The mixture is pumped to a preheater and then fed into a reactor or dissolver which operates at 800°-875° F, and a total pressure of approximately 1700 psig. Under these conditions, approximately 93 percent of the carbonaceous material in the coal feed is dissolved during the residence time of approximately 20 to 30 minutes. The product gases from the dissolver are separated and sent to a clean-up system to remove the hydrogen sulfide (about 60 percent of the organic sulfur in the coal is converted to hydrogen sulfide) and convert it to elemental sulfur. The liquid slurry is then subjected to a mineral separation step

**Figure I-3. WILSONVILLE SRC
PILOT PLANT OPERATION SCHEDULE**

wherein the undissolved solids are removed. Process solvent is recovered from the coal solution by distillation and recycled to slurry the coal feed. The residue which remains is the solvent refined coal product, which solidifies at about 350°F.

HISTORY OF THE PROJECT

In March 1972, the Edison Electric Institute and the Southern Company Services, Inc. began a joint project to study the key steps in the SRC process. Consequently, Catalytic, Inc. designed, built, and is operating the six-ton-per-day pilot plant. The facility was completed in August 1973. The Electric Power Research Institute (EPRI) assumed the responsibilities of the Edison Electric Institute for utility industry sponsorship in April 1973.

The pilot plant began operation in January 1974, and by the end of 1976, had been in operation for more than 13,500 hours, including periods of 45 and 75 days of sustained operation. To date, one subbituminous and six bituminous coals have been tested. SRC product meeting plant specifications of 0.16 percent maximum ash and 0.96 percent maximum sulfur has been produced from each coal. A fuel meeting these specifications and having a heating value of 16,000 Btu per pound could be used to achieve compliance with most emission limits and EPA's "new source performance standards."

PROGRESS DURING OCTOBER-DECEMBER 1977

Summary

Processing of Indiana V coal continued during the quarter. The results of installation of the distributor tray

in the bottom of the dissolver appeared to be higher hydrogen consumption and less sulfur in the SRC. Following a fire in December, one run was completed for environmental studies and to check out all sections of the plant.

Pilot Plant Operations

After a shut down of 30 days for modifications and repairs, the pilot plant began operation on October 11 with Run 130. The purpose of the run was to obtain data to compare with Run 124 which was made at identical operating conditions, but before a distributor plate was added to the dissolver bottom. Indiana V coal was used with a coal feed rate of 450 pounds per hour and a feed slurry concentration of 38.5 percent with dissolver conditions of 825°F and 1700 psig.

Preliminary results suggested that the installation of the distributor tray resulted in additional hydrogen consumption and less sulfur in the SRC. Slightly less SRC was produced. These results were confirmed by Run 131 in November. The purpose of Run 131 was to obtain data to compare to Run 125. The difference between the two runs was the installation of the distributor plate in the dissolver. Indiana V coal was used with a coal feed rate of 450 pounds per hour, 845°F dissolver outlet temperature, 1700 psig dissolver pressure and 50 percent of the dissolver volume in use.

A material balance was in progress when a fire caused an emergency shutdown of the plant. Sufficient data and samples were obtained to make a comparison with Run 125, confirming the results obtained in October.

Repairs to fire-damaged lines, wiring, and equipment continued into December. The reaction and distillation sections of the plant were conditioned to the extent that coal feed was started on December 19. The purpose of the run was to produce caustic effluent for environmental studies and to run the unit to check out all sections of the plant. Many fire-related items were corrected during the run. Indiana V coal was used with a coal feed rate of 450 pounds per hour, feed slurry concentration of 38.5 percent, and dissolver conditions at 1700 psig and 825°F. The entire dissolver volume was in service. Caustic waste water samples were collected for analysis and study as a basis for designing a caustic waste treatment facility.

The entire plant was shut down on December 27 to modify the service lines and electrical equipment to accommodate the installation of the Kerr-McGee Critical Solvent Deashing Unit.

II. H-COAL PROJECT

Plant Site: Catlettsburg, Kentucky
Total Funding: \$179,000,000
DOE: \$143,000,000
Industry: \$ 36,000,000

HYDROCARBON RESEARCH, INC.
TRENTON, NEW JERSEY

Contract Nos.: E(49-18)-1544 and
EF-77-C-01-2547

ASHLAND SYNTHETIC FUELS, INC.
ASHLAND, KENTUCKY

Contract No.: EX-76-C-01-2260

INTRODUCTION

The H-Coal process is being developed by Hydrocarbon Research, Inc. (HRI) under the joint sponsorship of (1) DOE, (2) a private industry consortium composed of Electric Power Research Institute, Ashland Synthetic Fuels, Inc., Conoco Coal Development Company, Mobil Oil Corporation, and Standard Oil Company (Indiana), and (3) the Commonwealth of Kentucky. The overall objectives of this project are to further develop the H-Coal process and to demonstrate its technical and economic feasibility on larger scales. The specific objectives are to:

- Perform laboratory research on all phases of the H-Coal process, using the existing bench-scale unit and process development unit to establish design criteria.
- Design a pilot plant capable of converting 600 tons of coal per day to 2,000 barrels per day of low-sulfur boiler fuel or 200 tons of coal per day to 2000 barrels per day of synthetic crude oil.

Ultimately, the pilot plant will be constructed and operated to provide data for further evaluation of the process and design of full-size commercial plant. The overall program plan is shown in Figure II-1.

The H-Coal Project is being conducted in three phases:

- Phase I - Design
- Phase II - Construction
- Phase III - Operation

Hydrocarbon Research, Inc. (HRI) developer of the H-Coal process, designed the H-Coal Pilot Plant under Phase I of the H-Coal Project.

Ashland Synthetic Fuels, Inc. (ASFI) is responsible for construction and operation of the H-Coal Pilot Plant under Phases II and III of the H-Coal Project. HRI will support construction and operation under Phases II and III as a subcontractor to ASFI. Badger Plants, Incorporated, Cambridge, Massachusetts, will construct the plant as a subcontractor to ASFI. The Lummus Company will design the antisolvent deashing unit for the pilot plant, also as a subcontractor to ASFI.

Figure II-1. H-COAL PROGRAM SCHEDULE

PROCESS DESCRIPTION

The H-Coal process is a catalytic hydroliquefaction process that converts high-sulfur coal to boiler fuels and synthetic crude. A schematic of the process is provided in Figure II-2. Coal is crushed to minus 60 mesh, dried, slurried with recycled oil, and then pumped to a pressure of about 200 atm. Compressed hydrogen is added to the slurry and the mixture is preheated and charged continuously to the bottom of the ebullated-bed catalytic reactor. The upward passage of internally recycled reaction mixture maintains the catalyst in a fluidized state. Catalyst activity is maintained by the periodic addition of fresh catalyst and the withdrawal of spent catalyst. The temperature of the ebullated-bed catalytic reactor is controlled by adjusting the preheater outlet temperature. Typically, the temperature of the mixture entering the reactor is 650° F to 700° F.

Vapor product leaving the top of the reactor is cooled to separate the heavier components as a liquid. Light hydrocarbons, ammonia, and hydrogen sulfide are absorbed from the gas stream and sent to a separator and a sulfur recovery unit. The remaining hydrogen-rich gas is recompressed and combined with the input slurry. The liquid from the condenser is fed to an atmospheric distillation unit. The liquid-solid product from the reactor, containing unconverted coal, ash, and oil, is fed into a flash separator. The material that is flashed off is passed to the atmospheric distillation unit which yields light and heavy distillate products. The bottoms product from the flash separator (solids and heavy oil) is further separated with a hydroclone, a liquid-solid separator, and by vacuum distillation.

The gas and liquid products, composed of hydrocarbon gas, hydrogen sulfide, ammonia, light distillate, heavy distillate, and residual fuel, may be further refined as necessary. A portion of the heavy distillate is recycled as the slurry medium. The stream containing the unre-

acted carbon and some liquid will eventually be processed in a commercial installation to produce additional hydrogen needed for the process.

The specific operating conditions of the H-Coal process affect the type of fuel produced. For example, to produce synthetic crude, more hydrogen is required and there is a lower yield of residual fuel oil. In this mode, the separation of the solids (unconverted carbon and ash) from the liquid can be accomplished by vacuum distillation, and a special liquid-solid separation unit is not required. To produce clean fuel gas and low-sulfur residual oil as major products, the temperature and pressure in the ebullated-bed reactor are lowered, and less hydrogen is required.

Before the H-Coal process can become commercially and economically competitive, an adequate supply of hydrogen must be generated from the process itself. The H-Coal process requires between 14,000 and 20,000 standard cubic feet of hydrogen for each ton of coal processed, depending on the type of oil produced. In the pilot plant, some of the hydrogen required has been obtained from the gas produced by the ebullated-bed reactor. Additional hydrogen has been purchased to meet processing demands. In a commercial operation, hydrogen demand will be met by manufacture on site. Because the H-Coal process converts about 90 percent of the carbon contained in the coal to a liquid, the feed to the hydrogen plant could be liquid rather than solid. This suggests that commercial hydrogen manufacturing processes could be adapted. The solid cake char could also be used to produce hydrogen.

In the H-Coal process, hydroclones are used to remove about two-thirds of the solids from recycled liquid used for slurring the coal. Anti-solvent precipitation is the method used to remove solids from H-Coal fuel oil. Solids will be removed from the syncrude as vacuum tower bottoms

HISTORY OF THE PROJECT

The H-Coal process was developed by HRI as a further application of the ebullated-bed processing technology originally used to convert heavy oil residues from petroleum into lighter fractions (H-Oil process). Early development of the H-Coal process, beginning in 1964, involved research with a bench-scale unit and a process development unit and preparation of a conceptual process design. An independent evaluation in 1968 confirmed the technical and economic feasibility of the H-Coal process.

Figure II-2. H-COAL PROCESS SCHEMATIC

The H-Coal process proved to be highly flexible in bench-scale and process development unit tests. The bench-scale unit can process 25 pounds of coal per day, the process development unit can process three tons of coal per day. Many types of coal have been tested satisfactorily on the bench-scale level, including eastern, mid-western, and western bituminous coals, western subbituminous coals, lignite from Texas and North Dakota, and Australian brown coal. The bench-scale unit has also provided information on the effect of process variables (temperature, pressure, coal feed rate, slurry oil composition, and catalyst age) on coal conversion and product yield. The process development unit tests demonstrated sustained H-Coal process operation with satisfactory control of the catalyst bed expansion.

Based on the data obtained from the bench-scale and process development units, design and engineering of the 200-600 ton-per-day pilot plant began in December 1973.

During 1974, design of the coal preparation section of the pilot plant was completed to the extent that engineering and plant layout could begin. HRI concentrated, however, on the design and engineering for the ebullated-bed reactor, since the equipment needed for this section requires long lead times. Engineering for other sections of the pilot plant was started in late 1974. Also during 1974, three possible sites for construction of a pilot plant were evaluated. Based on detailed environmental assessments, it was decided during the second quarter of 1975 to locate the plant adjacent to the Ashland Oil, Inc. refinery in Catlettsburg, Kentucky.

Except for the steam supply, this arrangement would allow the utility system of the pilot plant to be shared with that of the refinery. All process descriptions, equipment specifications, and planning drawings were completed for the coal preparation section. Work in the hydrogenation area involved the modification and rearrangement

of major equipment to conform to the physical characteristics of the new plant site. Preliminary process designs were also completed for the solid-liquid separation techniques of filtration, centrifugation, and solvent precipitation.

Ashland Synthetic Fuels, Inc. (ASFI) is responsible for the construction of the plant under contract No. EX-76-C-01-2260. Field work began in December 1976 by Badger Plants Inc., subcontractor to ASFI. The Lummus Company will design the antisolvent deashing unit for the pilot plant. HRI will provide engineering, design services, and field support as a subcontractor to ASFI during the construction and operation phases.

PROGRESS DURING OCTOBER-DECEMBER 1977

The H-Coal pilot plant was 30 percent complete at the end of the quarter. HRI met the December 15, 1977 target date for the Phase I Engineering effort with 99.6 percent of the 2,682 total drawings certified for construction. A 30-day run using Illinois No. 6 coal was successfully completed during the quarter on the Process Development Unit (PDU) as was a 30-day syncrude run on Wyodak coal. Both runs simulated syncrude operations of the pilot plant.

PDU Run 5 was completed in October simulating process conditions and operating objectives as closely as possible those for Illinois No. 6 syncrude operations of the pilot plant. The objectives of the run were successfully

achieved. Thirty-days of operation were successfully completed with catalyst replacement at a rate of one pound per ton with excellent stability and very few process and mechanical problems. Distillate equilibrium was achieved in ten days. Throughout the run, the addition and removal of catalyst was demonstrated using a remotely operated automatic valving system. The catalyst addition-withdrawal system performed according to the design concept with only minor problems. All target conditions were met with the exception of reactor residuum concentration. Only a 40 percent concentration was reached versus the targeted 44 percent due to temperature limitations of the slurry feed system.

Several modifications were made to the PDU prior to the starting of PDU Run 6 in November. The objective of Run 6 was to complete a thirty-day operation in the syncrude mode using Wyodak coal and simulating pilot plant conditions. There were two interruptions during the run when catalyst carried over into the let-down system. Early results from the run indicated a design yield of 47 percent C₄-950°F oils and conversions above 90 percent.

The drying equipment was not able to completely dry the Wyodak coal which has a high moisture level, and a wet coal was fed to the hot slurry system. However, up to 77 percent of the coal moisture was evaporated in the slurry mix system with no notable problems.

Modifications to the two bench-scale units continued throughout the quarter. Four catalysts were selected for testing under the program to study denitrogenation and improved-yield, higher-density catalysts. The program was scheduled to start in January.

III. DONOR SOLVENT LIQUEFACTION PROCESS

EXXON RESEARCH AND ENGINEERING
COMPANY

FLORHAM PARK, NEW JERSEY

Cooperative Agreement: No. EF-77-A-01-2893

Contract Funding: \$240,000,000
DOE: \$120,000,000
Industry: \$120,000,000

INTRODUCTION

The Exxon Donor Solvent (EDS) Coal liquefaction process produces distillate products from a wide range of coals. The development of the process represents a major contribution to the emerging coal liquefaction technology and is based on laboratory and engineering research and development efforts integrated with the operation of a 250 ton-per-day coal liquefaction pilot plant. The program features development of technologies for coal liquefaction, solids separation, hydrogen and process fuel generation, and for extensive product testing. Pursuance of these efforts will provide the basis for a process that is operable and reliable and with an excellent chance for commercialization. The project schedule is shown in Figure III-1.

The overall project objective is to develop the EDS process to the state of commercial readiness by 1982 by obtaining all of the data needed for a commercial plant design. This will be accomplished through the following more specific objectives:

- Develop the EDS process and design bases necessary to liquefy a range of different coals in an environmentally acceptable manner.
- Develop process correlations relating product yields and qualities to operating conditions and feed coals.
- Translate program results into estimates of commercial plant economics and operations.

- Identify and implement modifications to improve process economics and operability.
- Develop and demonstrate process technology at the scale necessary to insure a reliable commercial design.
- Conduct an aggressive product testing program to insure marketable products.

PROCESS DESCRIPTION

The chief features of the EDS Process are illustrated in the process schematic, Figure III-2. Crushed coal is liquefied in a non-catalytic tubular reactor in the presence of molecular hydrogen and the hydrogen-donor solvent. The liquefaction reactor operates at 800-880°F and 1500-2000 pounds per square inch.

The hydrogen-donor solvent is a 400°F-850°F boiling range material. The solvent is a catalytically hydrogenated recycle stream fractionated from the middle boiling range of the liquid product. After hydrogenation, the solvent is mixed with fresh coal feed and pumped through a preheat furnace into the liquefaction reactor. Slurry leaving the liquefaction reactor is separated by distillation into gas, naphtha, distillates, and a vacuum bottoms slurry. The vacuum slurry is coked to produce additional liquids.

PROJECT	FY 76	TR. Q.R.	FY 77	FY 78	FY 79	FY 80	FY 81	FY 82
PDU & PILOT PLANT PDU	△							▽
			○					
			△	▽				
				△	▽			
						△	▽	
								▽

LEGEND : △ BEGIN MILESTONE
▽ COMPLETE MILESTONE

Figure III-1. DONOR SOLVENT PROJECT SCHEDULE

Figure III-2. DONOR SOLVENT LIQUEFACTION PROCESS SCHEMATIC

The plant is "balanced" in that it is self-sufficient in both process fuel and hydrogen requirements. Process fuel and hydrogen are produced by gasifying the coke and by reforming C_1 - C_2 gases from the liquefaction process.

The process is simple, and critical processing steps are adaptations of Exxon's base in petroleum refinery tech-

nology. Distinguishing features are the decoupled configuration of the liquefaction and catalytic hydrogenation sections and the use of vacuum distillation for solids/liquid separation. The catalyst does not contact coal minerals or high-boiling liquids, thereby leading to longer catalyst life at high activity. Use of hydrogenated rather than unhydrogenated recycle solvent produces a

very significant improvement in process operability, particularly in downstream processing vessels. Also, hydrogenated solvent produces higher distillate product yields than unhydrogenated solvent. The use of mechanical separation devices for solids/liquids separation is avoided.

The process gives high yields of low-sulfur liquids from bituminous or subbituminous coals or lignites. For Illinois bituminous coal, the liquid yield is 2.6 barrels of C₄+ liquid per ton of dry coal. Ammonia and elemental sulfur are the only by-products of significance. By varying liquefaction conditions or adjusting solvent properties, product distribution may be varied over a wide range.

The Laboratory Process Research and Development studies were conducted at the Baytown, Texas, Research and Development Division; the Products Research Division at Linden, New Jersey; and the Exxon Research and Development Laboratories in Baton Rouge, Louisiana. The Engineering Research and Development studies were performed at the Synthetic Fuels Engineering Department and Exxon Engineering Technology Department at Florham Park, New Jersey.

HISTORY OF THE PROJECT

Phase I of EDS Process Development (1966-1973)

Phase I of Exxon's research, the Predevelopment Phase, spanned the period 1966 through 1973, and concentrated on identifying the basic process flow scheme for coal liquefaction. This research was very broad in scope and was conducted in experimental equipment ranging in complexity from 100 cubic centimeter batch units to a continuous 1/2-ton-per-day integrated pilot plant.

The following reactor configurations were evaluated: stirred tank, tubular with external and internal recirculation, ebullating bed, and tubular plug flow. Both hydrogenated and unhydrogenated recycle solvents were evaluated for both bituminous and subbituminous coals. Several solids/liquids separation techniques were studied, such as distillation, centrifugation, filtration and hydroclones. Wide ranges of temperatures, pressures and residence times were evaluated in both the liquefaction and solvent hydrotreating reactors. Based on these results, a hydrogenated recycle solvent process was selected for further development, and vacuum distillation was identified as the most promising product-separation technique. A noncatalytic, tubular plug-flow liquefaction reactor was selected. These choices circumvented the need for ash-tolerant catalysts and mechanical separation devices.

Engineering studies closely paralleled the laboratory effort during Phase I. These engineering studies concentrated on the following areas.

- Evaluation of laboratory data.
- Development of conceptual commercial plant designs.
- Studies of processing alternatives to define incentives and provide direction to the laboratory effort.
- Development of cost parameters.

Phase II of EDS Process Development (1974-1975)

Phase II of Exxon's research, the planning and design Phase, was conducted during 1974-1975. The major efforts during this phase were concentrated in the following areas.

- The basic design of the 250-ton-per-day Exxon Coal Liquefaction Pilot Plant (ECLP).
- The construction and initial operation of the one ton-per-day Coal Liquefaction Pilot Plant (CLPP).
- The supporting laboratory and engineering R&D program.

The laboratory and engineering R&D program more closely defined operating conditions for the process flow scheme selected in Phase I. This program concentrated on process variable studies of both bituminous and subbituminous coal. Wide ranges of temperature, pressure, residence time and solvent/coal ratio were studied. In addition, detailed investigations were conducted of solvent hydrogenation in fixed-bed catalytic reactors.

PROGRESS DURING OCTOBER-DECEMBER 1977

Summary

Studies continued during the quarter with Illinois No. 6 coal in the one-ton-per-day Coal Liquefaction Pilot Plant (CLPP). Solvent quality studies continued in the 50 pound-per-day Recycle Coal Liquefaction Unit on Wyoming coal. Supporting studies completed during the quarter included process variable screening studies on the two-train catalyst testing unit, Integrate Coker/Gasifier Pilot Plant (IKG) operation on CLPP Illinois coal liquefaction vacuum bottoms, and operations of the Large Stirred Coking Unit. Engineering and procurement work on the Exxon Coal Liquefaction Pilot Plant was approximately 12.6 percent completed at the end of the quarter.

Coal Liquefaction Pilot Plant

Studies continued on the one-ton-per-day Coal Liquefaction Pilot Plant (CLPP) with Illinois No. 6 bituminous coal in October. Ten yield periods were completed at liquefaction operating conditions to be used in the updated commercial study design. The average elemental balance yields for these runs were:

Yields, Lbs/100 Lbs Dry Coal	
H ₂	-3.3
H ₂ O	8.9
CO, CO ₂	1.4
NH ₃	0.4
H ₂ S	3.2
C ₁ -C ₃ Gas	6.6
C ₄ -400°F Naphtha	18.2
400-700°F Middle Distillate	7.7
700°F+ Heavy Distillate	7.2
1000°F+ Residual Bottoms	49.7

A high severity vacuum flash study was completed at temperatures up to 735°F. Also, the CLPP slurry viscometer was operated in-line with the feed preheaters. An expected, slurry viscosity peaking occurred in the 600-645°F temperature range. The unit was shut down in November for an extended maintenance turnaround.

Recycle Coal Liquefaction Unit

Process variable studies continued during the quarter with Wyoming subbituminous coal in the 50 pound-per-day Recycle Coal Liquefaction Unit (RCLU). The variables investigated were liquefaction temperature and solvent quality. The solvent quality (SQI) was decreased by lowering the hydrotreater temperature. Preliminary results indicated that Wyoming coal conversion was significantly affected by solvent quality. As the hydrotreater temperature was decreased to 90°F below the typical operating temperature, the 1000°F conversion decreased by about 14 weight percent.

The solvent quality runs also investigated the effects of increased solvent-to-coal ratio and liquefaction residence time. Preliminary results indicated that with low quality

solvent, Wyoming coal conversion increased from 35 weight percent to 43 weight percent as the solvent-to-coal ratio was raised to about twice the standard value.

The 100 pound-per-day RCLU-2 was recommissioned during November. Investigation began on the unit to determine the effectiveness of sulfur dioxide pretreatment of Wyoming coal as a method of reducing calcium carbonate deposition in the liquefaction reactor and consequent effect on operability and yields.

Supporting Studies

A process variable screening study utilizing Ni/Mo-10 and Co/Mo-20 catalyst and Wyoming spent solvent was completed on the solvent hydrogenation two-train catalyst testing unit. The variables investigated were start-of-run temperatures and space velocities.

The Large Stirred Coking Unit achieved extended operation on Wyodak Liquefaction bottoms in October and operations were completed in November. Sufficient coker liquids were produced to satisfy all Phase III commitments for both Illinois and Wyodak products.

Integrated Coker/Gasifier Pilot plant (IKG) operations on CLPP Illinois coal liquefaction vacuum bottoms were also terminated in November. Analysis of the results indicated that well over 80 percent of the original coke charge was displaced, and the end-of-run coke ash content exceeded 80 percent of equilibrium ash for the level of gasification. Slagging emerged as the primary study area.

Engineering support activities for the Exxon Coal Liquefaction Pilot Plant (ECLP) continued with the development of information for use by the Baytown Refinery in further confirmation of the impact of waste streams generated by ECLP which will be disposed of in existing refinery facilities. Also completed was engineering support work on the Texas Air Control Board Construction Permit Application which the Carter Oil Company must submit and have approved before ECLP construction can begin. The engineering and procurement work on the ECLP was approximately 12.6 percent complete at the end of the quarter.

IV. COMMERCIAL SCALE EXPANDED-BED HYDROPROCESSING OF SOLVENT REFINED COAL EXTRACT

CITIES SERVICE RESEARCH AND
DEVELOPMENT COMPANY

TULSA, OKLAHOMA

Contract No.: EF-77-C-01-2038

Contract Funding: \$1,982,000

INTRODUCTION

Cities Service, under DOE sponsorship, is determining the operating conditions and modifications necessary to utilize an existing LC-Fining unit (expanded-bed hydroprocessing unit) to upgrade a production quantity of Solvent Refined Coal (SRC) extract.

Present operations are being conducted at the New Brunswick process development unit site of C. E. Lummus, subcontractor for operations for Cities Service. After completion of the present tasks, experimentation will begin at Cities Service Lake Charles, Louisiana plant. The specific objectives of this contract are to:

- Determine the expanded bed conditions required to hydrotreat SRC extract at severity levels ranging from desulfurization to extensive hydrocracking.
- Upgrade a production quantity of SRC extract from the Ft. Lewis pilot plant using commercial-scale expanded-bed hydroprocessing equipment.
- Demonstrate that the SRC process and expanded-bed hydroprocessing can be combined to provide a valuable liquid fuel or refinery feedstock from coal.

PROCESS DESCRIPTION

Solvent Refined Coal (SRC) extract is a hydrocarbon mixture containing significant quantities of oxygen, nitrogen, sulfur, and ash. SRC extract composition and

properties are similar to heavy sour petroleum crude and process-derived residua. These are problem stocks which must be upgraded by treatment with hydrogen before they can be further refined to liquid finished products. Expanded-bed hydrotreating can upgrade these stocks and maintain continuous operation for extended periods of time as well. A flow diagram of the process is shown in Figure IV-1.

The SRC extract, together with hydrogen, is fed upward through the reactor as a liquid/gas mixture at a velocity such that the catalyst is in continuous motion. Part of the reactor effluent is recycled back through the reactors to maintain the necessary liquid velocity through the reactor system (the expanded-bed). Because of the movement of catalyst particles in the liquid/gas medium, deposition of tar and coke is minimized and fine solids entrained in the feed will not lead to reactor plugging.

Figure IV-1. EXPANDED-BED HYDROPROCESSING PROCESS SCHEMATIC

Reactor effluent is cooled by exchange, and a vapor-liquid separation is made. After scrubbing, the hydrogen is recycled and liquid product is either stored or further fractionated.

Bench-scale pilot plant studies will be performed for the purpose of determining operational conditions for the commercial unit. Engineering studies will define the degree of effort and resources required to adapt a commercially available expanded-bed unit to process a large quantity of SRC extract.

HISTORY OF THE PROJECT

Active work on the commercial scale expanded-bed hydroprocessing of Solvent Refined Coal (SRC) extract began on June 1, 1976 with bench-scale pilot plant operations. The primary contract was extended for an additional 18 months, commencing June 1, 1977 under contract EX-76-C-01-2038. An Interim Technical Progress Report was published in November 1977 and is available from NTIS, Springfield, Virginia.

PROGRESS DURING OCTOBER-DECEMBER 1977

Summary

On the basis of screening runs completed during the quarter, a commercially available denitrogenation/desulfurization catalyst was selected as the candidate for a 30-day catalyst aging run that began in December.

Discussion of Activities

Two additional catalyst screening runs were conducted in October, completing the 10-day catalyst screening program. A total of six catalysts were tested in the program: three nickel-molybdenum, two cobalt-molybdenum, and one nickel-tungsten catalysts.

A comparison of the liquid product properties for the five commercially available catalysts showed the following generalized results for the nickel-molybdenum (Ni/Mo) systems:

- Greater conversion of 850°F+.
- More selectivity in production of 500-600°F fraction.
- Small increase in the production of IBP-500°F.
- Better hydrogenation, oxygen removal, and denitrogenation of all heavy oil fractions.

The nickel-molybdenum catalyst (Ni/Mo-A) was chosen for the 30-day aging study after comparing the conversion and level of nitrogen in the total liquid product for the six catalysts. The Ni/Mo-A was superior to the other catalyst tested in this selection process. The conversion of the 850°F+ material was a preliminary value for the conversion, based only upon the weight percent 850°F+ fractions of the feed and oil products.

The second 30-day aging run (LCF-36) began in December. After 27 periods of operation (a period constituted 24 hours of operation equivalent to a 24-hour day), the conversion of 850°F+ was approximately 61-62 weight percent. The nitrogen level in the heavy oil product was representative of the entire oil product which contained unconverted 850°F+ material with its fraction nitrogen content of approximately 1.0 weight percent.

The distribution of product fractions remained essentially constant throughout the entire 27-period operation. The 500-650°F and 650-850°F fractions were approximately 42 and 28 volume percent of oil, respectively. The 850°F+ specific gravity increased slightly from approximately -11° API to -14° API.

From the second period of operation through the twenty-seventh period operation, the temperatures were increased only 20°F (from 780°F - Period 2 to 800°F - Period 27). The periodic temperature increases were predicated upon the desire to maintain a relatively stable nitrogen content in the heavy oil product.

One of the goals of the processing of SRC is the optimization of the 650°F-850°F fraction of the oil product. An optimum amount of this fraction, together with a low sulfur/nitrogen content, would provide not only a desirable feedstock for downstream processing by conventional petroleum refinery process schemes, but would also provide a clean fuel for combustion. Conversely, the production of light boiling fractions of the oil product should be minimized.

Run LCF-36 was scheduled to be terminated in January 1978, completing Subtask I of the contract extension.

V. CRESAP TEST FACILITY

FLUOR ENGINEERS AND CONSTRUCTORS,
INC.

LOS ANGELES, CALIFORNIA

Project Site: Cresap, West Virginia

Contract No.: EX-76-C-01-1517

Contract Funding: \$54,790,475

DOE: \$51,998,595

Industry: \$ 2,791,880

INTRODUCTION

Under contract to DOE, Fluor Engineers and Constructors, Inc., is renovating and will operate a DOE-owned facility in Cresap, West Virginia. Industrial financing is being provided by American Electric Power Corporation (\$1 million), Allegheny Power Corporation (\$1 million), and Fluor (\$657,640). This facility, inactive since 1970, contains much of the equipment required for coal liquefaction. Fluor is to (1) renovate the Cresap facility to test the various critical mechanical components required for coal liquefaction and (2) operate the renovated facility to demonstrate an integrated process for the production of low-sulfur liquid fuel. The project schedule is provided in Figure V-1.

PROCESS DESCRIPTION

In this process, shown schematically in Figure V-2, coal is converted to low-sulfur liquid fuel. The coal is first crushed to 100-mesh and then combined with aromatic solvent in a mixer. The coal-solvent slurry is then pressurized and preheated. The slurry passes to an extractor where coal dissolution occurs. The product stream is then

treated in a liquid-solid separation unit where the coal extract and solvent are removed, leaving a thickened slurry. This stream is sent to a carbonizer where the material is converted to char, oil, and gas. The oil is recycled to the liquid-solid separation unit and the char and gas are recovered.

One of the more persistent problems encountered in coal liquefaction processes is the separation of solids from the liquid product streams. At the Cresap Test Facility, several methods of separation will be tested. One of the most reliable methods for concentrating the solids is a system of hydroclones which was successfully used during prior pilot plant operations. Other processes to be evaluated will be filtration and chemical precipitation.

The coal extract and solvent leaving the liquid-solid separation unit are heated and fed into a flash still where the heavy components of the coal extract are separated from the light liquids. The light liquids are then fractionated into light and middle distillates. The middle distillate may either be recycled as a component of coal solvent or recovered as product fuel oil. The heavier components of the coal extract are heated in a fired preheater, hydrocracked and fractionated into three distillate fractions. The middle distillate is used as coal solvent and the heavy distillate is recovered as fuel oil product.

LEGEND:

 ▲ BEGIN MILESTONE
 ▼ COMPLETE MILESTONE

 ○ DECISION MILESTONE
 ● EVENT

 5 ORDER LONG-LEAD ITEMS

Figure V-1. CRESAP TEST FACILITY PROGRAM SCHEDULE

Figure V-2. LIQUID FUEL FROM COAL PROCESS SCHEMATIC

HISTORY OF THE PROJECT

Reactivation of the Cresap facility to develop an integrated process test center for producing low-sulfur liquid fuel from coal was initiated in May 1974. Since then, the facility has been upgraded to meet environmental requirements and current design standards of piping, electrical, and mechanical equipment codes. A task force has been established to organize, coordinate, and direct the testing program.

Renovation of the pilot plant involved the refurbishing of existing equipment and the installation of new equipment to replace outmoded or worn out equipment. Inquiry packages were developed for most of the new equipment, and flow diagrams and specifications were prepared for the refurbishing of some of the existing equipment. The Liquefied Coal Development Corporation, a wholly-owned subsidiary of Fluor, was formed during 1975 to operate the pilot plant.

PROGRESS DURING OCTOBER-DECEMBER 1977

Four extraction operation runs were conducted during the quarter. The first run in October lasted less than one

hour. It was terminated when the slurry preheater coked due to a leak-induced flow interruption. Run 002 was a 24 hour run that was terminated when the concentrated extract could not be removed. In Run 003, coal was fed for 144 hours before the run was terminated because of the inoperability of the extract granulation system. Thirty-two tons of coal were processed in an 11.5 percent slurry. Conversion averaged 62 percent, and more than one hundred 55-gallon drums of extract-rich intermediate were produced. Run 004 was abruptly halted after 55 hours of operation when the lower agitator shaft seal failed. During the run, satisfactory operation of the revised extract granulation system was demonstrated.

In December, warm solvent was circulated through most of the hydrotreating section of the pilot plant under a 2700 psig nitrogen blanket. Leaks at the heads of the absorber and the entrainment separator persisted. Maintenance and modification was scheduled for the hydrotreating section in January.

Phenols are produced during the liquefaction process, and during the quarter, an extensive effort was underway to improve the effectiveness of removing phenols from the industrial waste water.

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VI. SYNTHOIL PROCESS

FOSTER WHEELER ENERGY CORPORATION
LIVINGSTON, NEW JERSEY

Contract Funding: \$24,425,776

Plant Site: Bruceton, Pennsylvania

INTRODUCTION

Development of the Synthoil process, initiated by the U.S. Bureau of Mines, is currently being managed by DOE through the Pittsburgh Energy Research Center (PERC) in Pennsylvania. The objective of this project is to determine the technical and economic feasibility of the process for scale-up to commercial use. Foster Wheeler Energy Corporation is responsible for designing and managing the construction of a ten-ton-per-day process development unit to test the Synthoil process. PERC is conducting support research for the design of the unit. The schedule for the Synthoil process is shown in Figure VI-1.

PROCESS DESCRIPTION

The Synthoil process is a hydrodesulfurization process that converts high-sulfur coal to a low-sulfur, low-ash synthetic fuel oil. A schematic of the process is shown in Figure VI-2. The coal is first crushed to 70 percent through 200-mesh and dried to approximately two weight percent moisture. It is then mixed with process product oil to form a slurry of 35-40 percent coal. The resulting slurry is then combined with hydrogen and fed into a fired preheater. From the preheater, the slurry enters a fixed-bed catalytic reactor packed with catalyst pellets of cobalt molybdate on a silica-alumina base. The product mixture is then cooled and the liquid and unreacted solids are separated from the gases.

Hydrogen propels the slurry so violently through the fixed-bed catalytic reactor that plugging of the bed by the mineral matter in the coal is prevented as the coal becomes liquefied.

The liquids and unreacted solids leave the bottom of the separator and pass into a centrifuge. The solids are separated and may be fed into a pyrolyzer. Part of the liquid leaving the centrifuge is recycled to the mixer and the remainder is drawn off as fuel oil product. The solids are further pyrolyzed to yield additional quantities of product oil and an ash residue. This residue, which contains some carbonaceous material, will be sent to a gasifier and shift converter to produce additional hydrogen in any commercial operation.

The separator off gases are sent through a gas purification system where they are separated into five product streams: hydrogen sulfide, water, ammonia, hydrocarbon gases, and hydrogen. The hydrogen sulfide is sent to a sulfur recovery system yielding elemental sulfur as the product. The hydrocarbon gases are fed to the gasifier and shift converter. The hydrogen stream which is mixed with the hydrogen produced in the gasifier is fed into the slurry stream entering the preheater.

HISTORY OF THE PROJECT

Work on this process began in 1969 at PERC. The initial work on the Synthoil process used a reactor with an internal diameter of 5/16-inch in a bench-scale plant

Figure VI-1. SYNTHOIL PROGRAM SCHEDULE

Figure IV-2. SYNTHOIL PROCESS SCHEMATIC

that processed five pounds of slurry per hour. Experimental work was conducted on various coals, such as Pittsburgh seam, Indiana No. 5, Middle Kittenning Ohio No. 6, and Kentucky strip coal. All of these types of coal were satisfactorily converted to low-sulfur fuel oil with no significant attrition of the catalyst. Experiments were conducted to study the effects of hydrogen flow rate, the percentage of coal in the feed slurry, the recycle rate of the product oil, and the effect of hydrogen sulfide in the recycle gas.

To demonstrate the broad applicability of the process, a 1/2-ton-per-day slurry unit was constructed that used a reactor of 1.1-inch ID made of two interconnected stainless steel pipes that were each 14.5 feet long. The operations were carried out on several types of coal at reactor pressures of 140 atm and 280 atm and 840° F. High yields of low-sulfur and low-ash fuel oil were obtained. At the lower reactor pressure the calorific value, product oil yield, and hydrogen consumption were also lower. However, the sulfur and ash content of the

product oil were higher than that of the oil produced at 280 atm. Economic analyses will decide the appropriate pressure for this process, considering the capital and operating costs for high-pressure and low-pressure operations, and the environmentally acceptable sulfur and ash contents of the product oil.

The overall design package for the process development unit was completed during 1975. In addition, long lead equipment was placed on order. Actual field construction of the facility began during the fourth quarter of 1975.

PROGRESS DURING OCTOBER-DECEMBER 1977

Because of late delivery of equipment, the mechanical completion of the 10 ton-per-day PDU was estimated at June 30, 1978.

Experiment FB-62 was begun on the 1/2 ton-per-day unit in October and continued until mid-December when the plant was voluntarily put on standby for restart in January. During the run, a product which was low in sulfur and liquid at room temperature was produced for 1,440 hours of continuous uninterrupted operation. The objectives of this run were to make a long-term baseline noncatalytic run with a low-ash coal for direct comparison

with the previous experiment with high-ash (16.5 percent) Kentucky coal and to demonstrate the long-term operability and reliability of the plant. These objectives were accomplished with West Virginia coal (9.9 percent ash) at process conditions of 455°C and 4000 psi. The plant configuration consisted of a 1.1 inch ID by 42-foot unpacked preheater and a 1.1 inch ID by 29-foot reactor packed with 1/8 inch cylindrical pellets of inert Pyrex glass. The experiment indicated that the process would be applicable to any bituminous coal.

During the run, several hundred pounds of gross liquid product were also processed in the new continuous centrifuge and used for recycle oil. This experiment demonstrated that the continuous centrifuge could be integrated into normal plant operations to replace the semi-continuous centrifuge currently being used. Three additional experiments were conducted in the continuous centrifuge using feeds produced by the PDU at different conditions than that previously tested, and therefore having different properties. No difficulties were experienced because of the changes. Viscosity changes were less in the continuous centrifuge than in the batch centrifuge.

Construction of the new Synthoil-II unit continued and was about 25 percent complete by the end of the quarter.

Sixty-five additional experiments were completed with the heat transfer reactor model. Shakedown testing of the cold model, which will be used to verify flow regime determinations, was completed.

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VII. CLEAN COKE PROCESS

USS ENGINEERS AND CONSULTANTS, INC.
UNITED STATES STEEL CORPORATION
PITTSBURGH, PENNSYLVANIA

Plant Site: Pittsburgh, Pennsylvania
Contract No.: EX-76-C-01-2220
Contract Funding: \$11,792,329
DOE: \$8,337,176
Industry: \$3,455,153

INTRODUCTION

The Clean Coke process is being developed by USS Engineers and Consultants, Inc., a subsidiary of United States Steel Corporation, under the sponsorship of DOE. The contract was initiated in 1972 under the auspices of the Office of Coal Research (OCR, first a part of ERDA, subsequently DOE). The objective of the project is to develop a process design that is capable of converting low-grade, high-sulfur coal to low-sulfur, low-ash metallurgical coke, chemical feedstocks, and liquid and gaseous fuels. The pilot plant design will be based on the results of laboratory tests and data obtained from process development units. The schedule for developing the process is shown in Figure VII-1.

PROCESS DESCRIPTION

The Clean Coke process is shown schematically in Figure VII-2. This process combines coal carbonization and hydrogenation in an environmentally acceptable manner to produce solid, liquid, and gaseous fuel products. No external hydrogen source is required and only minimal amounts of external energy will be consumed.

PROJECT	FY 74	FY 75	FY 76	TR. OR.	FY 77	FY 78
PROCESS DEVELOPMENT UNIT INPUT: 1/2 TON/DAY OUTPUT: 1.5 BARRELS/DAY	OPERATION	EVALUATION				
EVALUATE 2 OTHER COALS						

LEGEND:
△ BEGIN MILESTONE
▽ COMPLETE MILESTONE

Figure VII-1. CLEAN COKE PROCESS SCHEDULE

The feed coal is first pulverized and classified into two portions. One portion is sent to the carbonizer. The coal is pyrolyzed in a fluidized bed yielding char, liquid, and a hydrogen-rich gas which is subsequently cleaned and recycled to fluidize the bed. The hydrogen-rich atmosphere of the bed provides a hydrodesulfurization environment that reduces the high-sulfur coal to low-sulfur char. This char is then pelletized using a process-derived heavy oil binder and the resulting pellets are cured and calcined in a kiln. The products are low-sulfur metallurgical coke and a hydrogen-rich gas.

The other portion of the coal is fed to the hydrogenation unit where it is slurried with a medium-weight oil recycled from the liquid processing section. This slurry is

Figure VII-2. CLEAN COKE PROCESS SCHEMATIC

reacted with hydrogen recovered from the gas treatment section. The non-catalytic hydrogenation reaction is conducted at approximately 900°F and 270 atmospheres.

The products of the reaction are sent to a vapor stripper where the lighter components in the slurry are vaporized by counter-current contact with hot process gas, leaving an ash which is removed through a lock hopper. The vapor/hot gas stream from the stripper unit is fed to a condenser; the resulting liquid stream is further treated in the liquids processing section, and the uncondensed gas is sent to the gas treatment section.

Gases from all operations are treated in a common facility, which includes ammonia and acid gas removal, a sulfur production unit, an ethylene plant, and a cryogenic gas separation system.

The products from a liquids processing unit are light, medium, and heavy oils. The lighter oil is treated to produce gas, gasoline, benzene, naphthalene, and residual tars. The medium oil is recycled as slurry oil, and the heavy oil is used as a coke binder.

Two major advantages of the Clean Coke process design are that hydrogen is produced for the liquefaction portion and that the carbonization char is used to

produce metallurgical-grade coke from high-sulfur, high-ash coal.

HISTORY OF THE PROJECT

Laboratory and bench-scale development studies were initiated by U.S. Steel in 1969, using Illinois No. 6 seam coal to establish the technical feasibility of the Clean Coke process. Emphasis was placed on coal preparation, carbonization-desulfurization of coal in fluidized beds, and high-pressure hydrogenation of coal. The effects of temperature, pressure, reaction time, and type of feed on the carbonization reaction were investigated. The purpose of the bench-scale hydrogenation studies was to determine the effects of reaction time, temperature, and pressure on the conversion to liquid and gaseous products. Preliminary investigations of coke production were also conducted.

Under contract to the government since 1972, U.S. Steel is operating the process development units. Total carbonization process development unit (PDU) operating time completed by the end of 1976 was 3,667 hours. The hydrogenation PDU was operated during the last

quarter of 1976, with an extended run planned for the beginning of 1977.

PROGRESS DURING OCTOBER-DECEMBER 1977

Summary

The carbonization process development unit (PDU) program for West Kentucky coal was completed during the quarter. Operation of the PDU with Pittsburgh seam coal was started, and a shakedown run was completed. Preparation and testing of binder materials and form-coke specimens also continued.

Carbonization Process Development Unit

The carbonization PDU program on West Kentucky coal was completed during the quarter, including duplicate material balance runs through both stages of carbonization. West Kentucky coal, preoxidized at 400° F, was first processed at 820° F, and the resulting semi-char was then processed at 1400° F to produce char having a sulfur content of 0.75 percent.

Carbonization of the coal produced the following yields in weight percent:

	<u>Yields, weight percent</u>
Char	63.4
Tar	11.5
Water	4.4
C ₁ to -C ₄ gas	7.8
Hydrogen	0.5
Acid gas	11.9
Ammonia	0.4

An initial PDU run with highly caking Pittsburgh Seam coal was completed without agglomeration problems by using fluidizing gas containing three percent oxygen in steam. Free-flowing Pittsburgh semi-char was produced in a fluid bed at 860° and 24 psia and with a 46-minute residence time. Prior to the run, operating conditions suitable for processing Pittsburgh coal without bed agglomeration were demonstrated in the three-inch bench unit.

Evaluation of formcoak pellets produced with several types of binder and Illinois char in the coke-preparation PDU during the quarter indicated that untreated hydrogenation-derived binder yielded weaker coke than did binders containing high concentrations of carbonization-derived heavy oil. However, bench studies showed that the quality of the hydrogenation heavy oil was significantly improved by air-blowing for two hours at 300° C followed by heat treating for four hours at 380° C. Form-coke specimens prepared with the treated binder had an average crushing strength of 10,825 psi, compared with 8,375 psi for specimens made with untreated binder.

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VIII. LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

ROCKETDYNE DIVISION
ROCKWELL INTERNATIONAL CORPORATION
CANOGA PARK, CALIFORNIA

Plant Site: Canoga Park, California
Contract No.: EX-76-C-01-2044
Contract Funding: \$994,375
(100% DOE)

INTRODUCTION

Under contract to DOE, the Rocketdyne Division of Rockwell International is conducting a research program to develop a technology to liquefy coal via direct hydrogenation. This technique would involve rapid mixing, reaction, and quenching of a gaseous hydrogen and pulverized coal mixture to form hydrocarbon liquids. These products would be suitable for conversion to fuel oil or chemical feedstocks.

The program is divided into two phases. Phase I involves the design and fabrication of cold-flow equipment and the operation of the equipment in a series of tests to evaluate methods of transport and injection of pulverized coal into the reactor.

Upon completion of Phase I, the results of the cold-flow tests were submitted to DOE for a decision on proceeding with Phase II. Upon Phase II authorization, small-scale reactor testing was initiated to evaluate the effects of reactor configuration and operating conditions. Based on these results, a larger reactor, with diverse injection methods and a coal flow rate of approximately one-ton-per-hour, will be built and tested. Test results and analyses will be evaluated to determine the feasi-

bility of this process. The schedule for the development of this process is shown in Figure VIII-1.

In addition to the DOE funded effort, Rockwell International is sponsoring a corollary effort to provide supporting facilities.

PROCESS DESCRIPTION

The basic concept of this process for the liquefaction of coal is that high liquid yields are favored by rapid mixing, reaction, and quenching of a mixture of pulverized coal and hot gaseous hydrogen. In this process, as shown in Figure VIII-2, coal is fed into the coal feeder from a batch feeder, with the transport gas. The coal passes through the feeder, is partially fluidized with hydrogen at the conical exit, and then passes to the entrained-flow reactor where it is mixed rapidly with 1500°F hydrogen. The pulverized coal and hydrogen react for about 10 to 100 milliseconds. The reactor is expected to be operated over a pressure range of 35 to 100 atmospheres and a temperature of approximately 1800°F.

The reactor effluent is passed into a quench section which has a set of water spray nozzles. From there the effluent passes into a char receiver.

Figure VIII-1. PROGRAM SCHEDULE FOR LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

Figure VIII-2. SCHEMATIC OF LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

HISTORY OF THE PROJECT

Development of a process for the liquefaction of coal by direct hydrogenation was started in August 1975. Phase I testing was completed in 1976, and Phase II testing began. The first 1/4-ton-per-hour test was conducted at the end of May, and final testing on the unit was completed by the end of 1976. Results of the tests allowed a confident transition to the one-ton-per-hour system and indicated the direction for testing at the larger scale.

Construction of the one-ton-per-hour test facility was completed in December 1976, and preparation for testing began.

PROGRESS DURING OCTOBER-DECEMBER 1977

Summary

Phase III of the contract was initiated in August and continued during this quarter with 20 additional reactor tests made on the one-ton-per-hour reactor. Residence time and reactor hydrogen requirements were studied. A new injector, allowing injection of hydrogen at higher temperatures, was designed, built, and tested. Successful operation was demonstrated with a hydrogen-to-coal flow rate ratio as low as 0.22, economically significant for a commercial plant.

Discussion of Activities

All tests during the quarter were made with Kentucky No. 9 coal. Three of the tests were unsuccessful due to operational and hardware problems.

Test 32 was performed in October with the 3.37-inch I.D. reactor tube. Following the test, the reactor tube and injector assembly were destroyed because of operator error. A new injector was designed and built to allow a higher hydrogen injection temperature than was possible with the configuration used previously. With the old injector, oxygen could be introduced in the preburner or into the reactor directly. Results from the 1/4-ton-per-hour testing showed that injection of oxygen directly into the reactor was detrimental to the product yields, and much higher conversions to oxides of carbon were obtained. With oxygen injection in the preburner, the allowable injection temperature was limited by the material strengths to about 2150°F. The new injector was designed to allow oxygen addition within the hydrogen injection passages and close to the injector face. A configuration was chosen to allow complete reaction of the oxygen within the hydrogen flow before it reached the coal, but with minimum heat load on the injector. A water cooling passage was also built into the injector face. However, the preburner was retained to allow oxygen addition in that mode as well.

In November, Test 37 through 42 were made with the preburner mode of oxygen addition and the 4.87 inch I.D. reactor. These tests were directed toward determining if suitable high carbon conversions could be obtained at lower reactor temperatures by lengthening the reactor residence time relative to previous testing.

Twelve tests were made in December with the 1.37-inch I.D. reactor tube and all with the injector operated in the high temperature mode. The tests were directed toward evaluating this mode of operation and, also, toward evaluating reactor behavior with very short residence times.

For tests 25 through 42, the average carbon balance was 93 percent. A distillation analysis of the total liquid product from two test shows a much larger proportion of light ends than in coal derived liquids from other processes.

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IX. FLASH PYROLYSIS COAL LIQUEFACTION PROCESS

OCCIDENTAL RESEARCH CORPORATION

LA VERNE, CALIFORNIA

Plant Site: La Verne, California

Contract No.: EX-76-C-01-2244

Contract Funding: \$3,780,000
(100% DOE)

INTRODUCTION

The Occidental Research Corporation (ORC), a wholly-owned subsidiary of Occidental Petroleum Corporation, will evaluate the commercial potential of its flash pyrolysis coal liquefaction process under the sponsorship of DOE. Figure IX-1 provides the schedule for the project.

The main objectives of the program are to:

- Demonstrate that caking coals can be processed continuously in a specially designed single-stage pyrolysis reactor without oxidative pretreatment, and that this method will result in a significantly higher yield of liquids than other proposed pyrolysis processes.
- Conduct extended runs in the three-ton-per-day process development unit (PDU) in order to obtain steady state heat and material balances.
- Produce and recover large quantities of the primary tar, and to evaluate methods for upgrading this material to a clean fuel or synthetic crude oil.
- Continue development of specific areas of the pyrolysis and liquids collection systems to ensure a technologically sound basis for future scale-up.
- Obtain sufficient process and environmental data for detailed design of a larger plant and conduct an assessment of the potential commercial viability of the process.

Figure IX-1. FLASH PYROLYSIS PROJECT SCHEDULE

PROCESS DESCRIPTION

The ORC process consists of rapidly pyrolyzing coal particles at a temperature of less than 1400°F in an entrained stream of hot coal char and a gas, substantially free of oxidizing constituents. The process features short residence time of coal particles thereby increasing the throughput per unit of cross sectional reactor area, and producing a relatively high yield of liquid products. Figure IX-2 provides a schematic flow diagram of the process.

After the coal is extensively milled and screened, it is transported to the pyrolysis reactor by heated nitrogen at rates of up to 300 pounds per hour. In the reactor, the

Figure IX-2. FLASH PYROLYSIS PROCESS FLOWSHEET

coal is mixed with recycled hot char which rapidly heats the coal to a pyrolysis temperature in the range of 950° to 1350°F. The char circulation rate ranges from five to ten times that of coal.

Char is separated from the pyrolysis vapors by a series of three cyclones. A portion of the char is cooled as product, and the remainder is fed to the char heater where it is partially burned with air to raise its temperature to 1200°F to 1700°F for recycle to the reactor. The second and third stage cyclones remove fine char from the pyrolysis vapor prior to quenching in the tar collection system. This system consists of three subsections: two tar recovery stages and a vacuum flash unit.

In the first recovery stage, raw pyrolysis gas is quenched to about 210°F, most of the heavy tar vapors are condensed, and any entrained char is removed. The pyrolysis gas is cooled to approximately 80°F in the second recovery stage, where water and light oils are removed. The vacuum flash unit is used to separate the quench liquid and recovered tars into high and low boiling fractions, the high boiling fraction representing the main product tar.

HISTORY OF THE PROJECT

The three-ton-per-day process development unit (PDU), constructed by ORC for development of pyrolysis processes, was revised and improved for use in this contract, awarded in June 1976.

Occidental Research Corporation (ORC) and the Commonwealth of Kentucky have a cost-sharing joint venture aimed at providing a detailed design for a 250-ton-per-day pilot plant. Subsystem tests and three shake-down runs were completed during 1976 on the PDU.

PROGRESS DURING OCTOBER-DECEMBER 1977

Summary

Work continued during the quarter to demonstrate the operability of the pyrolysis reactor on caking bituminous

coal. All eight runs completed during the quarter were made with Kentucky No. 9 coal. Progress was made in reducing circulating char upsets which have hindered operations in the past, but the basic problem remains. The process development unit was down for maintenance and revisions at the end of the quarter.

Process Development Unit

Two runs were conducted in October. The second of these runs was significant in that it confirmed the reactor design model under more stringent operating conditions than in previous runs. Relatively coarse (60 mesh top size) coal was fed for 22 hours at a feed rate up to 160 pounds-per-hour and at 1200°F. Both runs were terminated due to loss of circulating char inventory. There was no evidence of reactor fouling in either test.

Five runs were made in November. Twenty-five hundred pounds of Kentucky No. 9 coal were fed at rates up to 190 pounds-per-hour for short periods. Progress was made in reducing circulating char upsets caused by pressure variations which had hindered operations in the past, but the basic problem remains. The char storage bin was fluidized, and a fluidized lift pipe was installed to replace a mechanical feeder used to feed char to the burner loop.

Operations in November were hampered by ten mechanical failures which resulted in forced shutdowns. Most involved weld or valve failures. The unit was shutdown in December for maintenance and further revisions to improve char circulation.

Provisions have been made to substitute carbon dioxide and steam as the carrier gas for the PDU in place of nitrogen in an attempt to return process liquid yield to the expected high values.

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X. FILTRATION PROCESS AND EQUIPMENT STUDIES

JOHNS-MANVILLE SALES CORPORATION
DENVER, COLORADO

Project Site: Denver, Colorado
Contract No.: EX-76-C-01-2007
Contract Funding: \$190,000
(100% DOE)

INTRODUCTION

Under contract to DOE, Johns-Manville Sales Corporation is conducting research to improve solid-liquid separation, a critical step in all coal liquefaction processes. Rotary drum pressurized precoat filtration is being studied with the objective of the program to achieve major improvements in the filtration process and in the filtration equipment used in the coal liquefaction process. The goals are to double the filtration rate, to reduce filter aid usage, clarify filtrate and to demonstrate the results on a pilot-plant scale.

PROGRAM DESCRIPTION

The filtration research program is divided into two phases. The objectives of Phase I are to (1) develop basic knowledge of the coal oil filtration process and its dependence on liquefaction process parameters and (2) apply the knowledge gained to improve the performance of filtration operations in existing liquefaction plants. Phase II involves the design and construction of a rotary drum pressurized precoat filter capable of processing, at a rate not less than 300 pounds per square foot per hour, the product flow from an existing liquefaction pilot plant. Phase I is being funded entirely by DOE and Phase II is being supported by the Electric Power Research

Institute (EPRI) in cooperation with the DOE effort. One of the criteria established for a viable system is the capability of filtering in excess of 20 gallons of solid-liquid mixture per square foot per hour. Data obtained from operation of the pilot scale filtration unit will be used to design filters for commercial plants.

Phase I of the project is being implemented through laboratory bench-scale research. The initial step involves the design and construction of a laboratory test facility that will simulate the operation of a rotary drum pressurized precoat filter capable of operating at 750°F and 27 atmospheres. A series of tests will then be conducted to study pressure precoat filtration operating parameters at the temperatures and pressures expected in normal pilot plant operations. In these tests, two liquefaction filter feed process slurries are to be used: solvent-refined coal (SRC) from both the Wilsonville and Tacoma pilot plants and Synthoil boiler fuel supplied by the Pittsburgh Energy Research Center.

Upon completion of these tests, experiments will be performed on hot, fresh samples of Tacoma Filter Feed in order to establish the relationship between test results obtained on fresh feed and those on product that have been cooled. Due to the chemical and physical changes which may take place during aging and irreversible alterations which may occur due to various thermal histories, it is vital that product behavior during pilot testing be accurately correlated to the anticipated behavior during commercial operations.

In cooperations with P&M, Johns-Manville will apply its filtration knowledge to improve the filtration rate and reduce the filter aid usage on existing Goslin-Birmingham filters. Similar filter process optimization studies will then be conducted at other DOE-sponsored coal liquefaction plants. Johns-Manville is also responsible for assisting in the selection of a suitable precoat filter for the Cresap Test Facility in Cresap, West Virginia. This investigation will include (1) the feasibility of modifying the existing filtration equipment to accommodate the experimental work and (2) assistance in the start-up and establishment of operating conditions for the filter system selected.

HISTORY OF THE PROJECT

The first phase of this program was initiated in May 1975. During that year, emphasis was placed on designing and constructing a laboratory test facility. The experimental program for the filtration studies was also planned. The statistical method of fractional factorial design was selected as the experimental design with the principal independent variables of temperature, differential pressure, filtration time per cut, depth of cut, degree of submergence, immersion time and consumption rate of filter aid selected for investigation. The design involves treatment of each of the five variables at two levels in eight experiments with coal product and type of precoat kept constant.

The first filtration tests were run late in 1975 using water and organic media to establish precoating and cake cutting procedures. Work on the design and construction of the laboratory test facility continued during the first quarter of 1976. Technique and equipment refinements were developed from the experimental work. During 1976, filtration tests were conducted using samples of Tacoma Filter Feed (TFF), Synthoil product, Wilsonville Filter Feedstock, and hot, fresh TFF.

PROGRESS DURING OCTOBER-DECEMBER 1977

The new test leaf system to allow "in situ" operation for future runs was completed, installed, and partially evalu-

ated during October. No filtration runs were made as the system was moved and revised for the purpose of improving safety conditions. Operations were resumed in December, but start-up delays prevented obtaining filtration rate data.

Additional equipment features were installed in the system, including a retractable sleeve design and cake washing arrangement. Overall objectives in the immediate future are to develop operating techniques and obtain "in situ" data for application to the Pilot Model Rotary Pressure Precoat Filter being installed at Fort Lewis.

In future operations, the required pre-precoat will be applied with the use of suction and with the leaf outside the vessel as for previous data. While this procedure is not "in situ", effects on the overall run should be negligible. At the beginning of each run, the retractable sleeve around the leaf will be extended to a maximum distance beyond the leaf. This will allow at least 3/4-inch of pre-precoat which will be CELITE AF13 diatomite applied to the leaf from a two percent slurry in xylene/toluene.

A technique for applying the precoat "in situ" will be developed. Application will be from a slurry composed of about seven percent CELITE AF5 diatomite in liquefied coal process solvent contained in the closed filter vessel. Temperature and pressure will be controlled at about 275°F and 35 psid. The leaf will be dipped into the slurry and dried to obtain the desired overall precoat thickness of at least 1-inch. Filtrate will be collected in any or all of the seven filtrate receivers as required. After application, excess thickness may be trimmed off while the vessel is still closed, using a knife setting previously determined with the vessel open. The vessel will then be cooled and suction applied to relieve the pressure and maintain the precoat. The vessel will then be opened, condition of the precoat observed, and unused slurry removed. As soon as possible, the liquefied coal filter feed will be poured into the filter vessel. The vessel will then be closed and controlled at 600°F and 70-100 psid for "in situ" filtering and comparison with previous such runs. The leaf sleeve will be kept retracted slightly from the precoat surface as cutting progresses. A technique for washing the cake will be developed and corresponding data obtained.

XI. CHEMICALS FROM COAL

THE DOW CHEMICAL COMPANY
MIDLAND, MICHIGAN

Project Site: Midland, Michigan
Contract No.: E(49-18)-1534
Contract Funding: \$342,896
(100% DOE)

INTRODUCTION

Historically, coal was used as a chemical feedstock, but petroleum and natural gas were substituted as cheaper feedstocks when they became available. However, because of the recent rising costs and decreasing availability of petroleum and natural gas, The Dow Chemical Company, under sponsorship of DOE, is studying the possibility of using coal-derived liquids as chemical feedstock. The current contract was initiated in 1974. Dow is testing liquids from four coal liquefaction processes (COED, Synthoil, H-Coal, and SRC) in standard petrochemical processing experiments and is evaluating these results to select one process for further study. The selection will be based on process design, quality of chemicals produced, and overall plant economics. For the process selected, further development will include material balances and utility requirements for each major process step, a preliminary process flowsheet, and estimates of capital and operating costs for a commercial plant.

PROGRAM DESCRIPTION

The liquids produced by the four processes are tested in standard petrochemical processing operations to determine the advantages and disadvantages of using coal-derived liquids for petrochemical feedstocks. The experi-

mental work is being conducted in small units capable of operating unattended at pressures up to 204 atm., temperatures up to 1300° F, and feed rates from 25 to 400 milliliters per hour.

The processing sequence used for all of the samples is:

- (1) Distillation into nominally three cuts- straight-run naphtha (350° F or less), middle distillate (350° F to 650° F), and heavy gas oil (650° F to 950° F).
- (2) Hydrocracking of the middle-distillate and heavy gas oil to reduce molecular weight.
- (3) Hydrotreating of the straight-run naphtha and hydrocracked naphtha to remove heteroatoms.
- (4) Reforming of the hydrotreated naphthas to maximize aromatics.

Steam coil cracking of some of the fractions is also performed to evaluate potential ethylene yields.

Characterization studies on all of the samples in the hydroprocessing experiments are performed using the following procedures:

- Component analysis of the hydrocarbons.
- Atmospheric, vacuum, and American Society for Testing Materials (ASTM) distillations.
- Elemental analysis (carbon, hydrogen, oxygen, nitrogen, sulfur).
- Trace metal analysis.
- Liquid chromatography for class separations (aliphatics, monoaromatics, diaromatics).

HISTORY OF THE PROJECT

The laboratory investigation of the COED Syncrude and H-Coal oil began in early 1974. During that year, characterization studies and straight run distillations were completed on the COED material. Two stage hydrocracking of the mid-distillate, and steam coil cracking of naphtha, mid-distillate, and first stage hydrocrackate products followed in early 1975. Hydrocracking of the straight run heavy gas oil was completed in September 1975 and two stage hydrotreating of the straight run naphtha followed.

The H-Coal processing followed the pattern of the COED, but required an additional step. The H-Coal material was received as atmospheric still bottoms and overheads, and vacuum still bottoms slurry. While the former was processed in a manner similar to the COED Syncrude, a disposable still was constructed to separate the heavy gas oil from the solidified slurry for further processing. The resulting vacuum oil was hydrocracked in October 1975. The two stage hydrotreated H-Coal naphtha was reformed in December 1975.

Samples of Synthoil and SRC materials were also obtained during 1975. Based on characterizations of the samples, it was decided to limit the study of the Synthoil to the mid-distillate. The Synthoil straight run distillation was completed in December 1975.

Hydrocrackate SRC product was received from the U.S. Bureau of Mines. Simulated distillation data indicated a low conversion level for the mid-distillate oil. Therefore, it was decided that only the light oil produced in the process would be studied.

In the steam coil cracking experiments performed on COED and H-Coal oil naphthas and mid-distillates, the olefins yield was relatively low, but the aromatic yield made the naphtha feedstocks attractive. The results showed that:

- H-Coal straight-run naphtha was a slightly better ethylene feedstock than COED straight run naphtha.
- Cracking H-Coal mid-distillate produced twice as much fuel, 38 percent fewer olefins, and 22 percent less benzene than cracking H-Coal naphtha.
- Cracking H-Coal mid-distillate produced approximately the same amount of olefins and fuel, but nearly twice the benzene than cracking COED mid-distillate.

In order for the COED straight-run mid-distillate to be used as feedstock for petrochemical production, hydroprocessing would probably be necessary. A combination of hydroprocessing and steam coil cracking might convert the heavier COED oils to desirable petrochemicals more economically.

The petrochemical processing of COED syncrude was completed in the beginning of 1976. Reforming of H-Coal hydrocrackate naphthas was also completed after changing the presulfiding procedure for the hydrotreating catalyst. First and second pass hydrocracking of Synthoil and SRC II (slurry recycle) mid-distillates was completed as was the hydrotreating of the product naphthas.

PROGRESS DURING OCTOBER-DECEMBER 1977

The final report based on the HRI H-Coal Product was published in October. The report contained the final results of an investigation to determine the suitability of the COED, H-Coal, Synthoil, and SRC II syncrudes as petrochemicals feedstocks. Based on the results of laboratory investigations, conversions of the liquid products to petrochemical feedstocks per ton of coal fed to each liquefaction process was 15.0, 33.9, 26.9, and 23.0 percent respectively. While the samples obtained from liquefaction runs were not necessarily representative, based on the samples that were furnished, the H-Coal process produced the maximum yield of feedstocks; and, thus, was chosen as the basis for the preliminary design and economics for a commercial chemical refinery.

From a chemical refinery sized to process 50,000 barrels per stream day of H-Coal derived syncrude, yields of 653M pounds a year of ethylene and 245M gallons per year of benzene were calculated. Total annual revenues were estimated at \$447.4M (1980). Total capital investment of \$406.3M and production costs of \$371.5M per year were also calculated. For the base case, H-Coal syncrude was taken at \$16 per barrel, a price competitive with world crude oil (1980). At that feedstock price, a return on total capital (ROI) of greater than 18 percent is realized. The results of this investigation demonstrated the technical feasibility and economic viability of processing coal derived syncrudes for future production of petrochemical feedstocks.

XII. R&D ENGINEERING SERVICES — HYDROCARBONIZATION

OAK RIDGE NATIONAL LABORATORY
OAK RIDGE, TENNESSEE

Contract No.: E(49-18)-1740

Contract Funding: \$2,135,000
(100% DOE)

INTRODUCTION

The Oak Ridge National Laboratory (ORNL) is conducting several research and development projects in support of the increased utilization of coal as a source of clean energy. Under this agreement, begun in 1974, ORNL estimated the optimum operating conditions of a fluidized-bed hydrocarbonization reactor and designed, constructed, and operated a fluidized-bed reactor capable of continuously processing ten pounds of coal per hour. Operating conditions for this reactor are at least 20 atm and up to 1,200°F.

- Review and evaluation, involving the analysis of prior work to estimate operating conditions and guide reactor design.
- Experimental development, which includes the investigation of hydrocarbonizer components at atmospheric pressure and temperatures up to 1,200°F.
- Bench-scale studies involving the design, construction, and operation of continuous hydrocarbonization system capable of processing ten pounds of coal per hour at temperatures up to 1,200°F and pressures as great as 20 atm.

PROCESS DESCRIPTION

Hydrocarbonization is a type of coal liquefaction process which combines high-temperature, high-pressure, and fluidized-bed operation using hydrogen-rich gas for fluidization. The hydrocarbonization process can produce controlled yields of desulfurized char, liquid fuels, and substitute natural gas. The product char may be suitable for use in boilers, without stack gas treatment units. The liquid products may be composed of naphtha, light gas oil, and heavy gas oil. A flow diagram of the bench-scale hydrocarbonization unit is shown in Figure XII-1.

The work under this contract is divided into three tasks:

HISTORY OF THE PROJECT

An intensive literature survey conducted during 1975 led to the preliminary conclusions that very little reaction takes place below 750°F and the yield of both oil and gas increases directly with temperature. A maximum oil yield was noted at 1,020°F to 1,110°F. Gas production increased proportionally with temperature and therefore did not show an optimum over the temperature range studied.

Two Lucite mock-ups were constructed to study the mechanics of hydrocarbonization processes. An atmospheric pressure high-temperature ten-pound-per-hour hydrocarbonization bench-scale unit was designed based on tests on the Lucite mock-ups. The system was fabricated and installed during 1975 and shakedown tests were initiated late in the year. Modifications of the bench-scale

Figure XII-1. FLOW DIAGRAM OF BENCH-SCALE HYDROCARBONIZATION SYSTEM

system to permit recirculating fluidized-bed operation was completed in early August 1976.

PROGRESS DURING OCTOBER-DECEMBER 1977

Analytical data for Run HC-23, conducted last quarter, were received. For Run HC-23, Illinois No. 6 coal that had been treated with an aqueous mixture of lime and sodium hydroxide was used. The treatment removed approximately two-thirds of the sulfur from the coal but also resulted in a considerable increase in the calcium, sodium, and ash content. The oil yield from run HC-23 was 14 percent, based on MAF coal. The yield was somewhat lower than the maximum yield of 21 percent that had previously been achieved with Wyodak coal. It is expected that coal treated with other materials may result in higher oil yields.

In November, hydrocarbonization run HC-24 was made using Illinois No. 6 coal that had been pretreated with aqueous sodium carbonate at 300°F in an autoclave. The run duration was 7.7 hours with no interruptions of feed or other difficulties during the entire run. The fluidized-bed temperature was uniform at 1,060°F and the temperature at the top of the draft tube was 1,035°F indicating that there was good fluidization and

char recirculation through the draft tube. The reactor pressure was 300 psig.

After the run, the system was drained and cleaned for liquid yield determination, and appeared to be in good condition. There were no indications of caking or agglomeration when the char was removed from the reactor. The smooth operation of the system and the small quantity of fines in the feed material (14 percent -140 mesh) appeared to have resulted in a low carryover of char with the oil collected in the scrubber.

Results showed that the treatment of the coal removed only a small amount of the sulfur. The treated coal contained 2.9 percent (MAF) sodium and had a higher ash and moisture content than the untreated coal.

Preparation for Run HC-25 began late in the quarter. Most of the coal feed had been prepared for the run, which was to be made using Wyodak coal at 150 psig reactor pressure. Run HC-25 will be made using approximately one half of the operating pressure used in most previous runs and will provide data on the effect of pressure on yields.

During the quarter, the length of the plastic reactor mock-up was increased by four feet to study the flow characteristics of a longer reactor. The mock-up will be used to determine conditions that will give satisfactory fluidization and char circulation in the reactor.

XIII. ZINC HALIDE HYDROCRACKING PROCESS

CONOCO COAL DEVELOPMENT COMPANY
LIBRARY, PENNSYLVANIA

Project Site: Library, Pennsylvania
Contract No.: EX-76-C-01-1743
Contract Funding: \$11,410,400
DOE: \$10,269,360
Industry: \$ 1,141,040

INTRODUCTION

Continental Oil Company, with the participation of Shell Development Company, a division of Shell Oil Company, is under contract to DOE to develop the zinc halide hydrocracking process. The overall objective of the project is the production of clean liquid and gaseous fuels, of which approximately fifty percent will be distillate, equivalent to four barrels per ton of coal on a moisture and ash-free (MAF) basis.

Specifically, experimental studies will be performed involving both continuous bench-scale and process development unit work dealing with the investigation of zinc halide as a catalyst for the hydrogenation and hydrocracking of coal extract and coal. Studies will also involve the development of an economical regeneration process for the efficient recovery of zinc halide from the spent melt. The schedule for the program is shown in Figure XIII-1.

PROCESS DESCRIPTION

The zinc halide hydrocracking process is designed to maximize the conversion of coal into light distillates by severe catalytic cracking. A schematic of the process is provided in Figure XIII-2. The coal is dried and pulver-

ized and then introduced into a feed tank where it is slurried with a process-derived recycle oil. The slurry feed is transferred to a hydrocracking reactor where it is mixed with hydrogen and the zinc chloride catalyst. In the reactor, the coal is cracked to distillates in the gasoline range, producing on distillation a clear 90 RON gasoline without further processing, and a small quantity of very low sulfur, low-nitrogen fuel oil. The gas is subsequently separated from the liquid in a product receiver. Spent catalyst is then fed to a regenerator for catalyst recovery.

PROGRESS DURING OCTOBER-DECEMBER 1977

During the quarter, the hydrocracking section of the one-ton-per-day Process Development Unit (PDU) was completed. Pressure testing, electrical heater installation and insulation were also underway.

Four material balance runs were completed in the continuous bench-scale hydrocracker during October. These were two stage runs at two severities, involving both stages at the same temperature, and then at different temperatures.

The final run on the bench-scale hydrocracker was completed in November. Conversion data as functions of

LEGEND: BEGIN MILESTONE
COMPLETE MILESTONE

Figure XIII-1. ZINC HALIDE HYDROPROCESSING PROJECT SCHEDULE

Figure XIII-2. ZINC HALIDE HYDROCRACKING PROCESS FLOWSHEET

the operating variables (temperature, pressure, time) were correlated in a reaction model for Colstrip coal similar to that used earlier for bituminous coal extract.

A continuous bench-scale run using recycled 200 x 475°F distillate as the coal vehicle showed a higher conversion of the distillate (34 percent) than had been expected from batch tests. Subsequent batch tests appeared to confirm that higher conversion of recycle solvent to gasoline was obtained with recycled "used" zinc chloride as compared to fresh zinc chloride.

An economic study of the value of treating the coal ash effluent from the regeneration cyclone in a secondary

stage to recover zinc was completed. The treatments considered ranged from addition of a heated air stream to strip out zinc chloride vapors, to treatment with HCL plus air to also convert zinc oxide, silicates, aluminates, etc., to zinc chloride. The study showed an incentive to add secondary zinc recovery in all cases, with savings of 3.0 to 3.5 cents per gallon of gasoline.

During the quarter, test of PDU coal grinding equipment showed that subbituminous coal suffered loss in reactivity when dried and ground in the presence of six percent oxygen. Tests to reduce the oxygen level in the equipment were scheduled.

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XIV. FLUID COKING OF COAL LIQUEFACTION RESIDUES

EXXON RESEARCH AND ENGINEERING
COMPANY

Contract No.: EX-76-C-01-2422

Contract Funding: \$1,312,470

INTRODUCTION

Exxon Research and Engineering Company, under the sponsorship of DOE, is studying the potential of fluid coking to recover distillate liquids. The primary objective of this contract is to evaluate the applicability of current commercial fluid coking technology to processing of three residues from DOE sponsored coal liquefaction processes. One selected residue will be further processed in a 0.5 ton per day process development unit. The program schedule for this project is shown in Figure XIV-1.

PROGRAM DESCRIPTION

The treatment of residual, solids-containing liquids produced by various coal liquefaction processes is important in determining the overall economic viability of the process. Solids are removed from primary coal liquids by methods such as hydrocyclones, vacuum distillation, and solvent deashing. The solids-containing residues from these operations may contain additional valuable liquid products which may be recovered by fluid coking.

Three samples of coal liquid residue produced in DOE-sponsored coal liquefaction projects will be analyzed and processed using bench-scale equipment. A Continuous Stirred Coking Unit (CSCU) will be used to obtain quantitative yield data for fluid coking of the coal

Figure XIV-1. FLUID COKING PROGRAM SCHEDULE

liquefaction residues. This type of pilot unit was utilized successfully in scale-up of yield and quality data for petroleum. CSCU simulates the steam fluidization of a commercial fluid coker by means of a mechanical agitator. A schematic is shown in Figure XIV-2. One of the selected coal liquid residues will be processed in a nominal two-barrel-per-day fluidized-bed pilot unit.

HISTORY OF THE PROJECT

Work under this contract began in the last quarter of 1976. H-Coal solvent precipitation underflow was the first of three candidate coal liquefaction residues to be evaluated, with analytical tests completed by the end of 1976. Results were discussed in the Interim Technical Progress Report for First Residue published in June 1977 and available from NTIS.

Figure XIV-2. CONTINUOUS STIRRED COKING UNIT PROCESS SCHEMATIC

PROGRESS DURING OCTOBER-DECEMBER 1977

The Continuous Stirred Coking Unit (CSCU) was used to complete the simulated coker liquid recycle run using 1000°F coker liquid product from the Stripped SRC Antisolvent Deashing Underflow CSCU runs. Analyses of products and interpretation of data from

each of the CSCU runs continued throughout the quarter. A bogging test was scheduled on the sample for December, but equipment problems forced postponement until next quarter.

The fourth coal liquefaction residue sample, an H-Coal vacuum bottoms, was received for analytical characterization in two 55-gallon barrels. A representative sample of the totally solid uniform mass in each barrel was taken for analysis.

XV. UPGRADING OF COAL LIQUIDS

UOP, INC.

DES PLAINES, ILLINOIS

Contract No.: EF-77-C-01-2566

Contract Funding. \$945,584

INTRODUCTION

UOP, Inc. is under contract to DOE to determine the applicability of commercial UOP conversion processes for all distillate coal liquids. Coal liquefaction processes may be separated into two groups: those that produce resid containing coal liquids, and those that produce a distillate coal liquid. UOP completed studies on upgrading resid containing coal liquids in 1976.

Distillate coal liquids being studied under the present contract include H-Coal Syncrude Mode and Exxon Donor Solvent.

The objectives of this contract are to:

- Process selected DOE sponsored coal liquid distillates through a comprehensive hydrocracking scheme.
- Determine the response of the fluid catalytic cracking unit to hydrotreated coal liquid distillates.
- Maximize the production of either motor gasoline or No. 2 fuel oil by the hydrocracking scheme as stated in the first objective.
- Provide yield-octane curves by catalytic reforming naphthas produced in primary coal liquefaction programs and/or by the hydrocracking scheme as stated in the first objective.

- Correlate feedstock properties and process conditions data with product yields and qualities.

PROJECT DESCRIPTION

Four tasks are identified under this program. The first task involves two stage continuous catalyst bed hydrocracking of two coal liquid distillates. The second task entails processing of two coal liquid distillates through continuous fixed catalyst bed hydrotreating and fluid catalytic cracking units. The third task covers processing of two naphthas through continuous refining-reforming bench scale units. The fourth task involves the correlation of feedstock properties and process conditions with product yields and qualities which can be composed into a simple process model.

HISTORY OF THE PROJECT

The present contract is an extension of the program begun by UOP under contract E(49-18)-2010. Evaluations of the primary products of coal liquefaction generated by the Synthoil, Solvent Refined Coal (SRC), and

H-Coal processes were completed in 1976, and the final report of the project was written. The present program began in January of 1977 under contract EF-77-C-01-2566.

PROGRESS DURING OCTOBER-DECEMBER 1977

Second-stage hydrocracking of H-Coal gas oil to fuel oil was concluded in October with experiments using UOP-HCA catalyst at base pressure and 500 psig above base pressure. Complete conversion of 600°F+ hydrocrackate to fuel oil was successfully attained with highly stable operation.

A series flow second-stage hydrocracking of high nitrogen H-Coal liquid to gasoline was also conducted during the quarter. Complete conversion to gasoline was attained at moderate process conditions.

Three drums of raw Exxon Donor Solvent (EDS) process liquid were received in October. Because of the presence of significant amounts of heptane insolubles, the raw EDS process gas oil was not suitable as hydrocracker charge stock. Hydrotreating converted these

insolubles completely to distillate and lowered the nitrogen content well below the level required for a second-stage hydrocracking feedstock.

Hydrotreating process variable studies were also started on the raw EDS liquid product. The primary objective of the studies was to investigate the effects of process variables, and to arrive at optimum conditions for preparing suitable feedstocks for subsequent hydrocracking. The first series of experiments was conducted at 250 psig above base pressure, 25°C above base temperature, and three space velocities. Tests were also run at 11°C above base temperature and 5°C below base temperature, and three space velocities. In all cases heptane insolubles were completely converted and substantial amounts of nitrogen were removed. Nitrogen conversions as high as 99.9 weight percent were obtained.

Mass spectroscopy analyses of the EDS naphtha before and after upgrading were completed. The upgraded naphtha was reformed over a commercial Platforming catalyst over a series of conditions. Mass spectroscopy analyses, as well as other required analyses of four selected reformates were also completed.

Two preparative H-Coal gas oil hydrotreating runs aimed at producing a FCC feedstock with high hydrogen content (11 and 13 percent hydrogen) were completed.

XVI. GASIFICATION OF RESIDUAL COAL LIQUEFACTION PRODUCTS

TEXACO, INC.

Contract No.: E(49-18)-2247

Contract Funding: \$939,840
(100% DOE)

INTRODUCTION

Under this agreement, Texaco, Inc. will perform the research concerning the gasification of residual material from coal liquefaction samples supplied by DOE. The objective of the contract is to evaluate materials from several coal liquefaction pilot plants to determine their suitability as feedstocks for a coal gasification process. After initial laboratory testing and evaluation, promising materials selected by DOE will be tested in large scale pilot plant gasification runs.

PROCESS DESCRIPTION

The thermal efficiency of a coal liquefaction plant using a process that requires hydrogen or synthesis gas is dependent upon the efficient recovery of that gas from the non-liquefied fraction of the coal. This material, together with inorganic ash and some fraction of the converted coal, can be recovered after liquefaction as a residue from a vacuum distillation tower. The chemical and physical properties of the high-ash coal-derived residues determine both the method of introducing these materials into the gasifier and the efficiency of the gasifier. As Texaco has demonstrated that high-ash coal-derived residues can be gasified directly using the Texaco coal gasification process, the necessary design parameters will be determined for the materials resulting from DOE-sponsored liquefaction projects.

HISTORY OF THE PROJECT

The contract was awarded to Texaco in June of 1976. Work began with receipt of two H-Coal residue samples during the last quarter of the year. Results of the tests performed on the feedstocks indicated that the "Syn-crude Vacuum Bottoms" was a suitable feedstock for the Texaco coal gasification process, while the "Settler Stripped Underflow" seemed a poor candidate because of its physical properties.

PROGRESS DURING OCTOBER-DECEMBER 1977

A 12-pound sample of SRC ash concentrate was received during the quarter from Kerr-McGee Corporation. A type I evaluation to determine the chemical composition and physical properties was completed with this material and a report was written.

The ash concentrate cannot be melted and, therefore, must be charged to the gasifier as a slurry. A 55 weight percent slurry of ash concentrate in water was judged to be pumpable.

Because of the high ash content (about 41 weight percent) of the material, the gasification efficiency will be low on a total solids feed rate basis. A type II preliminary pilot plant test was recommended.

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XVII. PRODUCTION OF CLEAN INDUSTRIAL AND TRANSPORTATION FUELS FROM COAL

THE LUMMUS COMPANY
BLOOMFIELD, NEW JERSEY

Plant Site: Bloomfield, New Jersey
Contract No.: E(49-18)-2514
Contract Funding: \$4,700,000

INTRODUCTION

The Lummus Clean Fuel From Coal (CFFC) Process is being studied under this contract for the purpose of establishing optimum operating conditions and proving the reliability of the process. The primary objectives of this development program are to:

- Establish recycle oil self-sufficiency, catalyst life, and process operability for coal liquefaction; and demonstrate comparative hydroliquefaction performance of a proprietary Lummus catalyst versus a standard cobalt-molybdate catalyst.
- Convert the all-distillate coal liquid product to gasoline by continuous fixed-bed hydrotreating/hydrocracking operations.
- Provide a preliminary engineering design of a Lummus CFFC pilot plant facility.

The effort is divided into three tasks. Task I is Coal Liquefaction and involves the modification and operation of an existing 30-pound-per-hour expanded bed test unit. The modifications will permit continuous integrated operation of the process, including the hydroliquefaction reactors, solvent de-ashing module, the distillation unit, and the vacuum flashing units. Illinois No. 6 coal, containing at least three percent sulfur and one percent nitrogen, will be used in the bench-scale tests to evaluate the process. Operations will be conducted to produce an all-distillate product.

Task II, Hydrotreating/Hydrocracking, will involve the operation of a multistage fixed-bed hydrotreating/hydrocracking unit to determine the temperature, pressure, space velocity, and recycle rates required to convert the coal liquid to gasoline. A proprietary Lummus denitrogenation catalyst will be used in the first stage, and tungsten nickel sulfide will be used in the second stage.

In Task III, Engineering and Estimating, the results of studies conducted in the previous tasks will be used to formulate a preliminary engineering design of a pilot plant facility. The overall program plan is shown in Figure XVII-1.

PROCESS DESCRIPTION

The Lummus Clean Fuels From Coal (CFFC) Process is based on direct catalytic hydroliquefaction in a multi-stage, plug-flow ebullated-bed reactor. A schematic of the process is presented in Figure XVII-2. The process employs an integrated solvent de-ashing technique requiring no mechanical separation operations. The ebullated bed is produced by the upward flow of coal feed, slurry oil, and hydrogen-rich gas entering at the bottom of the reactor. The Lummus process takes advantage of both the high reaction rates of a plug-flow reactor and the non-fouling features of the ebullated bed reactor.

Figure XVII-1. CLEAN FUELS FROM COAL PROGRAM DEVELOPMENT SCHEDULE

Figure XVII-2. CLEAN FUELS FROM COAL PROCESS SCHEMATIC

The use of multiple staging permits superior control of reactor conditions resulting in minimal hydrogen consumption. Since most of the process heat release is due to hydrogen consumption, the reduced hydrogen uptake in the process also minimizes heat dissipation problems. While net hydrogen consumption is kept at a minimum, the Lummus process also exhibits a high degree of efficiency in hydrogen utilization.

The Lummus process uses a proprietary solvent de-ashing system which, unlike filters and centrifuges, does not contain costly and hard-to-develop mechanical equipment, thus simplifying scaleup. In addition, solvent de-ashing does not require increased process severity to produce a lighter product capable of recovery from a vacuum distillation.

HISTORY OF THE PROJECT

The original Lummus bench-scale coal liquefaction pilot plant became operational in 1972 and a one-year process development program was conducted using Illinois No. 6 coal. A technically feasible process capable of producing a liquid product with a sulfur content of 0.3 percent and an ash content of 0.1 percent was demonstrated. The Lummus de-ashing system was found to be operational. Since the initial work, two subbituminous coals have been processed successfully in the bench-scale pilot plant.

The program was initially submitted to ERDA, now DOE, in 1975 as an unsolicited proposal. Following contract negotiations, a revised experimental program was authorized and a contract granted by ERDA on September 28, 1976. Modifications were started to convert the expanded-bed liquefaction test unit and its ancillary anti-solvent de-ashing module, a distillation unit, and two vacuum flashing units to permit integrated continuous operation.

PROGRESS DURING OCTOBER-DECEMBER 1977

Operations during the quarter were directed toward the preparation of equilibrium recycle solvent. The severity of the hydroliquefaction operation was gradually increased to maximize distillate yield.

The unit continued to operate on 30 percent coal paste using Nalco nickel/molybdenum spherical catalyst. At the end of the quarter, the paste operations were in the third cycle and were conducted at 830° F, an operating pressure of 2,100 psig, and a coal space rate of 40 pounds of coal per hour per cubic foot of settled catalyst volume. The operations of all the units were satisfactory.

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GLOSSARY

absorption—an imprecise term suggesting the taking up of one substance by another by either a physical process or a chemical combination.

acceptor—calcined carbonate that absorbs carbon dioxide evolved during gasification, liberating heat.

acid gas removal—the process of selectively removing hydrogen sulfide and carbon dioxide from a gas stream.

activated carbon—carbon obtained by carbonization in the absence of air, preferably in a vacuum; has the property of absorbing large quantities of gases, solvent vapors; used also for clarifying liquids.

adiabatic—any process where heat is neither given off nor absorbed.

adsorption—the process by which the surface of a solid or liquid attracts and holds any atom, molecule, or ion from a solution or gas with which it is in contact.

agglomerate—assemblage of ash particles rigidly joined together, as by partial fusion (sintering).

anthracite coal—hard coal containing 86 to 98 percent fixed carbon and small percentages of volatile material and ash.

API—American Petroleum Institute.

API gravity—a scale adopted by the API for measuring the density of oils; $^{\circ}\text{API} = \frac{141.5}{\text{Specific gravity, } 60^{\circ}\text{F}/60^{\circ}\text{F}} - 131.5$

aromatic hydrocarbon—a cyclic hydrocarbon containing one or more six-carbon (benzene) rings.

ash—solid residue remaining after the combustion of coal.

ASTM—American Society for Testing Materials.

autoclave—a vessel, constructed of thick-walled steel, for carrying out chemical reactions under high pressures and temperatures.

bench-scale unit—a small-scale laboratory unit for testing process concepts and operating parameters as a first step in the evaluation of a process.

binder—carbon products, tars, etc., used to impart cohesion to the body to be formed; a coal-extract binder may be used to prepare formed-coke pellets from non-coking coals.

bituminous coal—a broad class of coals containing 46 to 86 percent fixed carbon and 20 to 40 percent volatile matter.

blow down—periodic or continuous removal of water from a boiler to prevent accumulation of solids.

bottoming cycle—the lower temperature thermodynamic power cycle of a combined-cycle system.

Btu—British thermal unit, the quantity of energy required to raise the temperature of one pound of water one degree Fahrenheit.

BTX—benzene, toluene, xylene; aromatic hydrocarbons.

caking—the softening and agglomeration of coal as a result of the application of heat.

calcination—the process of heating a solid to a high temperature to cause the decomposition of hydrates and carbonates.

calorific value—the quantity of heat obtained by the complete combustion of a unit mass of a fuel under prescribed conditions.

carbon fiber—fine filaments of carbon about eight microns in diameter which are used in composite materials, being bound with resins.

carbonization—destructive heating of carbonaceous substances with the production of a solid, porous residue, or coke, and the evolution of a number of volatile products. For coal, there are two principal classes of carbonization, high-temperature coking (about 900°C) and low-temperature carbonization (about 700°C).

catalyst—a substance that accelerates the rate of a chemical reaction without itself undergoing a permanent chemical change.

centrifuge—an apparatus rotating at high speed which utilizes the centrifugal force generated to separate materials of different densities, e.g., undissolved residue from coal solution in the SRC process.

char—the solid residue remaining after the removal of moisture and volatile matter from coal.

Claus process—industrial method of obtaining elemental sulfur through the partial oxidation of gaseous hydrogen sulfide in air followed by catalytic conversion to molten sulfur.

coal—a readily combustible rock containing more than 50 weight percent and more than 70 volume percent of carbonaceous material including inherent moisture, formed from compaction and induration of variously altered plant remains similar to those in peat.

coalification—metamorphosis of vegetable debris into coal.

coke—strong porous residue consisting of carbon and mineral ash formed when bituminous coal is heated in a limited air supply or in the absence of air. Coke may also be formed by thermal decomposition of petroleum residues.

coke breeze—the fine screenings from crushed coke usually passing a 1/2 inch or 3/4 inch screen opening.

combined cycle—two sequential thermodynamic power conversion systems operating at different temperatures.

combustion gas—gas formed by the combustion of coal, e.g., burning.

combustor—a vessel in which combustion takes place.

coupon—a polished metal strip used to measure the rate of corrosion of the metal in a specific gaseous or liquid environment.

cracking—the partial decomposition of high-molecular-weight organic compounds into lower-molecular-weight compounds, generally as a result of high temperatures.

crude gas—impure gas produced in a gasifier.

culm—the waste or slack from anthracite mines or preparation plants consisting of fine coal, coal dust, and dirt.

cyclone separator—essentially a settling chamber to separate solid particles from a gas, in which gravitational acceleration is replaced by centrifugal acceleration.

degasification—a process for removing naturally occurring methane from coal seams.

delayed coking—a process wherein coal is subjected to a long period of carbonization at moderate temperatures to form coke.

demineralization—removal of mineral matter (ash) from coal by solvent extraction, usually under hydrogen atmosphere.

depolymerization—the change of a large molecule into simpler molecules usually accompanied by the substitution of hydrogen for oxygen in the molecular structure.

destructive distillation—the distillation of coal accompanied by its thermal decomposition.

desulfurization—the removal of sulfur from hydrocarbonaceous substances by chemical reactions.

devolatilization—the removal of a portion of the volatile matter from medium- and high-volatile coals.

diatomaceous earth—a yellow, white, or light-gray, siliceous porous deposit made up of opaline shells of diatoms; used as a filter aid, paint filler, adsorbent, abrasive, and thermal insulator. Also known as kieselguhr.

diatomite—See Diatomaceous Earth.

disolution—the taking up of a substance by a liquid with the formation of a homogeneous solution.

distillation—a process of vaporizing a liquid and condensing the vapor by cooling; used for separating liquids into various fractions according to their boiling points or boiling ranges.

dolomite—a carbonate of calcium and magnesium having the chemical formula $\text{CaMg}(\text{CO}_3)_2$.

Dowtherm—trademark for a series of eutectic mixtures of diphenyl oxide and diphenyl used as high-temperature heat-transfer fluids.

ebullated bed—gas containing a relatively small proportion of suspended solids, bubbles through a higher density fluidized phase with the result that the system takes on the appearance of a boiling liquid.

economizer—heat exchanging mechanism for recovering heat from flue gases.

effluent gas—gas given off from a process vessel.

elutriation—the preferential removal of the small constituents of a mixture of solid particles by a stream of high-velocity gas.

endothermic reaction—a process in which heat is absorbed.

enthalpy change—the increase or decrease in heat content of a substance or system which accompanies its change from one state to another under constant pressure.

entrained bed (flow)—a bed in which solid particles are suspended in a moving fluid and are continuously carried over in the effluent stream.

eutectic—that combination of two or more components which produces the lowest melting temperature.

exothermic reaction—a process in which heat is liberated.

extraction—a method of separation in which a solid or solution is contacted with a liquid solvent (the two being essentially mutually insoluble) to transfer components into the solvent.

extractive coking—similar to delayed coking process, with the emphasis on high tar yields to produce liquids.

filter aid—finely divided solids used to increase efficiency of filtering.

filter cake—the moist residue remaining from the filtration of a slurry to produce a clean filtrate.

filtrate—a liquid free of solid matter after having passed through a filter.

filtration—the separation of solids from liquids by passing the mixture through a suitable medium, e.g., cloth, paper, diatomaceous earth.

Fischer assay—method for determining the tar and light oil yields from coal or oil shale; conducted in a retort under an inert atmosphere with a prescribed increase in temperature to 500°C.

Fischer-Tropsch catalyst—catalysts developed for the catalytic synthesis of liquid fuels from coal-derived synthesis gas; catalysts contain principally iron, cobalt, nickel, or ruthenium.

Fischer-Tropsch process—method of hydrogenating mixtures of

carbon monoxide and hydrogen produced from coal, lignite, or natural gas by means of steam, at 1-10 atmospheres and 360-410°F to yield liquid and gaseous fuels, and a wide spectrum of industrial chemicals.

fixed-bed—stationary solid particles in intimate contact with fluid passing through them.

fixed carbon—the solid residue, other than ash, obtained by destructive distillation; determined by definite prescribed methods.

flash carbonization—a carbonization process characterized by short residence times of coal in the reactor to optimize tar yields.

flue gas—gaseous combustion products.

fluidization (dense phase)—the turbulent motion of solid particles in a fluid stream; the particles are close enough as to interact and give the appearance of a boiling liquid.

fluidization (entrained)—gas-solid contacting process in which a bed of finely divided solid particles is lifted and agitated by a rising stream of gas.

fluidized bed—assemblage of small solid particles maintained in a balanced suspension against gravity by the upward motion of a gas.

fly ash—a fine ash from the pulverized coal burned in power station boilers, or entrained ash carried over from a gasifier.

fractionation—distillation process for the separation of the various components of liquid mixtures.

freeboard—the space in a fluidized-bed reactor between the top of the bed and the top of the reactor.

free swelling index—a standard test that indicates the caking characteristics of coal when burned as a fuel.

Friedel-Crafts reaction—a substitution reaction, catalyzed by aluminum chloride in which an alkyl (R-) or acyl (RCO-) group replaces a hydrogen atom of an aromatic nucleus to produce a hydrocarbon or a ketone.

fuel cell—a galvanic cell in which the chemical energy of a conventional fuel is utilized to produce electricity.

fuel gas—low heating value (150-350 BTU/scf) product generally utilized on site for power generation or industrial use.

gasification of coal—the conversion of solid coal into a gaseous form by various chemical reactions with steam.

gasifier—a vessel in which gasification occurs, usually utilizing fluidized-bed, fixed-bed, or entrained-bed units.

heat capacity—quantity of heat required to raise the temperature of one pound of a substance one degree Fahrenheit.

high-Btu gas—a gas having a heating value of 900 to 1,000 Btu per standard cubic foot, which approaches the value for natural gas.

higher-heating value (HHV)—the heat liberated during a combustion process in which the product water vapor is condensed to a liquid and the heat of condensation is recovered.

hydroclone—a small cyclone extractor for removal of suspended solids from a flowing liquid by means of the centrifugal forces set up when the liquid is made to flow through a tight conical vortex.

hydrocoking—coking of tars, SRC, etc., under hydrogenating conditions to form liquid products.

hydrocracking—the combination of cracking and hydrogenation of organic compounds.

hydrogasification—gasification that involves the direct reaction of fuels with hydrogen to optimize formation of methane.

hydrogenation—chemical reactions involving the addition of gaseous hydrogen to a substance in the presence of a catalyst under high temperatures and pressures.

hydrogen donor solvent—solvent, such as anthracene oil, tetralin (tetrahydronaphthalene), decalin, etc., which transfers hydro-

gen to coal constituents causing depolymerization and consequent conversion to liquid products of lower boiling range which are then dissolved by the solvent.

hydrotreating—a process to catalytically stabilize petroleum or other liquid hydrocarbon products and/or remove objectionable elements from products or feedstocks by reacting them with hydrogen.

ideal gas—any gas whose equation of state is expressed by the ideal gas law, namely $PV = nRT$ where P is the pressure, V is the volume, R is the gas constant, T is the absolute temperature, and n = number of moles.

ignition temperature—the minimum temperature necessary to initiate self-sustained combustion of a substance.

industrial gas—see fuel gas.

inerts—constituents of a coal which decrease its efficiency in use, e.g., mineral matter (ash) and moisture in fuel for combustion.

in situ—in its original place, e.g., underground gasification of a coal seam.

intermediate-Btu gas—synthesis gas product with a higher heating value between 350 and 500 Btu per standard cubic foot.

lignite—brownish-black coal containing 65-72 percent carbon on a mineral-matter-free basis, with a rank between peat and subbituminous coal.

limestone—sedimentary rock containing 50 percent carbonate (CO_3) of lime or magnesia. Chemical formula (for calcite limestone) is CaCO_3 .

liquefaction—conversion of a solid to a liquid; with coal, this appears to involve the thermal fracture of carbon-carbon and carbon-oxygen bonds, forming free radicals. These radicals abstract hydrogen atoms yielding low molecular weight gaseous and condensed aromatic liquids.

liquefied petroleum gas (LPG)—those hydrocarbons that have a vapor pressure (at 70°F) slightly above atmospheric (such as propane and butane); kept in liquid form under a pressure higher than 1 atm.

lock hopper—a mechanical device that permits the introduction of a solid into an environment of different pressure.

low-Btu gas—a gas having a heating value up to 350 Btu per standard cubic foot.

lower heating value—the heat liberated by a combustion process assuming that none of the water vapor resulting from the process is condensed, so that its latent heat is not available.

MAF—moisture and ash-free; a term that relates to the organic fraction in coal.

mesh—measure of fineness of a screen, e.g., a 400-mesh sieve has 400 openings per linear inch.

methanation—the production of methane (CH_4) from carbon monoxide or dioxide and hydrogen.

methane— CH_4 , a colorless, odorless, and tasteless gas, lighter than air; the chief component of natural gas.

methanol—methyl alcohol, CH_3OH .

micron—a unit of length equal to one millionth of a meter; 10^{-6} meter.

moving bed—particled solids in a process vessel that are circulated (moved) either mechanically or by gravity flow.

natural gas—naturally occurring gas extracted from sedimentary structures consisting mainly of methane and having a higher heating value of approximately 1,050 Btu per standard cubic foot.

noncoking coal—a coal that does not form coke under normal coking conditions.

olefinic hydrocarbon—a class of unsaturated hydrocarbons containing one or more double bonds and having the general chemical formula C_nH_{2n} .

open cycle—a thermodynamic power cycle in which the working fluid passes through the system only once and is then exhausted to the atmosphere.

peat—an unconsolidated, hydrophilic, yellowish-brown to brownish-black, carbonaceous sediment, formed by accumulation of partially fragmented and decomposed plant remains in swamps and marshes which retains more than 75 percent inherent moisture and less than 12 percent mineral matter in saturated natural deposits.

petrochemicals—those derived from crude oil or natural gas, or their coal-derived substitutes; they include light hydrocarbons such as butylene, ethylene and propylene, the raw materials for the production of plastics by polymerization.

phenols—a group of aromatic compounds having the hydroxyl (OH) group directly attached to the benzene ring.

pilot plant—a chemical process plant containing all the processes of a commercial unit, but on a smaller scale, for the purpose of studying the technical and economic feasibility of the process.

pipeline gas—a methane-rich gas that conforms to certain standards and has a higher heating value between 950 and 1,050 Btu per standard cubic foot.

plenum chamber—an enclosed space through which air is forced for slow distribution through ducts.

precoat—layer of suitable filtering medium, e.g., diatomaceous earth, laid down on a rotary filter cloth prior to operation.

prilling tower—a tower that produces small solid agglomerates by spraying a liquid solution in the top and blowing air up from the bottom.

process development unit—a system used to study the effects of process variables on performance; sized between a bench-scale unit and a pilot plant.

proximate analysis—analysis of coal based on the percentages of moisture, volatile matter, fixed carbon (by difference), and ash, using prescribed methods. Reported on different bases, such as as-received (or as-fired), dry, mineral-matter-free (mmf), and dry mineral-matter-free (dmmf).

purification—removal of a wide range of impurities present in gases from coal gasification.

pyrolysis—thermal decomposition of organic compounds in the absence of oxygen.

quenching—cooling by immersion in oil, water bath, or water spray.

Raney nickel catalyst—specially prepared nickel catalyst used in the hydrogenation of organic materials and the methanation of synthesis gas to methane.

raw gas—see crude gas.

reactivity—susceptibility to chemical change; for example, in coal liquefaction, the reactivity of the coal for conversion to liquid products is a function of the coal rank, among other things.

reactor—vessel in which coal-conversion reactions take place.

Rectisol process—a process for the purification of coal-gasification gas based on the capability of cold methanol to absorb all gas impurities in a single step; gas naphtha, unsaturated hydrocarbons, sulfur compounds, hydrogen cyanide, and carbon dioxide are removed from the gas stream by the methanol at temperatures below 0°C.

reducing gas—a gas which, at high temperatures, lowers the state of oxidation of other chemicals.

reforming processes—a group of proprietary processes in which low-grade or low molecular weight hydrocarbons are catalytically converted to higher grade or higher molecular weight materials; also applies to the endothermic reforming of methane, for the production of hydrogen, by the reaction of methane and steam in the presence of nickel catalysts.

refractory—a material capable of withstanding extremely high temperatures and having a relatively low thermal conductivity.

residence time—time spent by a typical particle in a particular zone.

saturated hydrocarbon—a carbon-hydrogen compound with all carbon bonds filled; that is, there are no double or triple bonds as in olefins and acetylenes.

scrubber—apparatus in which a gas stream is freed of tar, ammonia, and hydrogen sulfide.

seam coal—coal which is intermediate in rank between bituminous coal and anthracite; contains 8 to 22 percent volatile matter and from 91 to 93 percent carbon.

semi-water gas—a mixture of carbon monoxide, carbon dioxide, hydrogen, and nitrogen, obtained by passing an air-steam mixture through a hot bed of coke, having a higher heating value of about 120 Btu per standard cubic foot.

sensible heat—that heat which results in only the elevation of the temperature of a substance with no phase changes.

shift conversion—process for the production of gas with a desired carbon monoxide content from crude gases derived from coal gasification; carbon monoxide-rich gas is saturated with steam and passed through a catalytic reactor where the carbon monoxide reacts with steam to produce hydrogen and carbon dioxide, the latter being subsequently removed in a scrubber employing a suitable sorbent.

sintering—the agglomeration of solids at temperatures below their melting point, usually as a consequence of heat and pressure.

slag—molten coal ash composed primarily of silica, alumina, iron oxides, and calcium and magnesium oxides.

slurry—a suspension of pulverized solid in a liquid.

solvation—the association or combination of molecules of solvent with solute ions or molecules.

solvent—that component of a solution which is present in excess; liquid used to dissolve a substance.

solvent extraction—selective solution of coal constituents from finely divided coal particles into a suitable solvent after intimate mixing, usually at high temperatures and pressures in the presence of hydrogen, with or without a catalyst, followed by phase separation.

solvent refined coal (SRC)—a coal extract derived by solvent extraction; a brittle, vitreous solid (m.p. 300° F to 400° F) containing about 0.1 percent ash and about 10 percent of the sulfur in the original coal feedstock; calorific value is about 16,000 Btu per pound; may be used as a clean fuel for power generation by combustion; utilized for the production of high-grade metallurgical coke, anode carbon, and activated carbon by coking, or hydrogenated to produce synthetic crude oil.

space velocity—volume of a gas (measured at standard temperature and pressure) or liquid passing through a given volume of catalyst in a unit time.

specific gravity—ratio of the weight of any volume of a substance to the weight of an equal volume of water at 4°C.

specific heat—heat capacity of a substance as compared with the heat capacity of an equal weight of water.

standard cubic foot (SCF)—the volume of a gas at standard conditions of temperature and pressure. The American Gas Association

uses moisture-free gas at 60° F and 30 inches of mercury (1.0037 atm) as its standard conditions. The pressure standard is not universal in the gas industry: 14.7 psia (1.000 atm) and 14.4 psia (0.980 atm) are also used. The scientific community uses 32° F and 1 atm as standard conditions.

stoichiometry—the definite proportions in which molecules react chemically to form new molecules.

stripping—the removal of the more volatile components from a liquid mixture of compounds.

subbituminous coal—the rank of coal between bituminous and lignite, classified by ASTM as having a range of heating values between 8,300 and 11,000 Btu per pound on a moist mineral-matter-free basis.

substitute natural gas (SNG)—a gas produced from coal, oil sands, or oil shale conforming to natural gas standards.

superficial velocity—the linear velocity of a fluid flowing through a bed of solid particles calculated as though the particles were not present.

superheater—a heat exchanger which adds heat to the saturated steam leaving a boiler.

syncrude—synthetic crude oil, oil produced by the hydrogenation of coal, coal extracts, oil sands, or oil shale, which is similar to petroleum crude.

synthesis gas—a mixture of hydrogen and carbon monoxide which can be reacted to yield a hydrocarbon.

tail gas—a gas issuing from a gas-treatment unit which may be recycled to the process or exhausted.

tar (coal)—a dark brown or black, viscous, combustible liquid formed by the destructive distillation of coal.

therm—a unit of heat used as a basis for the sale of natural gas; equal to 100,000 Btu.

topping cycle—the higher temperature thermodynamic power cycle of a combined-cycle system.

turndown ratio—the minimum ratio of actual flowrate to design flowrate at which a process unit can be operated.

ultimate analysis—the determination by prescribed method of carbon and hydrogen in the material as found in the gaseous products of its complete combustion, the determination of sulfur, nitrogen, and ash in the material as a whole and the estimation of oxygen by difference; may be reported on different bases, such as as-received (or as-fired), dry, mineral-matter-free (mmf), and dry mineral-matter-free (dmmf).

Venturi scrubber—a gas cleaning device which involves the injection of water into a stream of dust-laden gas flowing at a high velocity through a contracted portion of a duct, thus transferring the dust particles to the water droplets which are subsequently removed.

volatile matter—those constituents of coal, exclusive of moisture, that are liberated from a sample when heated to 1750° F for seven minutes in the absence of oxygen.

water gas—gas produced by the reaction of carbon (in coal or coke) and steam to yield mixtures of carbon monoxide and hydrogen; similar to synthesis gas.

water gas shift—the reaction between water vapor and carbon monoxide to produce hydrogen and carbon dioxide or the reverse: $\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{H}_2 + \text{CO}_2$.

working fluid—a gas stream which directly does work, e.g., powering a gas turbine.