U.S. DEPARTMENT OF ENERGY
Division of Coal Conversion

QUARTERLY REPORT

July - September 1977

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COAL

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COAL LIQUEFACTION

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Washington, D.C. 20545

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FOREWORD

This document was prepared by the Energy Research and Development Administration (ERDA) prior to the activation of the Department of Energy (DOE) by P.L. 95-91 on October 1, 1977. Therefore, wherever ERDA is mentioned, it should be noted that its functions have been transferred to DOE.

The technical reviews contained in this document were prepared by Cameron Engineers, Inc., Denver, Colorado, based on information supplied by DOE and its various contractors. The reviews, as they appear herein, have been authorized for publication by representatives of DOE. Cameron Engineers, Inc. assumes responsibility for the accuracy of the reviews but not for the accuracy of the information provided by either DOE or its contractors.

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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 Office of Fossil Energy of the Energy Research and Development Administration (ERDA) 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.

ERDA's program for the conversion of coal to liquid fuels was begun by two of ERDA'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 also have the potential for use as chemical feedstocks.

To provide efficient and practical means of utilizing coal resources, ERDA is sponsoring the development of several conversion processes that are currently in the pilot plant stage. Responsibility for the design, construction, and operation of the *Solvent-Refined Coal (SRC) Process* pilot plant at Ft. Lewis, Washington is assigned to Pittsburg and Midway Coal Mining Company. A record of 61 days of continuous operation was completed during the quarter. Operability and yields with a blend of Kentucky No. 9 and 14 coal were explored, and testing with Illinois No. 6 coal also began. The Merriam Laboratory continued process variable studies with coals other than the Kentucky coal previously used for the SRC process studies.

Ashland Synthetic Fuels, Inc. (ASFI) is responsible for management of the construction of the *H-Coal Project* pilot plant. Construction continued on schedule and was 27 percent complete at the end of the quarter. Hydrocarbon

Research, Inc. (HRI) successfully completed a 30-day PDU run using Illinois No. 6 coal with very few operational problems. Other studies continued throughout the quarter on the bench-scale units and the solvent precipitation unit.

ERDA, 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 was processed most of the quarter in order to provide data for producing SRC from this 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. During the quarter, both Wyoming subbituminous and Illinois No. 6 bituminous coals were studied in the one-ton-per-day Coal Liquefaction Pilot Plant. Process variable studies continued on the 50-pound-per-day unit and comparisons were made between Wyoming and Illinois coal feeds.

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*. The contract was extended during the quarter, and catalyst screening runs were completed on four commercially available desulfurization/denitrogenation catalysts. A fifth was under investigation at the end of the quarter.

Fluor Engineers and Constructors, Inc. is responsible for reactivating the ERDA-owned *Cresap Test Facility* in Cresap, West Virginia. Final construction activities were completed during the quarter, and the plant was returned to Liquefied Coal Development Corporation. Start-up operations continued throughout the quarter.

Construction of the 10-ton-per-day process development unit (PDU) to test the *Synthoil Process* is being managed by Foster Wheeler Energy Corporation. Associated laboratory research was conducted by the Pittsburgh Energy Research Center (PERC), Sandia Laboratories, Argonne National Laboratory, and Battelle Memorial Institute (Columbus Laboratories). The longest continuous run accomplished to date was completed on the 1/2 ton-per-day unit during the quarter. A quality liquid product was produced for 1068 hours of total run time demonstrating operability of the process.

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. Carbonization PDU tests were conducted

during the quarter with West Kentucky coal. In the hydrogenation area, a comparison was made of vapor stripping and vacuum distillation as techniques for separating oil from coal-conversion effluent. Tests were made to improve the properties of binder materials for cokemaking also.

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. The second series of one-ton-per-hour reactor tests was completed during the quarter. The Phase III effort was initiated in August.

Studies are being conducted by the Occidental Research Corporation to evaluate the commercial potential of the company's *Flash Pyrolysis Coal Liquefaction Process*. Work during the quarter consisted of efforts to demonstrate the operability of the process development unit with caking bituminous coal. Modifications made to the PDU improved the char rate control and a 22 hour run was completed.

Research to improve solid-liquid separation is being conducted by Johns-Manville Sales Corporation in their *Filtration Process and Equipment Studies*. During the quarter 33 filtration runs were conducted before modifying the leaf system for testing.

Dow Chemical Company has conducted studies dealing with the production of *Chemical From Coal* using the liquids from four coal liquefaction processes (COED, Synthoil, H-Coal, and SRC) in standard petrochemical processing experiments. Efforts during the quarter were directed at compiling the final report.

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. Three hydrocarbonization runs were conducted 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. Nearly all the major equipment for the one-ton-per-day process development unit was in place at the end of the quarter, and the feed system was being readied for pressure testing.

Exxon Research and Engineering is studying the potential of *Fluid Coking of Coal Liquefaction Residues* to recover distillate liquids. The continuous

stirred coking unit process variable scoping study of stripped SRC Antisolvent Deashing Underflow began in July and was completed by the end of the quarter.

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*. First stage hydrocracking process variable studies on H-Coal gas oil were concluded during the quarter. Second stage hydrocracking of 600°F+ H-Coal hydrocrackate to fuel oil began during September. Hydrotreating studies also began on three catalysts. Platforming runs on H-Coal process liquids were completed.

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. During the quarter, laboratory evaluations were performed on a sample from the SRC II plant in Tacoma and a sample from the Exxon Donor Solvent coal liquefaction process.

The *Production of Clean Industrial and Transportation Fuels From Coal* is being investigated by the Lummus Company using a multistage ebullated-bed catalytic reactor developed through the firm's activities with a bench-scale pilot plant. Integrated shakedown operations continued during the quarter on the modified 30-pound-per-hour bench-scale expanded-bed test unit.

I. SOLVENT-REFINED COAL (SRC) PROCESS

THE PITTSBURG & MIDWAY COAL MINING
COMPANY

MERRIAM, KANSAS

Plant Site: Tacoma, Washington

Contract No.: E(49-18)-496

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

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 ERDA. 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, now part of ERDA) to study the technical feasibility of a coal de-ashing

LEGEND: △ BEGIN MILESTONE
▽ COMPLETE MILESTONE

Figure I-1. SOLVENT-REFINED COAL PROGRAM SCHEDULE

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 temperatures. 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.

Figure I-2. SRC PROCESS SCHEMATIC

PROGRESS DURING JULY-SEPTEMBER 1977

Summary

A record of 61 days of continuous operation was completed during the quarter. Operability and yields with a blend of Kentucky No. 9 and 14 coal were explored with emphasis on conditions yielding organic residues compatible with hydrogen generation needs. Testing with Illinois No. 6 coal also began. The Merriam Laboratory continued process variable studies with coals other than the Kentucky coal previously used for the SRC process investigations.

Pilot Plant

During July, a blend of Kentucky No. 9 and 14 coal was processed during 25 days of operation in the SRC II

mode. One SRC II material balance run was completed during this period. A total liquid yield of 33 percent was obtained with SRC yield about 20 percent. The run was made at conditions of 25 percent coal, 0.8 hour residence time, 860° F, and 1900 psig. Total slurry feed rate was 8026 pounds per hour. The increased coal feed rates led to problems with the eductor coal mixing system. A number of coal outages were taken to clean the scrubber, and two major plugs occurred when slurry recirculation problems reduced motive fluid flow. The vapor removal system of the new mix tanks was modified and the unit returned to operation.

The pilot plant processed coal for 31 days in August, completing four material balance runs feeding a blend of Kentucky No. 9 and 14 coal. Data from these runs indicated that increasing coal concentration results in a lower distillate yield and higher SRC yield. The effect of coal concentration is non-linear, partly due to the fact

that some solvent must be added to the slurry to permit steady operation at higher coal concentrations.

Operating problems continued to be concentrated in the coal slurry mixing system, especially at higher coal concentrations. Frequent plugs occurred in the eductor system, usually as a result of slurry recirculation pumping problems. The new mix tank performed well during the first part of the month, but plugging problems necessitated further modifications.

A record of 61 days of continuous operation ended on September 17 when a loss of both recycle compressors forced a complete plant shutdown. The feed coal was switched from the blend of Kentucky No. 9 and 14 previously used to Illinois No. 6 on September 2. Two material balance runs were made with the Illinois coal. Numerous plugging problems were experienced in the new vacuum flash system. It was demonstrated, however, that consistent production of a vacuum bottoms product containing 32 percent ash and 44 percent total solids was possible.

Laboratory — Merriam, Kansas

Three SRC II runs were made with a blend of Kentucky No. 9 and 14 coal in July to explore the effect of varying pressure. The composition of the feed slurry was 30 percent coal and 70 percent unfiltered coal solution,

nominal liquid residence time was one hour and nominal dissolver temperature was 455°C. Yields varied in the expected manner with oil yield increasing and SRC yield decreasing with increasing pressure. There was little variation in heavy distillate fuel composition with varying pressure. Hydrogen content varied only between 8.13 and 8.19 percent and sulfur content between 0.20 and 0.24 percent.

Eight SRC II runs were made in August to determine the suitability of coals other than the Kentucky coal used normally for the process. Two Pittsburgh seam coals, Consol Ireland and Blacksville No. 2, and one Illinois No. 6 coal were investigated. The Pittsburgh seam coal investigated initially (from the Ireland mine) was found to be as fully satisfactory as the Kentucky coal. In fact, yields with this coal were found to be more favorable than those obtained with a Kentucky coal under the same conditions.

The other Pittsburgh seam coal, from the Blacksville No. 2 mine, was found to be less reactive with an organic residue yield of 37.5 percent being observed under the same conditions. The Blacksville coal was lower in iron content and higher in apparent rank than the Ireland coal. Two additional runs on the Blacksville coal in September confirmed the earlier results that the coal is less reactive than either Kentucky No. 9 or 14 from the Colonial mine or the Illinois No. 6 from the River King mine for SRC II processing.

II. H-COAL PROJECT

Plant Site: Catlettsburg, Kentucky
Total Funding: \$179,000,000
ERDA: \$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) ERDA, (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 a 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.

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

Figure II-2. H-COAL PROCESS SCHEMATIC

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 con-

firmed the technical and economic feasibility of the H-Coal process.

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, midwestern, 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. E(49-18)-2260. Field work began in December 1976 by Badger Plants, Inc., subcontractor of 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 JULY-SEPTEMBER 1977

Summary

The H-Coal pilot plant was 27 percent complete at the end of the quarter. Amendments to the Clean Air Act now require that Ashland obtain authority from the State of Kentucky to construct and operate the facility. A 30-day PDU run using Illinois No. 6 coal was successfully completed during the quarter with very few operational problems. Other studies continued throughout the quarter on the bench-scale units, fluid dynamics, and the solvent precipitation unit.

Pilot Plant Construction

As of the end of the quarter, the pilot plant was approximately 27 percent complete and progressing on schedule. Mechanical completion of the plant is scheduled for completion in June 1978, with overall completion in July 1978. Some delay was encountered during the quarter due to late approval of the Lummus anti-solvent de-ashing unit and requests by ERDA to consider alternative construction materials for the Change House. The HRI Phase I Engineering effort was 75 percent complete by the end of the quarter and is expected to be finished by December 15, 1977.

Due to amendments to the Clean Air Act made in August, authority must now be obtained from the State of Kentucky to construct the pilot plant and operate an air emission source. A letter was prepared and sent to the Air Pollution Control Division describing the steps being taken to complete the air permit application.

Laboratory Support

During the month of July, PDU Run 5, a demonstration of steady state operation in the syncrude mode with semi-continuous catalyst replacement, was terminated after seven days of operation due to severe corrosion of the thermocouple assembly controlling reaction temperatures. The cause of the problem was traced to the erroneous use of Inconel 600 alloy rather than 316 stainless steel. During the seven-day period, however, catalyst was successfully removed from the reactor, while under ebullation, through the catalyst removal bomb.

Run 5 was restarted in August using Illinois No. 6 coal, starting with fresh catalyst and using catalyst replacement to approach equilibrium conditions. The run was completed in September, and all target conditions were met with the exception of reactor residuum concentration. A concentration of 40 percent was achieved, versus the targeted 44 percent. This was due to temperature limitations of the slurry feed system. A catalyst replace-

ment rate of one pound per ton was achieved with excellent stability and very few process problems.

Modifications to the two bench-scale units continued on schedule throughout the reporting period. These units, designed for startup in December, will be used for testing denitrogenation and higher density desulfurization catalysts. Fluid dynamics studies to confirm the scale-up of the tubular recycle cup were completed during the quarter. Further PDU testing is planned to optimize the product withdrawal tube location.

In preparation for the addition of an on-line solvent precipitation unit to the PDU, various fractions of atmospheric still overhead and reagent grade hydrocarbons were analyzed for Kauri-butanol value. The anti-solvent currently used in the H-Coal solids separation system has a value of 133.6, whereas a good anti-solvent is said to have a value in the mid-twenties. The discrepancy between the two values is attributed primarily to the parameters of processes involved. In spite of its high value, the anti-solvent now in use produces a product with less than, or equal to, 0.1 percent ash.

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III. OPERATION OF SOLVENT-REFINED COAL PILOT PLANT

SOUTHERN COMPANY SERVICES, INC.
BIRMINGHAM, ALABAMA

Plant Site: Wilsonville, Alabama
Contract No.: E(49-18)-2270
Contract Funding: \$8,729,892
ERDA: \$5,741,892
Industry: \$2,988,000

INTRODUCTION

The operation of the solvent-refined coal (SRC) pilot plant at Wilsonville is jointly sponsored by ERDA and the Electric Power Research Institute (EPRI). The schedule for the project is shown in Figure III-1. 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 clean distillate fuel and a clean high-Btu gas. A schematic of the process is shown in Figure III-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 single-stage reactor or dissolver which operates at 800°-875°F, and a total pressure of approxi-

Figure III-1. SRC PILOT PLANT OPERATION SCHEDULE

mately 1700 psig. Under these conditions, approximately 93 percent of the carbonaceous material in the coal feed is dissolved during the residence time of approximately 45 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 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.

Figure III-2. SRC PROCESS FLOW DIAGRAM

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. One subbituminous and four 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 JULY-SEPTEMBER 1977

The Wilsonville SRC pilot plant operated a total of 1308 hours during the quarter and the distillation section was on-stream for 1420 hours. All of the operational time was used to process Indiana coal from the Old Ben No. 1 mine. Two material balance runs were completed during the quarter. In July, the distillation system was adjusted and operated to yield a consistent boiling range process solvent. During one run in August, the sulfur content of the SRC product was in the range of 0.7 percent.

During August and September, the Sharples and Dorr-Oliver centrifuges were operated in series, with the Sharples underflow filtered in the Funda filter. Both machines operated satisfactorily for 123 hours. The

Sharples centrifuge reduced the solids content of the overflow to the expected level; however, specification product could not be obtained from the Dorr-Oliver unit. During a run in September, it was determined that a temperature of 550°-600°F was necessary to achieve a minimum ash content with the Dorr-Oliver centrifuge.

A series of checkout runs were conducted using hydroclones and anti-solvent for solids separation; however, several changes in equipment and piping are necessary before meaningful data can be obtained. The pilot plant was shut down on September 10 for modifications and repairs.

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IV. DONOR SOLVENT LIQUEFACTION PROCESS

EXXON RESEARCH AND ENGINEERING
COMPANY

FLORHAM PARK, NEW JERSEY

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

Contract Funding: \$227,259,604

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 IV-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 modification 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 IV-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°-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 bottoms slurry is coked to produce additional liquids.

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 C₁-C₂ gases from the liquefaction process.

LEGEND : BEGIN MILESTONE
 COMPLETE MILESTONE

Figure IV-1. DONOR SOLVENT PROJECT SCHEDULE

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

Figure IV-2. DONOR SOLVENT LIQUEFACTION PROCESS

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 three 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 JULY-SEPTEMBER 1977

Summary

Both Wyoming subbituminous and Illinois No. 6 bituminous coals were studied during the quarter in the one-ton-per-day Coal Liquefaction Pilot Plant. Studied during the tests were the effects of residence time, temperature, higher end-point solvents, and solids withdrawals. Process variable studies continued on the 50-pound-per-day unit and comparisons were made between Wyoming and Illinois coal feeds. A study was conducted to determine areas in which water usage could be improved. Other investigations revealed that increased liquid yields at higher reactor pressures do not compensate for the added equipment costs for high pressure operation. Arthur G. McKee Company was awarded the

engineering and procurement contract for the 250-ton-per-day plant and Daniel Construction Company was awarded the construction contract.

Coal Liquefaction Pilot Plant

Studies on the one-ton-per-day Coal Liquefaction Pilot Plant (CLPP) continued throughout the quarter. Two types of coal feed were used; Wyoming subbituminous and Illinois No. 6 bituminous. Tests with the subbituminous coal were designed to obtain more information at longer residence times than previously investigated. Five yield periods were made during the run, and conversions were in very good agreement with earlier CLPP studies.

The Illinois No. 6 tests were designed to determine the effects of recycling a "heavy-end" solvent to the reactor. This solvent had a higher boiling point than the standard solvent and closely approximated the solvent which will be tested in the Exxon Coal Liquefaction Pilot Plant (ECLP). The typical CLPP solvent contains less than ten weight percent material boiling above 700°F; however, to minimize flashing in the slurry preheater, the solvent may contain more than 20 weight percent of this material. The composition, and therefore the boiling point, of this material is controlled by varying the relative amounts of heavy vacuum gas oil contained in the solvent. No operational problems or yield effects were noted during the "heavy-end" solvent run.

Also studied during the bituminous coal tests was the effect of withdrawing solids from the liquefaction reactor. The initial results indicate that there are no significant yield effects associated with the solids withdrawal operation.

Engineering studies with the sectionalized slurry preheaters also continued during the quarter. The preheaters were operated with extended residence times at 840°F and mixed phase flow. It is generally believed that coking rates are a strong function of residence time at a given temperature. Therefore, the residence times in these preheaters are much longer than those expected for the ECLP slurry preheaters. The purpose of the current runs was to reinforce the data indicating that coking is not a problem at 840°F with mixed phase flow.

Other Studies

Process variable studies also continued during the quarter using Wyoming subbituminous coal in the 50-

pound-per-day Recycle Coal Liquefaction Unit (RCLU). The variables investigated were residence time and temperature. Analyses made on various liquefaction products showed that the liquids produced from Wyoming coal were ten times lower in sulfur content than liquids produced from Illinois coal. This result was expected as the sulfur content of the Wyoming coal (0.5 weight percent) was much lower than that of Illinois coal (4.0 weight percent). The carbon, hydrogen, nitrogen, and oxygen levels of the product from the two coals were similar.

Experiments also continued on the Fluidized Bed Unit (FBU) and Integrated Coking Gasification Unit (IKG). The FBU gasification tests showed that high carbon utilization may be attained in coal liquefaction bottoms flexicoking. Coke gasification levels during the tests varied from 70 to 90 percent. The IKG operated on Illinois coal bottoms from the CLPP and performed much as it did during previous testing.

A study was conducted to determine possible methods of improving water usage in a commercial EDS plant. Four water-saving categories were studied:

- Steps resulting from process modifications which are economically attractive.
- Reduction in makeup water by replacing steam reforming with partial oxidation.
- Use of effluent water to partially or totally provide cooling tower makeup requirements.
- Areas in which reductions are difficult or where fresh water use is mandatory.

A screening study was completed to define the economics of increasing reactor pressure from 1500 psi to 2500 psi hydrogen partial pressure (3125 psi total pressure). The study showed that the increased liquid yield obtained at the higher pressure is not sufficient to compensate for the significant increase in investment required. Attritability tests and engineering evaluations of alternative hydrogen manufacture process were also conducted during the quarter.

Contracts were awarded in September to Arthur G. McKee Company for engineering and procurement for the 250-ton-per-day Exxon Coal Liquefaction Pilot Plant (ECLP). The construction contract was awarded to Daniel Construction Company. McKee began engineering and procurement activities immediately following the contract award.

V. 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: \$591,122

INTRODUCTION

Cities Service, under ERDA 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 V-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,

Figure V-1. EXPANDED-BED HYDROPROCESSING
PROCESS SCHEMATIC

deposition of tar and coke is minimized and fine solids entrained in the feed will not lead to reactor plugging. 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. Experimental runs designed to determine the operating conditions necessary to process Solvent Refined Coal (SRC) in the commercial LC-Fining unit were completed during the year. The first 30-day catalyst aging run LCF-24, processing a blend of 67.0 weight percent SRC and 33.0 weight percent Koppers heavy residue creosote oil, was also completed. Conversion of the 850°F-plus material averaged 45.9 volume percent.

PROGRESS DURING JULY-SEPTEMBER 1977

Summary

The contract was extended during the quarter with the object of determining the effectiveness of commercially available catalysts for denitrogenation/desulfurization of an SRC extract in the LC-Fining Process expanded bed reactor. Catalyst screening runs were completed on

four commercially available desulfurization/denitrogenation catalysts. A fifth was under investigation at the end of the quarter.

Discussion Activities

The base case catalyst screening run (LCF-29) on a cobalt-molybdenum catalyst was completed at the beginning of the quarter. Two hydrogenated Koppers Heavy Residue Creosote Oil solvents, a 400°F and a 500°F IBP solvent were prepared in the Lummus HDS unit. No significant differences in conversion and denitrogenation were noted for the two solvents.

Following an initial conversion of 77 weight percent of 850°F+ material, the conversion remained approximately constant at 65 weight percent from period 5 through period 12 of a 13 period run. The 400°F solvent was run first and changed to the 500°F IBP solvent at period 7/8. At that time the denitrogenation function increased a small amount, which only slightly perturbed the declining trend. The hydrogen content of the heavy oil through periods 5 through 12 was approximately 7.5 weight percent.

Of the catalyst tested, a nickel-molybdenum catalyst was the best for desulfurization and denitrogenation. After approximately four 24-hour periods, this catalyst stabilized for an additional six periods at a conversion level of approximately 75 weight percent of 850°F plus material. The conversion level was a preliminary value based only upon the weight percent 850°F plus fractions of the feed and heavy oil product.

A nickel-tungston catalyst had the same initial activity with respect to conversion and nitrogen removal, but did not appear to stabilize after four periods.

Neither of the cobalt-molybdenum catalysts tested were as active as the nickel-molybdenum catalyst. A second nickel-molybdenum catalyst was being tested at the end of the quarter.

VI. CRESAP TEST FACILITY

FLUOR ENGINEERS AND CONSTRUCTORS,
INC.

LOS ANGELES, CALIFORNIA

Project Site: Cresap, West Virginia

Contract No.: E(49-18)-1517

Contract Funding: \$45,699,780

ERDA: \$43,042,140

Industry: \$ 2,657,640

INTRODUCTION

Under contract to ERDA, Fluor Engineers and Constructors, Inc., is renovating and will operate an ERDA-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 compo-

nents 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 VI-1.

PROCESS DESCRIPTION

In this process, shown schematically in Figure VI-2, coal is converted to low-sulfur liquid fuel. The coal is first

Figure VI-1. CRESAP TEST FACILITY PROGRAM SCHEDULE

Figure VI-2. LIQUID FUEL FROM COAL PROCESS SCHEMATIC

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 extracted 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.

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 JULY-SEPTEMBER 1977

All construction activities were completed during the quarter at the Cresap Test Facility, and the Fluor engineering and construction field office was closed. Care, custody, and control of the plant was returned to Liquefied Coal Development Corporation in August. Startup efforts for the front-end began August 1, and

startup activities for the back-end commenced on August 19.

By the end of the quarter, integrated hot solvent circulation was established among the extraction, solids separation, and solvent recovery sections of the pilot plant. Char combustion was established in the low temperature carbonization section.

The front-end startup was interrupted on different occasions to repair the numerous leaks which developed as normal operating temperatures were approached; to correct a high pressure drop in the extraction section; and for further repairs to the extractor shaft seals. The front-end should be ready to process coal early next quarter. Extract granulation will start shortly thereafter.

Operability, using nitrogen, of one of the makeup hydrogen compressors in series with one of the recycle hydrogen compressors was demonstrated at 3000 psig. The pressure checking of the hydrogenation section was slowed due to leaks of some of the conval valves. Temporary craftsmen were added to correct the problem and speed winterization activities.

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VII. SYNTHOIL PROCESS

FOSTER WHEELER ENERGY CORPORATION

LIVINGSTON, NEW JERSEY

Contract Funding: \$24,425,776

SANDIA LABORATORIES

ALBUQUERQUE, NEW MEXICO

Contract Funding: \$1,636,000

ARGONNE NATIONAL LABORATORY

ARGONNE, ILLINOIS

Contract Funding: \$1,574,000

BATTELLE MEMORIAL INSTITUTE,

COLUMBUS LABORATORIES

COLUMBUS, OHIO

Contract Funding: \$156,000

Plant Site: Bruceton, Pennsylvania

INTRODUCTION

Development of the Synthoil process, initiated by the U.S. Bureau of Mines, is currently being managed by ERDA 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. In addition, PERC is monitoring laboratory research on various aspects of the Synthoil process being conducted by Sandia Laboratories, and Argonne National Laboratory. Research being conducted at Exxon Research and

Engineering and Battelle Memorial Institute is being monitored by the Morgantown Energy Research Center (MERC) in West Virginia. The schedule for the Synthoil project is shown in Figure VII-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 VII-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

Figure VII-1. SYNTHOIL PROGRAM SCHEDULE

Figure VII-2. SYNTHOIL PROCESS SCHEMATIC

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 that processed five pounds of slurry per hour. Experimental work was conducted on various coals, such as Pittsburgh seam, Indiana No. 5, Middle Kittanning 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. Economics 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 JULY-SEPTEMBER 1977

Overall activity on the 10 ton-per-day process development unit (PDU) was 82 percent complete, but progress was slow during the quarter due to late delivery of equipment and materials which has caused a three-month slippage in the schedule. The Request for Proposal to select an operating contractor for the PDU was cancelled in July.

Run FB-61 was in progress at the beginning of the quarter on the 1/2 ton-per-day unit. After 492 hours of successful operation, the plant was carefully shut down to accommodate annual laboratory maintenance and electrical system modifications. The unit was first flushed with a large amount of Synthoil centrifuged liquid product, then cooled by flowing recycle gas, and finally depressurized and maintained under a hydrogen blanket at 600 psig. After being held in this condition for 45 days, the plant was restarted and an additional 576 hours of successful operation completed for a total of 1068 hours.

Throughout the extended run period, the quality of the product oil was very good and remained consistent. Viscosity of the centrifuged liquid product was about 65 SSF (180°F) while the sulfur content held nearly constant at about 0.6 weight percent. Double centrifuging of the oil product controlled the ash content to 2 percent.

Construction of the Synthoil II one ton-per-day unit was 20 percent complete at the end of the quarter. The installation of the preheater and reactor furnaces was completed.

As part of the engineering design for scale-up of the Synthoil process, a library of computer programs capable of solving complex design-related equations was developed and tested.

Exxon Research and Engineering Company published their final report "Chemical Properties of Synthoil Products and Feeds" in September. The publication is available from the National Technical Information Service, U.S. Department of Commerce, Springfield, Virginia, 22167.

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VIII. CLEAN COKE PROCESS

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

Plant Site: Pittsburgh, Pennsylvania
Contract No.: E(49-18)-1220
Contract Funding: \$11,792,329
ERDA: \$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 ERDA. The contract was initiated in 1972 under the auspices of the Office of Coal Research (OCR, now a part of ERDA). 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 VIII-1.

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 VIII-1. CLEAN COKE PROCESS SCHEDULE

PROCESS DESCRIPTION

The Clean Coke process is shown schematically in Figure VIII-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.

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

Figure VIII-2. CLEAN COKE PROCESS SCHEMATIC

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 hinder.

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 ERDA 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 JULY-SEPTEMBER 1977

Summary

Work in the carbonization area during the quarter included PDU processing of West Kentucky coal through first-stage carbonization at 820°F, and second stage carbonization at both 1400°F and 1470°F. To date, the PDU has been operated for a total of 4370 hours with about 2960 hours at temperatures ranging from 1250 to 1470°F.

In the hydrogenation area, a comparison was made of vapor stripping and vacuum distillation as techniques for separating oil from coal-conversion effluent.

Tests were made to improve the properties of binder materials for cokemaking. A method was also devised to evaluate binders in small coke specimens prior to their use in pelletizing tests. Preparation and testing of binders from SRC product also continued.

Carbonization Process Development Unit

In July, a first stage carbonization run at 820°F and 165 psia was completed to obtain material-balance data. Steady-state operation for 56 hours produced 2214 pounds of semichar from 2592 pounds of oxidized coal. Weight-balance data showed an accountability of 99.2 percent.

A second stage carbonization run was made at 1400°F and 165 psia to evaluate residence time versus char sulfur content. Sulfur contents of the chars produced at 190 and 106 minutes residence time were 0.67 and 0.96 percent, respectively. The feed for this run was West Kentucky semichar containing 1.9 to 2.0 percent sulfur.

A high temperature run at 1470°F and 165 psia was also completed. At a residence time of 112 minutes, 831 pounds of West Kentucky semichar was processed to yield 625 pounds of char containing 0.68 percent sulfur.

Tests were also made in the 3-inch continuous carbonizer to determine optimum conditions for carbonizing Pittsburgh Seam coal in the PDU.

Hydrogenation Process Development

The products obtained by vapor stripping and vacuum distillation of converter effluent were compared. The

effluent for this work was prepared in a coal-conversion run at 850°F and 300 psig with a 35 percent slurry of raw Illinois coal in oil. Vapor stripping recovered 77 percent of the converter effluent as oil and produced a residue having a softening point of 71.6°C. The distillation yielded less oil, 75 percent, and produced a residue having a higher softening point, 75.3°C.

The results demonstrated the unique ability of the vapor stripper to carry over or recover high-boiling high-molecular-weight materials without subjecting them to conditions that could result in degradation or repolymerization.

Binder-Preparation Process Development Unit

The flash vaporizer was operated during the quarter to process liquid blowdown from the carbonization PDU. This operation recovers the clean wash oil for recycle, and the carbonization tar (heavy oil) is used in binder preparation. Air blowing was tested during the quarter as a means of improving the properties of this heavy oil as a binder in cokemaking.

In September, tests were made to determine the effects on binder properties of a treatment consisting of air blowing followed by heat soaking. The treated binder was a 50/50 blend of hydrogenated heavy cresote and SRC.

A laboratory program was also initiated to allow an evaluation of prospective binders prior to their use in pelletization tests. Mixtures of pulverized (minus 100 mesh) process char and binder in various proportions are pressed in a mold at 3000 psi to form small cylindrical test specimens. After the specimens are cured and calcined at controlled temperatures, their crushing strength is determined. An increase in strength would indicate an improvement in binder preparation.

Coke Preparation

Testing of binders prepared from SRC product continued throughout the quarter. In September, a pelletization run was made with a binder consisting of 60 percent SRC and 40 percent methylnaphthalene. It was anticipated that the curing step would remove the methylnaphthalene and leave only the SRC as the binder during calcining. Thus, the binding properties of the SRC alone can be evaluated.

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IX. LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

ROCKETDYNE DIVISION
ROCKWELL INTERNATIONAL CORPORATION
CANOGA PARK, CALIFORNIA

Plant Site: Canoga Park, California
Contract No.: E(49-18)-2044
Contract Funding: \$994,375
(100% ERDA)

INTRODUCTION

Under contract to ERDA, 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 ERDA 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 feasibility of this process. The schedule for the development of this process is shown in Figure IX-1.

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

In addition to the ERDA 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 IX-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.

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 made at the end of May, and final testing on the unit was completed by the end of the year. 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.

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

PROGRESS DURING JULY-SEPTEMBER 1977

Summary

The second series of one-ton-per-hour reactor tests was completed during the quarter. The Phase III effort was initiated in August. Testing of two new injector configurations is planned during future tests.

Discussion of Activities

The second series of one-ton-per-hour reactor tests was completed in July. Three tests were made with the coal used during the earlier tests (Pittsburg and Midway, Colonial mine, Kentucky No. 9 and 14). Three additional tests were made with a similar western Kentucky coal (Island Creek, Hamilton No. 2 mine, Kentucky No. 9)

pulverized to 70 percent minus 200 mesh. Two tests made with Kentucky No. 9 pulverized to 90 percent minus 200 mesh.

Results indicated that the overall conversion was insensitive to reactor residence time over the range from 30 to 640 milliseconds, but the conversion to liquids decreases at longer residence time. At 640 milliseconds, all of the carbon was converted to gas.

In August, Phase III of the contract was initiated. By the end of the quarter, seven tests had been completed in the one-ton-per-hour reactor with a 3.37 inch I.D. A residence time of approximately 120 milliseconds is obtained in the reactor at the nominal flow rates of 1000 psi and 1700°F. Previous test results indicated that a lighter liquid product was obtained at longer residence times and higher temperatures and pressures than those which gave the maximum liquid yield. Consequently, the testing was directed toward exploring these effects.

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X. 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% ERDA)

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 ERDA. Figure X-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.

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 X-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

LEGEND: ▲ BEGIN MILESTONE
▼ COMPLETE MILESTONE

Figure X-1. FLASH PYROLYSIS PROJECT SCHEDULE

made to the PDU improved the char rate control and a 22 hour run was completed.

Process Development Unit

During the quarter several successive changes were made to the char circulation system to improve unit operability for extended operation of the PDU while processing Kentucky No. 9 seam coal. The mechanical char feed system was replaced with a lift-tube feeder. Initial tests with the new feeding device indicated char rate control was better and the pyrolysis system was less sensitive to upsets.

Bench-scale runs confirmed the low tar yield from subbituminous coal of about 8 to 10 percent obtained in

the PDU baseline runs with char heating as compared to 15 percent yield using electric wall heating without char. In addition, PDU tests indicated that when the char to coal ratio was increased by lowering the coal feed rate from 200 pounds per hour to 50 pounds per hour at nominal char rate of 2000 pounds per hour, the tar yield was further reduced from about 10 percent to about 5 percent. The results suggest that product tar is absorbed on the char surface and cracked.

Tar characterization studies also continued with a modified SARA separation method developed to provide information on the major chemical components present in the full-range tar.

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XI. FILTRATION PROCESS AND EQUIPMENT STUDIES

JOHNS-MANVILLE SALES CORPORATION
DENVER, COLORADO

Project Site: Denver, Colorado
Contract No.: E(49-18)-2007
Contract Funding: \$190,000
(100% ERDA)

INTRODUCTION

Under contract to ERDA, 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 ERDA, and Phase II is being supported by the Electric Power Research Institute (EPRI) in cooperation with the ERDA 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 products that have been cooled. Due to the chemical and physical changes which may take place during aging and irreversible alternations 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 cooperation with P&M, Johns-Manville will apply its filtration knowledge to improve the filtration rate and reduce the filter aid usage on existing Goslin-Birming-

ham filters. Similar filter process optimization studies will then be conducted at other ERDA-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 JULY-SEPTEMBER 1977

During July, 16 filtration runs were conducted on unfiltered solvent-refined (SRC) filter feed from the

Wilsonville SRC plant. Each of the runs consisted of 15 to 25 immersions of the filter leaf under controlled conditions of temperature and differential pressure. Usually six filtration rate data points were obtained during each run, each point being based on one to four immersions. With the Wilsonville material, a blade advance of one mil per cycle was adequate to remove the solid particles which penetrate and tend to blind the CELITE AF5 precoat material. This had not been the case with the Tacoma Filter Feed TFF 216 material.

Seventeen filtration runs were conducted in August using Tacoma Filter Feed TFF 216. The runs using AF5 precoat confirmed the results of similar runs made in June. Later tests used AF2 precoat, a finer diatomite with less permeability. It was suggested that AF2 might be practical on a large scale in the interest of filter aid economy. It would, however, have to be proven on a large scale with a thickness up to six-inches. This idea may be evaluated on the new 50-square-foot pilot filter scheduled for operation in February 1978 in Tacoma.

During August, efforts were made to modify the leaf system to allow "in situ" operation for future runs. The new features would permit precoating to be done in place at operating temperatures and pressures. The unit was fabricated, installed, and mechanically tested, but proved unsatisfactory when used for filtration operations in September. A new concept was designed and construction began in preparation for testing in October. When the system begins operating satisfactorily, it will be used to evaluate AF5 precoat, AF6 precoat, AF2 precoat, and Standard SUPER-CEL precoat for filtering Tacoma feed.

The 50-square-foot pilot model rotary pressure precoat filter was completed by Stearns-Roger and received by Pittsburgh and Midway Coal Mining Company in August. Installation of the unit at the Fort Lewis SRC facility is scheduled to begin in October and to be completed by February 1978.

XII. CHEMICALS FROM COAL

THE DOW CHEMICAL COMPANY
MIDLAND, MICHIGAN

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

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 ERDA, 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 gas 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 JULY-SEPTEMBER 1977

Efforts during the quarter were directed at compiling the final report.

XIII. R&D ENGINEERING SERVICES — HYDROCARBONIZATION

OAK RIDGE NATIONAL LABORATORY
OAK RIDGE, TENNESSEE

Contract No.: E(49-18)-1/4U
Contract Funding: \$2,135,000
(100% ERDA)

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.

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 broilers, 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 XIII-1.

The work under this contract is divided into three tasks:

- 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.

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 1110°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 XIII-1. FLOW DIAGRAM OF BENCH-SCALE HYDROCARBONIZATION SYSTEM

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

PROGRESS DURING JULY-SEPTEMBER 1977

Summary

Three hydrocarbonization runs were conducted during the quarter, one using Wyodak subbituminous coal feed and two using Illinois No. 6 coal. For the runs with Illinois coal, the feed was pretreated with lime and sodium hydroxide in an autoclave to reduce caking tendencies. The second test with the Illinois No. 6 coal was quite successful, exhibiting good fluidization and no caking. As part of the supporting research, a newly-designed gas distributor was evaluated. The gas requirement for the new design was greater than the standard design, but good downcomer fluidization was achieved.

Bench-Scale Tests

The first of three hydrocarbonization runs conducted during the quarter was on Wyodak subbituminous coal. This run, HC-21, was performed to prepare char for future runs with char/Illinois No. 6 coal feed and also to measure the oil yield in the uniformly fluidized-bed reactor using coal which was prepared under an inert gas blanket. Conditions for the run were 1035°F and 267-300 psig with the average coal feed rate approximately 10.1

pounds per hour. Temperature control and operation was generally good, but coal feed stopped several times.

The second run, H-22, was conducted in August. The feed used was 3/1 ratio of char to Illinois No. 6 coal. To reduce caking properties, the coal was pretreated with an aqueous mixture of lime and caustic in an autoclave. The primary objective of the run was to determine if the caking problems in the reactor, which had been experienced in previous runs with Illinois No. 6 coal, could be eliminated by ensuring that the material leaving the draft tube was heated above 1000°F.

During HC-22, caking began to occur after approximately 20 minutes. The draft tube became plugged after about 35 minutes of run time. Evidently the coal was not sufficiently devolatilized in the draft tube to prevent caking after it was discharged into the downcomer region.

The third run, HC-23, used Illinois No. 6 coal as the feed. In order to eliminate the caking tendency of the coal, and in some cases improve the liquid yield, the coal was pretreated with an aqueous mixture of lime and sodium hydroxide at approximately 250°C. The treated coal was passed through a separator and it was found that the caustic treatment had caused considerable comminution of the coal particles. Fines were removed until the resulting material had desirable flow characteristics. Using the same conditions as in HC-22, operation was generally good except that the feed rate was lower than planned, possibly due to the large amount of fines in the

feed. Fluidization of the bed appeared to be good, with no indication of caking.

Supporting Research

During July, cold model tests indicated that dimensionless gas velocity, the ratio of actual velocity to minimum fluidization velocity, may be used satisfactorily

to evaluate and scale fluidized-bed behavior. An alternative gas distributor was also designed to assure downcomer fluidization. The two-dimensional mock-up was modified and operated to test the new configuration. It was found that the new design required more gas to achieve the same solid circulation rates as the standard distributor and it appeared limited to circulation rates below those achievable with the standard distributor. However, the new design did permit downcomer fluidization independent of whether the bed was circulating.

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XIV. 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
ERDA: \$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 ERDA 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 XIV-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 XIV-2. The coal is dried and pulverized 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 JULY-SEPTEMBER 1977

Summary

Nearly all the major equipment was in place for the one-ton-per-day Process Development Unit (PDU) at the end of the quarter, and the feed system was being readied for pressure testing. Runs on the bench-scale hydrocracker in August completed the original program on the variable study of Colstrip coal hydrocracking kinetics.

Process Development Unit

Construction of the one-ton-per-day PDU continued during the quarter with emphasis on the sections needed for coal liquefaction. By the end of the quarter, the feed systems were being readied for pressure testing. Progress was made on revising the high-pressure valves to withstand the high temperature climate. A valve with a 17-4 PH gland and a Haynes Alloy No. 25 stem demonstrated sufficient life in heated hydrogen.

PROJECT	FY 75	FY 76	TR. QR.	FY 77	FY 78	FY 79	FY 80
LABORATORY SUPPORT	2-5 lb./hr. BENCH SCALE UNIT						
PROCESS DEVELOPMENT UNIT	PROCUREMENT-DESIGN						
			CONSTRUCTION	SHAKEDOWN	OPERATION		

LEGEND : BEGIN MILESTONE
COMPLETE MILESTONE

Figure XIV-1. ZINC HALIDE HYDROPROCESSING PROJECT SCHEDULE

Figure XIV-2. ZINC HALIDE HYDROCRACKING PROCESS FLOWSHEET

Bench-Scale Hydrocracker Studies

The bench-scale hydrocracker was revised at the beginning of the quarter to permit operation with two reactors in series.

Material balance calculations with product yields were completed for four Colstrip coal hydrocracking single-

stage runs. The run at 120 minutes at 775°F reactor temperature and 3500 psig gave a conversion of 87 percent MAF coal, the highest conversion to -475°C material achieved to date with Colstrip coal. The C₅ x 200°C yield was 55 percent of the MAF coal feed. Correspondingly lower conversions and lower yields were obtained at the higher throughput rates.

Nine single-stage run attempts were made in August. Three successful runs with material balances were completed. These runs completed the original program on the variable study of Colstrip coal hydrocracking kinetics. Results indicated that conversions to -475°C product of over 80 weight percent MAF coal could be achieved with very little MEK-soluble and MEK-insoluble material remaining behind in the spent catalyst. The lowest observed yield of MEK-soluble oil remaining in the product catalyst was five percent, and the lowest yield of MEK-insoluble, three weight percent.

An unusually large number of mechanical problems plagued the bench-scale unit in September. In addition to run interruptions in both pumping systems, leaks developed when the two reactors were connected in series. The

pipng design was changed to correct the leaks. Preliminary run results indicated that the benefits of higher pressure increase as temperature is increased.

Supporting Laboratory Studies

Supporting laboratory work tested conditions for secondary recovery of zinc chloride. A design and material balance for an economic study of secondary zinc recovery was completed.

Batch autoclave work showed that Amax subbituminous coal has good reactivity in zinc chloride hydrocracking, being about as reactive as Colstrip coal.

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XV. 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 ERDA, 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 ERDA 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 XV-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 ERDA-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 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

Figure XV-1. FLUID COKING PROGRAM SCHEDULE

commercial fluid coker by means of a mechanical agitator. A schematic is shown in Figure XV-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 the year. Bench-scale studies in the one-gram unit on coking yields for the H-Coal sample were completed and tests began in November on the Continuous Stirred Coking Unit (CSCU).

Figure XV-2. FLUID COKING PROCESS SCHEMATIC

PROGRESS DURING JULY-SEPTEMBER 1977

The Continuous Stirred Coking Unit (CSCU) process variable scoping study of stripped SRC Antisolvent Deashing Underflow began in July and was completed by the end of the quarter. The CSCU yields agreed with the one gram per test bench coking unit to 1100° F. At 1200° F,

30 percent steam, low residence time, the liquid yields were significantly higher than predicted by the bench scale unit.

Two of the runs completed in September were of extended duration to generate coker liquid product for a 1000° F+ product recycle simulation run. Liquid product analyses were underway for samples from all the completed operations.

XVI. UPGRADING OF COAL LIQUIDS

UOP, INC.

DES PLAINES, ILLINOIS

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

Contract Funding: \$997,782

INTRODUCTION

UOP, Inc. is under contract to ERDA 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 ERDA 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 JULY-SEPTEMBER 1977

Summary

First stage hydrocracking process variable studies on H-Coal gas oil were concluded during the quarter. Second stage hydrocracking of 600°F+ H-Coal hydrocrackate to fuel oil began during September. Hydrotreating studies also began on three catalysts. Platforming runs on H-Coal process liquids were completed.

Bench-Scale Operations

First-stage hydrocracking process variable studies on H-Coal gas oil were concluded in July. At base pressure, data were obtained at one temperature and three space velocities. At 500 psig below base pressure, data were obtained at one temperature and one space velocity.

The stripper bottoms from the preparative first-stage H-Coal gas oil hydrocracking run were blended to provide one drum of charged stock for second-stage hydrocracking to gasoline. All required analyses of this stock were completed in July. Five runs were completed during the quarter.

A second first-stage hydrocracking preparative run was started in July also. The object of the run was to produce a 600°F+ stock with a nitrogen content of less than 10 weight-ppm and a 400°F+ with less than 500 weight-ppm nitrogen. The first stock was to be used as

feed for second-stage hydrocracking to maximum fluid oil. The second stock will be a feed for a series-flow second-stage hydrocracking to maximum gasoline.

The attempt to hydrocrack the rerun H-Coal atmospheric bottoms to fuel oil in a single-stage recycle operation was not successful. Even at hydrocracking conditions considered to be relatively severe, conversion to 600°F- distillate was only near 90 weight percent.

Second-Stage hydrocracking of 600°F+ H-Coal hydrocrackate to fuel oil began during September and was in progress at the end of the quarter.

Rerun H-Coal atmospheric still bottoms were used to evaluate the relative hydrogenation activity of three commercial UOP catalyst. For FCC studies, the hydrotreating catalyst is to be selected on the basis of its ability to saturate aromatic rings, rather than to remove nitrogen. Hydrotreating studies began in September for three catalysts.

Exxon Donor Solvent (EDS) process naphtha was successfully hydrotreated to reduce nitrogen to less than one part per million in a preparatory run to catalytic reforming. Platforming runs were also carried out with two H-Coal hydrocrackate naphthas at identical pressure and space velocity. The first hydrocracked naphtha was a blend of products from the second-stage hydrocracking of H-Coal gas oil to gasoline studies conducted at 500 psig below base pressure. The second hydrocracked naphtha was a blend of product from studies conducted at 500 psig above base pressure. This completed the platforming runs on H-Coal process liquids.

XVII. GASIFICATION OF RESIDUAL COAL LIQUEFACTION PRODUCTS

TEXACO, INC.

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

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

INTRODUCTION

Under this agreement, Texaco, Inc. will perform the research concerning the gasification of residual material from coal liquefaction samples supplied by ERDA. 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 ERDA 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 the ERDA-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 JULY-SEPTEMBER 1977

During the quarter, laboratory evaluations were performed on a sample from the SRC II coal liquefaction plant in Tacoma (See Section I) and a sample from the Exxon Donor Solvent coal liquefaction process (See Section IV).

Both samples were judged to be suitable feedstocks for the Texaco Coal Gasification Process. They may be charged to a Texaco gasifier as a molten fluid at 700° F. Both samples were too viscous to be pumped with the pilot plant equipment available. Consequently, they would require the addition of a cutter to reduce viscosity for pilot plant testing.

The SRC II vacuum drum bottoms sample was obtained from the liquefaction of Kentucky No. 9 and

14 coal. The Exxon vacuum tower bottoms sample was obtained from the liquefaction of Illinois No. 6 coal.

XVIII. 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 XVIII-1.

PROCESS DESCRIPTION

The Lummus Clean Fuels From Coal (CFFC) Process is based on direct catalytic hydroliquefaction in a multistage, plug-flow ebullated-bed reactor. A schematic of the process is presented in Figure XVIII-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.

PROJECT	FY 77	FY 78	FY 79
30 LB/HR. CONTINUOUS TEST UNIT MODIFICATION	△		
OPERATION	△	△	△
PROJECT EVALUATION			○

LEGEND: △ BEGIN MILESTONE
▽ COMPLETE MILESTONE
○ DECISION MILESTONE

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

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.

Figure XVIII-2. CLEAN FUELS FROM COAL PROCESS SCHEMATIC

The program was initially submitted to ERDA 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 JULY-SEPTEMBER 1977

Prehydrogenation of the start-up inventory of paste oil was completed in July. Thirteen hundred gallons of paste oil were prepared to satisfy the initial inventory requirements until the integrated operation begins functioning on its recycle paste solvent. Catalyst used was American Cyanamid 1443B catalyst, a nickel/molybdenum catalyst in the form of 1/32" extrudates.

Shakedown and calibration operations on the integrated downstream units, including the de-asher, the

vacuum flash units, and the continuous distillation unit were concluded.

During the hydroliquefaction unit start-up in August, 400 gallons of prehydrogenated solvent were prepared for use in another ERDA contract. The work on the demonstration of solvent self-sufficiency was then initiated. After a short operating period, high differential pressures across the first reactor required a shutdown. Additional heater capacity was added, and the hydroliquefaction unit was operating satisfactorily at the end of August.

Interruptions to the operation of the hydroliquefaction reactors in September were encountered due to catalyst blocking of the top screens in the reactors. It was established that the hydrodynamics of the 1/32" extrudate catalyst was limiting throughput. As a step to improve both operability and throughput, 10 to 14 mesh nickel/molybdenum spherical catalyst was substituted.

Operations of the downstream continuous units on the hydroliquefaction unit first-cycle product were successfully conducted during the quarter.

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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.

dissolution—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—an assemblage of small solid particles maintained in 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-stream 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.