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U.S. DEPARTMENT OF ENERGY
Division of Coal Conversion

QUARTERLY REPORT

July-September 1978
DOE/ET-0068/3
Publication Date: May 1979

COAL

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COAL LIQUEFACTION

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Washington, D.C. 20545

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FOREWORD

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EXECUTIVE SUMMARY

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

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

To provide efficient and practical means of utilizing coal resources, DOE is supporting the development of several conversion processes that are currently in the pilot plant stage. The development of the solvent refined coal process is being carried out in pilot plants at Fort Lewis, Washington and Wilsonville, Alabama. Responsibility for the operation of the *Solvent-Refined Coal (SRC) Process* pilot plant at Ft. Lewis, Washington is assigned to Pittsburg and Midway Coal Mining Company. The plant was converted from the SRC-I mode to the SRC-II mode of operation during this quarter and operated on Kentucky coals in the SRC-II mode during the later part of the quarter.

The Merriam (Kansas) Laboratory conducted a series of runs exploring the effects of short residence time. The bench-scale equipment at Merriam was upgraded with the addition of a new, one-liter upflow reactor.

DOE, together with the Electric Power Research Institute, has contracted with Southern Services Company, Inc. for the *Operation of a Solvent-Refined Coal Pilot Plant* at Wilsonville, Alabama. Most of the operational time this quarter utilized Indiana V coal from the Old Ben #1 Mine. The newly-installed Kerr-McGee Critical Solvent Deashing Unit was placed in operation, and a U.S. Filter was installed for future testing.

Construction of the *H-Coal Project* pilot plant was 68 percent complete at the end of June. In July, responsibility for construction of the 600 ton-per-day pilot plant was transferred from Ashland Synthetic Fuels Inc. to Badger Plants Inc. Plant construction is somewhat behind schedule.

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. A residual bottoms recycle study was initiated and completed during this quarter, utilizing the 50 pound-per-day Recycle Coal Liquefaction Unit. The one ton-per-day Coal Liquefaction Pilot Plant operated on Wyoming coal. Operations in the two barrel-per-day Integrated Coking/Gasification Pilot Plant were completed for 1978. Engineering and procurement work on the 250 ton-per-day coal liquefaction pilot plant was 65 percent complete at the end of this quarter.

Also included in the coal liquefaction program are support research and development activities that provide backup for advanced liquefaction processes and improvements in liquefaction technology in general. Under this category of studies, Cities Service Research and Development Company is studying *Commercial Scale Expanded-bed Hydroprocessing of Solvent Refined Extract*. Current studies under this contract are being done by C. E. Lummus, a subcontractor, who is determining the conditions required to hydrotreat SRC extract at levels ranging from desulfurizing to extensive hydrocracking.

Fluor Engineers and Constructors, Inc. is responsible for the reactivation of the DOE-owned *Cresap Test Facility* in Cresap, West Virginia. Three runs were made during this quarter. Two of the runs were terminated by pump failures involving ruptures which released fluids. Two separate fires of modest size resulted. Pump modifications and fire damage repair should be completed by mid-November.

The *Clean Coke Process* is under development by USS Engineers and Consultants, Inc., a subsidiary of United States Steel Corporation. The objectives are to convert low-grade, high-sulfur coal to low-sulfur, low-ash metallurgical coke, chemical feedstocks, and liquid and gaseous fuels. The Clean Coke Process PDU program was completed this quarter and preparation of the project's final report is in progress.

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 technique involves rapid mixing, short reaction time at high temperature, and quick quenching to prevent liquids from being hydrogenated further to gases. During this quarter, the design of the benzene-toluene-xylene (BTX) collection system was completed. Construction of the high pressure liquid produce recovery system continues.

Studies were being conducted by the Occidental Research Corporation to evaluate the commercial potential of the company's *Flash Pyrolysis Coal Liquefaction Process*. Process development unit operations were halted in June to close out the contract. A final report is being prepared.

Research to improve solid-liquid separation is being conducted by John-Manville Sales Corporation in their *Filtration Process and Equipment Studies*. Rotary drum pressurized precoat filtration is being studied. The project, initiated in 1975, drew to a close this quarter. Work on a final report has commenced.

Oak Ridge National Laboratory is providing *R&D Engineering Services* dealing with *Hydrocarbonization of Coal*. During this quarter, small-scale equipment was modified to prepare it for use in investigations of rapid hydropyrolysis. Contract work on this project is coming to a close. Future work will focus on preparation of a final report.

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 coals or coal extract. During this quarter, break-in testing of portions of the one ton-per-day PDU commenced. Writing continues on a final report of bench-scale work. A supporting laboratory study continues for the purpose of defining the effects of ash metal chlorides on hydrocracking activity of zinc chloride.

Exxon Research and Engineering is studying the potential for *Fluid Coking of Coal Liquefaction Residues* to recover distillate liquids. During this quarter, compilation and interpretation of data are in progress. The project terminates on December 31. A final report is being prepared.

To facilitate the upgrading of primary coal liquids using technology in the petroleum industry, UOP, Inc. is conducting a study of *Upgrading Coal Liquids*. Three series flow hydrocracking tests were run this quarter. All hydrocracking runs on Exxon Donor Solvent derived feedstocks have been completed. Fluid Catalytic Cracking studies of EDS derived feedstock are underway.

Texaco, Inc. is evaluating the feasibility 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 this quarter, a preliminary pilot plant evaluation of gasification of char obtained from short residence time liquefaction of Kentucky coal in the Rocketdyne high-mass flow pilot process development unit was completed.

The production of *Clean Industrial and Transportation Fuels from Coal* is being investigated by the Lummus Company using a multi-stage ebullated-bed catalytic reactor developed through the firm's activities with the bench-scale pilot plant. Test work covering catalyst life confirmation was initiated in April. During this quarter, the Process Development Unit was operated to obtain data on a number of process variables.

I. SOLVENT-REFINED COAL (SRC) PROCESS

THE PITTSBURGH & MIDWAY COAL MINING COMPANY

MERRIAM, KANSAS

Plant Site: Fort Lewis, Washington

Laboratory: Merriam, Kansas

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

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

SOUTHERN COMPANY SERVICES, INC.

BIRMINGHAM, ALABAMA

Plant Site: Wilsonville, Alabama

Contract No.: EX-76-C-01-2270

Contract Funding: \$8,729,892
DOE: \$5,741,892
Industry: \$2,988,000

INTRODUCTION

The SRC pilot plant at Ft. Lewis is being operated by Pittsburgh & Midway Coal Mining Company (P&M), a subsidiary of Gulf Oil Corporation, under the sponsorship of DOE. The specific objectives of contract EX-76-C-01-496 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-product from the process for market development studies. Figure I-1 provides a schedule for the development of the project.

The operation of the SRC pilot plant by Southern Company Services, Inc. at Wilsonville is jointly sponsored by DOE and the Electric Power Research Institute (EPRI). The schedule for the project is shown in Figure I-2. The objectives of contract EX-76-C-01-2270 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-I (solid fuel) process converts high-sulfur, high-ash coals to a low sulfur, low ash solid fuel. A schematic of the process is shown in Figure I-3. The pulverized coal is mixed with process-derived solvent in a slurry mix tank. The slurry is combined with

LEGEND: BEGIN MILESTONE
COMPLETE MILESTONE

Figure I-1. FT. LEWIS SRC PILOT PLANT PROGRAM SCHEDULE

LEGEND: BEGIN MILESTONE
COMPLETE MILESTONE

Figure I-2. WILSONVILLE SRC PILOT PLANT OPERATION SCHEDULE

Figure I-3. SRC-I PROCESS SCHEMATIC

hydrogen and is then pumped through a fired preheater and into a dissolver which operates at 800°F-875°F, and a total pressure of approximately 1,500-2,000 psig. Under these conditions, approximately 93 percent of the carbonaceous material in the coal feed is dissolved during the residence time of approximately 30 minutes. In the dissolver, 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, yielding lower molecular weight hydrocarbons ranging from light oil to methane. 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 hydrogen recovery and gas desulfurization units. The recovered hydrogen is then recycled and combined with the slurry from the slurry mix tank. Hydrocarbon gases

are recovered and the hydrogen sulfide is converted to elemental sulfur.

The slurry of undissolved solids and coal solution is then treated in a solids separation unit. In a commercial-scale plant, the separated 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 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.

The Fort Lewis pilot plant has been modified to permit recycle of the reactor effluent slurry for blending with the coal feed instead of distillate solvent (SRC-II). By increasing severity of operating conditions, the major product is converted from a solid to a liquid distillate fuel with a 350°F to 850°F boiling range.

The general process is the same for both modes from the slurry mix tank through the dissolver. In the SRC II case, however, the dissolver slurry is split, one portion is recycled to provide solvent for coal slurry mixing, while the other is fractionated. The products from the fractionation system are naphtha, low-sulfur fuel oil, and a vacuum residue consisting of heavy oil, ash, and undissolved organic material from the coal.

HISTORY OF THE PROJECT

The SRC project was begun in 1962 when Spencer Chemical Company was awarded a research contract by the Office of Coal Research (OCR, subsequently a part of ERDA, now DOE) to study the technical feasibility of a coal de-ashing process (now called the SRC process). In 1965, the process was successfully demonstrated in a 50-pound-per-hour continuous-flow unit, and the work on the contract was therefore completed. During the term of the contract, Gulf Oil Corporation acquired Spencer Chemical Company. After reorganization, the contract was assigned to the research department of P&M.

To develop the SRC process further, a contract was awarded to P&M to design, construct, and operate a pilot plant that could 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.

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 at Wilsonville, Alabama. The facility began operation in January 1974. The Electric Power Research Institute assumed the responsibilities of the Edison Electric Institute for utility industry sponsorship in April 1973.

Test runs during 1975 at Ft. Lewis were designed to study the effects of different coal feed rates and dissolver temperatures. A 3,000-ton run was conducted during 1975 and 1976 to produce specification SRC for use in combustion testing by Georgia Power Company. After modification of filtration instrumentation following the production run, filtration tests were conducted. The plant was then shut down in January 1977 for a major maintenance turnaround and SRC II (slurry recycle)

pipings tie-in. Modifications were completed in May and testing began with a blend of Kentucky No. 9 and 14 coal. Operability and yields were explored with emphasis on conditions yielding organic residues compatible with hydrogen generation needs. Testing with Illinois No. 6 coal began in September, and Pittsburgh seam coal was used for testing in November and December. SRC-II mode operations on Illinois coal and on Pittsburgh seam coal continued through the first quarter of 1978. Following a plant turnaround, the Ft. Lewis pilot plant resumed operations on June 10 in the SRC-I mode.

The laboratory unit at Merriam, Kansas, has been run in both the distillate solvent mode and SRC II mode in support of Ft. Lewis pilot plant operations. The variables investigated included solvent recycle rate, slurry residence time, concentration of coal in the slurry, and temperature. Tests were also run to simulate operating conditions at the pilot plant. The effect of process variables in SRC-I and II operations was investigated.

The six ton-per-day Wilsonville SRC pilot plant began operation in January 1974. During 1976, 1977, and 1978, the following have been processed: Kentucky 9 and 14, Monterey (Illinois No. 6), Amax Belle Ayr, Emery coal from Consolidation's Emery Coal Mine in Utah, and Indiana V from the Old Ben No. 1 Mine.

PROGRESS DURING JULY-SEPTEMBER 1978

Ft. Lewis Pilot Plant

Summary

The Ft. Lewis Pilot Plant, which had started up on June 10, operated until August 23 in the SRC-I mode. It was then shut down to prepare for SRC-II mode operations. During this third quarter, Kentucky coals No. 9 and 14 and Kentucky coal from the P&M Colonial Mine were processed successfully.

Pilot Plant Operation

Except for minor problems in the coal preparation section and in the solvent fractionation system, the pilot plant operated dependably. Filtration tests on Filter C, which is of the precoat type, were unsatisfactory. These were the first filtration tests on coal solutions using Filter C. Mechanical problems, particularly with screen supports, resulted in screen movement and tearing and led to loss of filtrate clarity and to erratic operation. All activities on Filter C were suspended, and major design changes to the filter are anticipated.

Construction work on the Lummus antisolvent deashing system unit neared completion during this quarter. Systems testing of this unit will begin once the current SRC-II test program has been completed and the plant has been re-converted to the SRC-I mode.

Laboratory - Merriam, Kansas

A series of runs exploring SRC-I operations at short slurry residence times was conducted, and this exploratory work program will continue.

A new one-litre upflow reactor was built for the upgraded bench-scale coal liquefaction system. One SRC-I and three SRC-II runs were completed with the new reactor this quarter.

A short series of trials was made to evaluate the effect on recycle solvent of processing without coal or mineral matter and with the addition of pyrite or mineral residue. A 455°C (850°F) mild cracking together with slight desulfurization and slight hydrogenation of the solvent were observed with and without additives. No significant additive effect was apparent. At a lower temperature, 400°C (752°F) no light hydrocarbon gas production was detected, and the elemental composition of a product solvent was essentially that of the feed solvent.

Wilsonville Pilot Plant

Summary

Most of the operational time was used to process Indiana V coal from the Old Ben No. 1 Mine. The Kerr-McGee Critical Solvent Deashing Unit was operated most of the quarter, and a U.S. Filter was installed for

future testing. Late in September a shift was made to Kentucky No. 9 coal.

Pilot Plant Operations

The six ton-per-day pilot plant operated 595 hours in July, 650 hours in August, and 275 hours in September. During pilot plant runs 140 and 147, 48-hour material balances were obtained. Conditions during the material balance runs were similar, except that in Run 147 a reduced inventory of solids was maintained in the dissolver. The properties of the ash-containing SRC feeds for the material balance runs were considerably different, however, in terms of critical solvent deashing parameters.

The pilot plant was shut down from September 9-23 to facilitate installation of the U.S. Filter Unit and to tie in the third stage solvent return system of the Kerr-McGee Critical Solvent Deashing Unit. Coal feeding to the pilot plant was resumed on September 23. The last of the Indiana V coal was consumed on September 27 and a switch to Kentucky No. 9 coal was made to start run 148. Conditions for Run 148 were: 450 pounds coal feed per hour, 38.5 percent coal concentration, 10,000 SCFH of gas at 85 percent purity, 1,700 psig dissolver pressure and 825°F dissolver outlet temperature. The SRC produced at these conditions was difficult to deash on the Kerr McGee Critical Solvent Deashing Unit. To produce a heavier SRC, the dissolver temperature was lowered to 810°F (in Run 149) on September 29. The lower temperature improved deashing on the Kerr-McGee CSD Unit.

Plans call for an extended run of 20 to 30 days with the Kerr-McGee CSD Unit in an attempt to maximize SRC recovery. After completion of this extended run, testing of the U.S. Filter Unit will begin.

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II. H-COAL PROJECT

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

HYDROCARBON RESEARCH, INC.
TRENTON, NEW JERSEY

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

ASHLAND SYNTHETIC FUELS, INC.
ASHLAND, KENTUCKY

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

BADGER PLANTS, INC.
CAMBRIDGE, MASS.

Contract No.: ET-78-C-01-3224

INTRODUCTION

The H-Coal process is being developed by Hydrocarbon Research, Inc. (HRI) under the joint sponsorship of (1) DOE, (2) a private industry consortium composed of Electric Power Research Institute, Ashland Synthetic Fuels, Inc. (ASFI), 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 and construct a pilot plant capable of converting 600 tons of coal per day to low-sulfur boiler fuel or 200 tons of coal per day to synthetic crude oil.

The pilot plant will be 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

HRI, developer of the H-Coal process, designed the H-Coal Pilot plant under Phase I of the H-Coal Project.

Figure II-1. H-COAL PROGRAM SCHEDULE

Badger Plants, Incorporated, Cambridge, Massachusetts is construction manager and constructor of the pilot plant under Phase II. ASFI is responsible for operation of the H-Coal Pilot Plant under Phase III of the H-Coal Project. HRI will support construction and operation under Phases II and III. The Lummus Company is responsible for the design of the antisolvent deashing unit for the pilot plant.

PROCESS DESCRIPTION

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

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

The gas and liquid products, composed of hydrocarbon gas, hydrogen sulfide, ammonia, light distillate, heavy distillate, and residual fuel, may be further refined as necessary. A portion of the heavy distillate is recycled as the slurry medium. The stream containing the unreacted carbon and some liquid will eventually be processed in a commercial installation to produce additional hydrogen needed for the process.

The specific operating conditions of the H-Coal process affect the type of fuel produced. For example, to produce synthetic crude, more hydrogen is required and

Figure II-2. H-COAL PROCESS SCHEMATIC

there is a lower yield of residual fuel oil. In this mode, the separation of the solids (unconverted carbon and ash) from the liquid can be accomplished by vacuum distillation, and a special liquid-solid separation unit is not required. To produce clean fuel and low-sulfur residual oil as major products, the temperature and pressure in the ebullated-bed reactor are lowered, and less hydrogen is required.

The H-Coal process requires between 14,000 and 20,000 standard cubic feet of hydrogen for each ton of coal processed, depending on the type of oil produced. In the pilot plant, some of the hydrogen required has been obtained from the gas produced by the ebullated-bed reactor. Additional hydrogen has been purchased

to meet processing demands. In a commercial operation, hydrogen demand will be met by manufacture on site. Because the H-Coal process converts about 90 percent of the carbon contained in the coal to a liquid, the feed to the hydrogen plant could be liquid rather than solid. This suggests that commercial hydrogen manufacturing processes could be adapted. The solid cake char could also be used to produce hydrogen.

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

HISTORY OF THE PROJECT

The H-Coal process was developed by HRI as a further application of the ebullated-bed processing technology originally used to convert heavy oil residues from petroleum into lighter fractions (H-Oil process). The H-Coal Project, supported jointly by government and industry, is a three-phase project involving the design, construction, and operation of a 600 ton-per-day coal liquefaction pilot plant at Catlettsburg, Kentucky. Early development of the H-Coal process, beginning in 1964, involved research with a bench-scale unit and a process development unit and preparation of a conceptual process design. An independent evaluation in 1968 confirmed the technical and economic feasibility of the H-Coal process.

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 sub-bituminous 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 rear-

rangment 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.

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

In 1977, construction of the H-Coal Pilot Plant continued, and was 30 percent complete at the end of the year. HRI met the mid-December target date for the Phase I engineering effort with 99.6 percent of the total drawings certified for construction. Phase II PDU modification to simulate syncrude operations on the pilot plant were completed in July, and two 30-day runs were successfully completed during 1977. In July 1978, responsibility for construction of the 600 ton-per-day pilot plant was transferred from ASFI to Badger Plants, Inc.

PROGRESS DURING JULY-SEPTEMBER 1978

HRI, Inc. — Contractor, Laboratory Support of Design and Construction

HRI, Inc. continued to provide laboratory support of the design and operation of the H-Coal Pilot Plant. Using bench-scale equipment, Kentucky No. 11 coal was evaluated for comparison with Illinois 6 coal. The two coals were quite similar in yields of distillates and residual oils. PDU Run No. 7, a boiler fuel operation to test emergency procedures and process limitations, was completed during this quarter.

ASFI — Contractors, Construction Management

The month of July terminated ASFI's contractual obligations for construction management of the H-Coal Pilot Plant. The transfer of responsibility from ASFI to DOE became effective July 12, 1978.

The weighted average of all contracts for design, procurement, and erection of the H-Coal Pilot Plant (Phase I, and Phase II, construction), was 70.2 percent complete in July. Erection of the pilot plant at the end of September was 50 percent complete.

Badger Plants, Inc. — Prime Contractor to DOE

Efforts to alleviate the shortage of pipefitters, a major constraint on construction, continued during the period. The overall construction program has slipped behind original schedules. Following is the newly projected

schedule for mechanical completion dates, based on the current construction schedule:

100 Unit - Coal Preparation	- March 31, 1979
200 Unit - Hydrogenation	- September 30, 1979
400 Unit - Offsites	- April 30, 1979
500 Unit - Deashing	- November 30, 1979
600 Unit - Waste Oil	- September 30, 1979

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III. DONOR SOLVENT LIQUEFACTION PROCESS

EXXON RESEARCH AND ENGINEERING
COMPANY

FLORHAM PARK, NEW JERSEY

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

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

INTRODUCTION

The Exxon Donor Solvent (EDS) coal liquefaction process produces distillate products from a wide range of coals. The development of the process represents a major contribution to the emerging coal liquefaction technology and is based on laboratory and engineering research and development efforts integrated with the operation of a 250 ton-per-day coal liquefaction pilot plant. The program features development of technolo-

gies for coal liquefaction, solids/liquid separation, hydrogen and process fuel generation, and for extensive product testing. Pursuance of these efforts will provide the basis for a process that is operable and reliable and with an excellent chance for commercialization. The project schedule is shown in Figure III-1.

The overall project objective is to develop the EDS process to the state of commercial readiness by 1982 by obtaining all of the data needed for a commercial

LEGEND: △ BEGIN MILESTONE
▽ COMPLETE MILESTONE

Figure III-1. DONOR SOLVENT PROJECT SCHEDULE.

plant design. This will be accomplished through the following more specific objectives:

- Develop the EDS process and design bases necessary to liquefy a range of different coals in an environmentally acceptable manner.
- Develop process correlations relating product yields and qualities to operating conditions and feed coals.
- Translate program results into estimates of commercial plant economics and operations.
- Identify and implement modifications to improve process economics and operability.
- Develop and demonstrate process technology at the scale necessary to insure a reliable commercial design.
- Conduct an aggressive product testing program to insure marketable products.

A cooperative agreement to accomplish these objectives was signed between industry and the DOE in 1977. Funding participants for industry are: Exxon affiliate, Carter Oil Company, \$59,090,909; Electric Power Research Institute, \$30,909,091; Japan Coal Liquefaction Development Kabushiki Kaisha, \$20,000,000; Phillips Petroleum, \$5,000,000; and Atlantic Richfield, \$5,000,000.

PROCESS DESCRIPTION

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

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

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

Figure III-2. DONOR SOLVENT LIQUEFACTION PROCESS SCHEMATIC

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, produce distribution may be varied over a wide range.

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

HISTORY OF THE PROJECT

Phase I of EDS Process Development (1966-1973)

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

The following reactor configurations were evaluated: stirred bed, tubular reactor (with external and internal solids recirculation), ebullating bed, and tubular reactor (plug flow). Both hydrogenated and unhydrogenated recycle solvents were evaluated for bituminous and subbituminous coals. Several solids/liquids separation techniques were studied, such as distillation, centrifugation,

filtration and hydroclones. Wide ranges of temperatures, pressures and residence times were evaluated in both the liquefaction and solvent hydrotreating reactors. Based on these results, a hydrogenated recycle solvent process was selected for further development, and vacuum distillation was identified as the most promising product-separation technique. A noncatalytic, tubular plug-flow liquefaction reactor was selected. These choices circumvented the need for ash-tolerant catalysts and mechanical separation devices.

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

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

Phase II EDS Process Development (1974-1975)

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

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

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

Phase III-A	January 1, 1976 - June 30, 1977 Contract E(49-18)2353
Phase III-B	January 1, 1977 - December 31, 1982 Contract EF-77-A-01-2893
Phase IV	January 1, 1977 - December 31, 1982 Contract EF-77-A-01-2893

Phase IV Started January 1, 1978, but is reported as the 6th monthly progress report as a continuation of Phase III-B

PROGRESS DURING JULY-SEPTEMBER 1978

Summary

A residual bottoms recycle study was initiated and completed during this quarter, utilizing the 50 pound-per-day Recycle Coal Liquefaction Unit (RCLU-1). The vacuum bottoms had been produced in the one ton-per-day Coal Liquefaction Pilot Plant (CLPP) the previous quarter. Variables investigated were coal-to-bottoms ratio and slurry residence time.

The CLPP operated on Wyoming coal throughout this quarter. This operation is a continuation of a study to define preferred operating conditions.

Catalyst activity studies continued, using Ni/Mo-10 and Co/Mo-20 catalysts on Wyoming spent solvent.

Engineering and procurement work on the 250 ton-per-day Exxon Coal Liquefaction Pilot Plant (ECLP) was 65 percent complete at the end of the quarter. Construction of the ECLP was 16 percent complete at the end of the quarter.

Operations in the two barrel-per-day Integrated Coking/Gasification Pilot Plant (IKG) on EDS coal liquefaction vacuum bottoms was completed for 1978. Post-operations work centered on data analysis. Preliminary calculations show 75 percent gross coke carbon gasification was achieved during Wyoming coal bottoms operations, compared with 80 percent gasification attained during Illinois coal bottoms operations.

Recycle Coal Liquefaction Unit (RCLU-1)

The residual bottoms recycle study conducted with the Recycle Coal Liquefaction Unit was for the purpose of evaluating the effect of bottoms recycle on Exxon Donor Solvent (EDS) liquefaction yields for Illinois

No. 6 coal. The recycle study concluded with a run in which once-through bottoms were recycled a second time through the liquefaction section. No operational problems were encountered. The recycle of one-through bottoms appears to give lower conversion than recycle of the original CLPP bottoms. Toward the end of the quarter, a screening study with Illinois No. 6 bituminous coal from the Burning Star No. 2 Mine was initiated in the RCLU-1. The process variables investigated were liquefaction temperatures (800°/840° F) and slurry residence time (40/100 minutes). Burning Star coal conversion and liquid yields were significantly lower than for Illinois No. 6 coal from the Monterey No. 1 Mine at the four conditions investigated.

Coal Liquefaction Pilot Plant (CLPP)

Continuous runs of 12½ and 10 days were completed in the CLPP. A residual bottoms viscosity problem was overcome by increasing solvent boiling range. Good service was obtained on key pieces of equipment.

Supporting Studies

- The fundamental solvent hydrogenation simulator was employed to carry out a simulated process variable study for Wyoming coal MPSS and Ni/Mo-10 catalyst at initial activity. Temperatures and space velocities were varied over a wide range of operating conditions.
- A scoping study using methanol/water mixtures as a solvent to extract phenolics from EDS naphtha revealed higher recoveries at higher methanol contents but lower purity. Additional work is planned with a light paraffin as a dual solvent.
- A study is in progress to determine the feasibility of hot storage of the vacuum tower bottoms from the Exxon Coal Liquefaction Pilot Plant (ECLP). A desirable design basis is that the viscosity at 500° F be less than 25 poise after 30 days storage.
- Cold model studies of the Fluidized Bed Unit (FBU) coke gasification run at operating conditions similar to the nominal conditions that formed slag in the Integrated Coking/Gasification Pilot Plant (IKG). This was the first instance of slag formation in the FBU with stable temperature control. The apparent key variable in this slagging demonstration was the reduction in the superficial velocity.

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

CITIES SERVICE RESEARCH AND
DEVELOPMENT COMPANY

TULSA, OKLAHOMA

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

Contract Funding: \$1,982,000

INTRODUCTION

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

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

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

PROCESS DESCRIPTION

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

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

Figure IV-1. EXPANDED-BED HYDROPROCESSING PROCESS SCHEMATIC

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 SRC extract began on June 1, 1976 with benchscale pilot plant operations. The primary contract was extended for an additional 18 months, commencing June 1, 1977 under contract EX-76-C-01-2038. An Interim Technical Progress Report was published in November 1977 and is available from NTIS, Springfield, Virginia.

PROGRESS DURING JULY-SEPTEMBER 1978

Summary

Reporting of accumulated and revised data from a series of PDU runs comprise the monthly technical progress reports (MPR) for the July/September quarter of 1978. The purposes of the various PDU runs for which data are reported, were:

PDU Run 2 LCF-3	Recycle Run - SRC-I
2 LCF-5	100% 500°F IBP Solvent
2 LCF-6	70/30 Volume % SRC-I/ 500°F Solvent
2 LCF-7	50/50 Volume % SRC-I/ 680°F Solvent
2 LCF-8	50/50 Volume % Amax SRC/500°F Solvent
2 LCF-9	100% Volume % 680°F IBP Solvent

The next several monthly technical reports will be only compilations of data tables. It is expected that by February 1979, the MPR's will be current.

Discussion of Activities

At a reactor operating temperature of 810°F, both cobalt-molybdenum (Co/Mo) and nickel-molybdenum (Ni/Mo) catalysts show little loss in conversion of 850°F+ over extended periods of operation.

At a temperature of 780°F, the Ni/Mo catalysts previously showed a conversion decline from 58.4 to 48.1 volume percent 850°F+ over 30 periods of operation.

A run was made processing a 70/30 volume percent SRC-I/solvent feed blend to investigate the behavior of higher SRC ratios. At a constant temperature, this run gave the same conversion of 850°F+ as the run processing the normal 50/50 ration.

A PDU run was made to investigate the operability and performance of a solvent refined coal obtained from the Belle Ayr (Amax) mine in Wyoming. As previously reported, it was substantiated that the Amax SRC had a lower conversion of 850°F+ than SRC-I at the same operating conditions.

A detailed data analysis is in progress and will be documented in future reports.

Work Forecast

A revised statement of work for the Expanded Bed Hydroprocessing of Coal Extracts is being prepared. Future work will encompass some or all of the following:

- Repeat run 50/50 SRC/680°F IBP KC-Oil;
- 30-day run with cool non-catalytic process zones;
- A catalyst check run on another batch of modified Shell 324 Ni/Mo catalyst;
- Deashed low severity extract (LSE) from Wilsonville (once-through)
- Non-deashed low severity extract (LSE) from Wilsonville (once-through)
- Effect of higher hydrogen pressure and increased space velocity;
- Recycle operation on deashed low severity extract (LSE).

This technical information obtained from this work will be in support of a proposed DOE sponsored two-stage liquefaction program.

V. CRESAP TEST FACILITY

FLUOR ENGINEERS AND CONSTRUCTORS,
INC.

LOS ANGELES, CALIFORNIA

Project Site: Cresap, West Virginia

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

Contract Funding: \$66,358,545

DOE: \$63,445,545

Industry: \$ 2,913,000

INTRODUCTION

Under contract to DOE, Fluor Engineers and Constructors, Inc., has renovated and is operating a DOE-owned facility in Cresap, West Virginia. Industrial financing is being provided by American Electric Power Corporation (\$1 million), Allegheny Power Corporation (\$1 million), and Fluor (\$913,000). This facility, inactive since 1970, contains much of the equipment required for coal liquefaction. Fluor (1) has renovated the Cresap facility to test the various critical mechanical com-

ponents required for coal liquefaction and (2) is operating the renovated facility to demonstrate an integrated process for the production of low-sulfur liquid fuel. The project schedule is Figure V-1.

PROCESS DESCRIPTION

In this process, shown schematically in Figure V-2, coal is converted to low-sulfur liquid fuel. The coal is first crushed to minus 100-mesh size and is then slurried with aromatic solvent in a mixer. The coal-solvent

LEGEND: BEGIN MILESTONE DECISION MILESTONE 5 ORDER LONG-LEAD ITEMS
COMPLETE MILESTONE EVENT

Figure V-1. CRESAP TEST FACILITY PROGRAM SCHEDULE

Figure V-2. LIQUID FUEL FROM COAL PROCESS SCHEMATIC

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 most of the liquid coal extract and solvent are removed, leaving a thickened slurry. The thick slurry 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. Hydroclones were successfully used during prior pilot plant operations and the plant is now operating on solvent deashing.

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. The light distillates are combined and stored as naphtha to be used for product blending.

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

pipng, 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, flow diagrams were made, and specifications were prepared for the refurbishing of some of the existing equipment. The Liquified Coal Development corporation, a wholly-owned subsidiary of Fluor, was formed during 1975 to operate the pilot plant.

Construction activities continued through 1976 and were completed September 1977. 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 activities began by mid-August 1977.

Four extraction runs had been conducted by the end of 1977. During longest run, coal was fed for 144 hours, processing 32 tons of coal in an 11.5 percent slurry. Conversion averaged 62 percent, and more than 100 55-gallon drums of extract rich intermediate were produced.

After repairs and maintenance were completed at the beginning of 1978, the plant was run at a rate of 12 tons coal per day in a 25 percent slurry to obtain baseline data from the solvent deashing system. At the end of February, hydrogen was introduced into the hydrotreating portion of the pilot plant. Fully integrated solvent circulation was established between the hydrogenation and the fractionation portions of the pilot plant. Extract hydrogenation was accomplished at the initial

operating conditions selected, and the fuel oil product contained less than 0.7 weight percent sulfur.

PROGRESS DURING JULY-SEPTEMBER 1978

During July, pilot plant Runs 011 and 012 were made, but each was plagued with operational and mechanical problems. At the end of Run 012, while reactor temperatures were being reduced, a recycle pump internal liner rupture released solvent and hydrogen to the atmosphere. A resulting fire caused relatively minor damage to electrical equipment and to instrumentation in Section 600, the Extract Hydrogenation section. Operating problems during the two runs resulted from repeated valve and bearing failures in compressors and pump, from line pluggages and leaks, from instrumentation problems, and from equipment leaks.

During August, Run 013 was conducted; which, at 18 days duration proved to be the most successful to date. The run terminated when the reactor recycle pump pressure liner failed releasing fluids which ignited, again causing damage to the Extract Hydrogenation section. During run 013, the coal concentration in the slurry feed was 20 percent, which resulted in an 18.5 percent concentration in the extractor. The nominal coal feed was 900 pounds-per-hour. With the extractor temperature at 730° F, the extractor effluent samples indicated an extraction depth of 77 percent.

Major efforts in September were directed toward repairing the fire damage in the Extract Hydrogenation section. Modifications were made to the reactor recycle pumps to prevent the type of pump failure which caused the fires. Repairs should be completed by November 15.

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VI. CLEAN COKE PROCESS

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

Plant Site: Pittsburgh, Pennsylvania
Contract No.: EX-76-C-01-1220
Contract Funding: \$11,792,329
DOE: \$8,337,176
Industry: \$3,455,153

INTRODUCTION

The Clean Coke process is being developed by USS Engineers and Consultants, Inc., a subsidiary of United States Steel Corporation, under the sponsorship of DOE. The contract was initiated in 1972 under the auspices of the Office of Coal Research (OCR, first a part of ERDA, subsequently DOE). The objective of the project is to develop a process design that is capable of converting low-grade, high-sulfur coal to low-sulfur,

low-ash metallurgical coke, chemical feedstocks, and liquid and gaseous fuels. The pilot plant design will be based on the results of laboratory tests and data obtained from process development units. The schedule for developing the process is shown in Figure VI-1.

PROCESS DESCRIPTION

The Clean Coke process is shown schematically in Figure VI-2. This process combines coal carbonization

PROJECT	FY 74	FY 75	FY 76	TR QR	FY 77	FY 78	FY 79
PROCESS DEVELOPMENT UNIT INPUT: 1/2 TON/DAY OUTPUT : 1.5 BARRELS/DAY EVALUATE 2 OTHER COALS	OPERATION		EVALUATION			▽	
		△					
					△		▽

LEGEND:

- △ BEGIN MILESTONE
- ▽ COMPLETE MILESTONE

Figure VI-1. CLEAN COKE PROCESS SCHEDULE

Figure VI-2. CLEAN COKE PROCESS SCHEMATIC

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 hydrogen-rich gas which is subsequently cleaned and recycled to fluidize the bed. The hydrogen-rich atmosphere of the bed provides a hydrosulfurization 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.

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

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

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

Major advantages of the Clean Coke process design are that:

- Hydrogen is produced for the liquefaction portion.

- Carbonization char is used to produce metallurgical-grade coke from high-sulfur, high-ash coal.
- A highly desulfurized char is produced which could provide low sulfur combustible product.

HISTORY OF THE PROJECT

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

Under contract to the government since 1972, U.S. Steel is operating the process development units. Total carbonization PDU operating time completed by the end of 1977 was 4,560 hours. Testing of Illinois No. 6 seam coal and West Kentucky No. 9 seam coal was completed in 1977, and testing of Pittsburgh seam coal began. Testing of Pittsburgh seam coal was completed in May 1978. Yield of char from the coal was 61.4 percent. The study of treating requirements to convert SRC-II heavy oil into a binder pitch was completed in May. As the clean coke carbonization process development unit program is essentially completed, preparation of the final report was begun.

PROGRESS DURING JULY-SEPTEMBER 1978

As the Clean Coke process development unit (PDU) program was completed in June, work during the July-September quarter consisted of preparing the final report for the project.

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VII. LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

ROCKETDYNE DIVISION
ROCKWELL INTERNATIONAL CORPORATION
CANOGA PARK, CALIFORNIA

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

INTRODUCTION

Under contract to DOE, the Rocketdyne Division of Rockwell International is conducting a research program to develop a technology to liquefy coal via direct hydrogenation. This technique would involve rapid mixing, reaction, and quenching of a gaseous hydrogen and pulverized coal mixture to form hydrocarbon liquids. These products would be suitable for conversion to fuel oil or chemical feedstocks. The process is based on a concept that high liquid yields are favored when coal is allowed to react with hydrogen for a brief period at high temperature. Quenching prevents liquids from being hydrogenated further to gas.

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

Upon completion of Phase I, the results of the cold-flow tests were submitted to DOE for a decision on proceeding with Phase II. Upon Phase II authorization, small-scale reactor testing was initiated to evaluate the effects of reactor configuration and operating conditions. Based on these results, a larger reactor, with diverse injection methods and a coal flow rate of approximately one ton-per-hour, was 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 VII-1 which includes the initiation of a third phase leasing to a full PDU.

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

PROCESS DESCRIPTION

The basic concept of this process for the liquefaction of coal is that high liquid yields are favored by rapid mixing, reaction, and quenching of a mixture of pulverized coal and hot gaseous hydrogen. In this process, as shown in figure VII-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.

LEGEND: $\triangle$ BEGIN MILESTONE
 ∇ COMPLETE MILESTONE
 $\circ$ DECISION MILESTONE

Figure VII-1. PROGRESS SCHEDULE FOR LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

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

The reactor effluent is passed into a quench section which has a set of water spray nozzles. From there the effluents are passed 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 ¼-ton-per-hour test was conducted at the end of May, and final testing on the unit was completed by the end of 1976. Results of the tests allowed a confident transition to the one-ton-per-hour system and indicated the direction for testing at the larger scale.

Construction of the one-ton-per-hour test facility was completed in December 1976. Shakedown tests on the new one ton-per-hour reactor were completed in January 1977, and the first series of tests completed in April. The second series began in June. Residence time and reactor hydrogen requirements were studied. Phase III of the contract was initiated in August. A redesigned injector, allowing injection of hydrogen at higher temperatures was designed, built, and tested. Successful operation was demonstrated with a hydrogen-to-coal flow rate ratio low enough to be economically significant for a commercial plant.

During the first quarter of 1978, eleven tests were conducted in the 1.37-inch I.D. reactor, using Island Creek coal which was crushed and ground to 90 percent minus 200 mesh size. During the second quarter, efforts were directed to the design of a BTX system and to analysis of results from previous short-duration runs.

An annual technical progress report covering the period August 1977-July 1978 has been published and is available as NTIS No. FE-2044-37. It states that results from the first three years of the program have demonstrated that coal can be effectively processed on a significant scale by flash hydropyrolysis to yield desirable products. Although the entire liquid product from the tests made to date have not been recovered in an easily analyzable form, the quality of the products indicated by the results that have been obtained is attractive.

The influence of reactor operating conditions on the product distribution, quality, and yield has been determined sufficiently to allow selection of near-optimum operating conditions based on an economic analysis.

Further, the operating requirements of the reactor are sufficiently flexible to allow large variations in the product slate to meet economic demands. Conditions can be adjusted to maximize the yield of benzene, toluene, xylene (BTX) and other light liquids or to produce a heavier liquid with lower hydrogen consumption. With the caking western Kentucky coals used to date, the reactor temperature should be greater than $\approx 1,500^{\circ}\text{F}$ to avoid agglomeration in the reactor. It is expected that less strongly caking coals can be processed at lower temperatures without a similar problem. The products are only weakly dependent on reactor pressure, although somewhat heavier liquid products appear to be formed at low pressure. Operation at 500 psi is entirely feasible.

The annual technical progress report notes also that the influence of coal particle size is small, but somewhat greater conversion and lighter liquid products are favored with finer coal. A coal particle size of 90 percent < 200 mesh is preferred, but 70 percent < 200 mesh is nearly as good.

Oxygen addition by either the preburner or high-injection-temperature technique is satisfactory. However, the high-temperature configuration is expected to give better long-term durability.

Additional work is planned or proposed. Complete liquid recovery is needed which will allow the entire product to be characterized. The high-pressure product recovery system being installed currently, coupled with the proposed BTX recovery system, will meet this need and also allow long-term operation with fouling.

PROGRESS DURING JULY-SEPTEMBER 1978

During August, the design of the BTX system was completed. Bids have been requested for the fabrication and installation of the system. Receipt of bids by mid-October is anticipated.

Schedule problems have been encountered on the construction of the high pressure product recovery system because of material procurement and fabrication problems experienced by suppliers of high pressure vessels, and because of a strike at the condenser fabricator.

A heat recovery quench unit is being designed. This unit will allow the water spray quench system to be replaced by a heat exchanger.

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VIII. FLASH PYROLYSIS COAL LIQUEFACTION PROCESS

OCCIDENTAL RESEARCH CORPORATION
LA VERNE, CALIFORNIA

Plant Site: La Verne, California

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

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

INTRODUCTION

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

The main objectives of the program are to:

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

Figure VIII-1. FLASH PYROLYSIS PROJECT SCHEDULE

PROCESS DESCRIPTION

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

Figure VIII-2. FLASH PYROLYSIS PROCESS FLOWSHEET

reactor, the coal is mixed with recycled hot char which rapidly heats the coal to a pyrolysis temperature in the range of 950° to 1,350°F. The char circulation rate ranges from five to ten times that of coal.

Char is separated from the pyrolysis vapors by a series of three cyclones. A portion of the char is cooled as product, and the remainder is fed to the char heater where it is partially burned with air to raise its temperature to 1,200°F to 1,700°F for recycle to the reactor. The second and third stage cyclones remove fine char from the pyrolysis vapor prior to quenching in the tar

collection system. This system consists of three sub-sections: two tar recovery stages and a vacuum flash unit.

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

HISTORY OF THE PROJECT

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

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

The operability of the PDU was demonstrated at the beginning of 1977 by three extended baseline runs with subbituminous coal. Testing with bituminous coal began in May 1977, but continued to be hindered by circulating char upsets. Work was discontinued, by DOE order, in June 1978.

PROGRESS DURING JULY-SEPTEMBER 1978

After operations were halted in June to close out the contract, efforts were initiated at writing the final report through this quarter.

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IX. FILTRATION PROCESS AND EQUIPMENT STUDIES

JOHNS-MANVILLE SALES CORPORATION
DENVER, COLORADO

Project Site: Denver, Colorado
Contract No.: EX-76-C-01-2007
Contract Funding: \$366,963
(100% DOE)

INTRODUCTION

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

PROGRAM DESCRIPTION

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

tering in excess of 20 gallons of solid-liquid mixture per square foot per hour. Data obtained from operation of the pilot scale filtration unit will be used to design filters for commercial plants.

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

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

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

HISTORY OF THE PROJECT

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

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

In early 1977, tests were performed on TFF stock. Differential pressure tests, evaluations of immersion time to investigate filter aid usage relationship, and evaluation of knife blade advance were studied. Tests were also conducted on Wilsonville unfiltered filter feed. A new test leaf system to allow "in situ" operation for future use was designed and built, but no filtration rate data had been secured from the new system by the end of 1977. Filtration experiments continued in 1978, but were hampered by mechanical and operational problems.

PROGRESS DURING JULY-SEPTEMBER 1978

Efforts during July continued toward the solution of problems related to coal solids by-passing the precoat with resultant poor quality filtrate and erroneously high filtration rates (in the order of 1,000 pounds-per-hour-per-square foot).

Three precoats were applied under temperature and pressure. No results were obtained on the first precoat as a result of cracking followed by by-passing of the filter feed. Rates in the order of 1,000 pounds-per-hour-per-square foot were obtained on the second precoat, but with filtrate ash of approximately 0.5 percent. The third precoat was lost due to a leaf modification design to reduce by-passing.

Buchner funnel tests were run on three basecoat filter aids, CELITE AF7, CELITE AF9, and CELITE AF13 (the material currently being used) with Wilsonville Indiana Coal Filter Feed No. 29717. Suspended solids in the filtrate from the CELITE AF13 precoat amounted to two percent, whereas those for CELITE AF9 and AF7 were 0.4 and 0.35 percent respectively. It is concluded from these tests that the CELITE AF13 basecoat is too coarse to prevent coal solids reaching the basecoat by means of precoat cracks or other imperfections from passing through it.

Two precoats were applied in the first two weeks of August. Both precoats went on well with no cracks in the first and one hairline crack in the second precoat. Data was obtained from only one precoat, however, as a leak in the filter vessel agitator packing gland necessitated stopping the second run.

Filtration rates were in the order of 300 pounds-per-hour-per-square foot at a 2 rpm drum speed with filtrate ash of 0.11 percent average; indicating, once again, the very high filtration rates of this sample of Wilsonville Filter Feed.

An aluminum foil lip or baffle applied to the inner rim of the leaf appeared to give improved ash removal.

These are the final tests to be run under this contract. September activities were confined to cleaning up the test unit and placing it in standby condition. Work on a final report commenced.

X. R&D ENGINEERING SERVICES - HYDROCARBONIZATION

OAK RIDGE NATIONAL LABORATORY
OAK RIDGE, TENNESSEE

Contract No.: E(49-18)-1740
Contract Funding: \$2,135,000
(100% DOE)

INTRODUCTION

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

PROCESS DESCRIPTION

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

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

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

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

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

The operability experiments with Wyodak coal were completed in March 1977, and testing began with Illinois No. 6 coal. The Lucite mock-up was used to determine conditions that would give satisfactory fluidization and char circulation in the reactor.

The atmospheric pressure carbonizer which had been used for coal residue carbonization studies was reconverted to support coal hydrocarbonization research during 1977.

Testing continued in 1978, with runs on Wyodak coal at lower system pressures (150 psig) than was used previously. Oil yields were somewhat less at the lower pressure, as was expected. Modifications were made to the bench-scale hydrocarbonizer to increase coal residence time.

PROGRESS DURING JULY-SEPTEMBER 1978

During July, preparations were made to investigate the effects of rapid hydrolysis. For this, changes were made in the four-inch I.D. Lucite mock up system by connecting the draft tube directly to a cyclone to permit rapid disengagement and quenching of volatile products.

Run HC-28 was made in the bench-scale hydrocarbonization equipment to study rapid hydrolysis of Wyodak coal. Analyses of products was not completed in time for inclusion in this report. A considerable amount of char had been entrained in the reactor off-gas. HC-28 was the last scheduled run for the bench-scale setup. Material balances and yields will be determined for Run HC-28. Following that, work will focus on completion of a final report for the project.

XI. ZINC HALIDE HYDROCRACKING PROCESS

CONOCO COAL DEVELOPMENT COMPANY
LIBRARY, PENNSYLVANIA

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

INTRODUCTION

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

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

HISTORY OF THE PROJECT

During 1976, successful liquefaction of subbituminous coal with zinc chloride catalyst was demonstrated in the two pound-per-hour continuous bench-scale unit. Tests of the distillate oil recycle as a vehicle for feeding coal provided assurance of an ample supply of vehicle. In addition to the tests performed, 322 pounds of low-carbon spent melt were produced for use in regeneration studies.

A variable study was completed during 1977 on the bench-scale continuous hydrocracker. Functions of temperature, hydrogen partial pressure, and residence rate time were studied. The data were obtained to develop a reaction rate model for zinc chloride liquefaction to be used for design and economic studies.

A study of the economic value of treating coal ash effluent from the regeneration cyclone in a secondary stage to recover zinc was also completed by the end of

LEGEND: BEGIN MILESTONE
COMPLETE MILESTONE

Figure Xi-1. ZINC HALIDE HYDROPROCESSING PROJECT SCHEDULE

Figure Xi-2. ZINC HALIDE HYDROCRACKING PROCESS FLOWSHEET

1977. The study showed an incentive to add secondary zinc recovery to all cases, with saving of 3.0 to 3.5 cents per gallon of gasoline.

The process design and engineering of the one ton-per-day process development unit were completed and construction continued throughout 1977 with completion of the hydroliquefaction section accomplished early in 1978, and the balance of the PDU completed by mid-1978.

PROGRESS DURING JULY-SEPTEMBER 1978

During July, break-in testing of the hydroliquefaction section of the one TPD PDU continued. Back-up hydrogen compressors were installed and a new type of sheathed heating cable was installed in lines carrying molten salt catalyst. During August, catalyst melt was recirculated through the reactors for the first time. By the end of September, break-in testing of the liquefaction section of the zinc chloride PDU was substantially completed. Two operational periods of 16 and eight hours were conducted feeding zinc chloride and a 250 to 400°C distillate feedstock (Neville solvent)

at rates up to 50 kg/hr. The reactor pressure and temperature were 26.2 MPa (3,800 psig) and 370 to 435°C (688 to 815°F). A 50/50 mixture of Ft. Lewis SRC-I and Neville solvent was fed successfully for the final hour, before shutdown was required by a leaking stirrer gland. The reaction went smoothly with high conversions (> 50 percent to gasoline). These runs demonstrated the basic mechanical integrity of the unit and provided information on control systems and process analyzers.

A supporting laboratory study continues to define the effects of ash metal chlorides on hydrocracking activity of zinc chloride. At mild conditions of temperature and pressure, high concentrations of NaCl, KCl, CaCl₂ or FeCl₂ (20 percent of ZnCl₂) caused the expected severe activity loss: conversion loss of 44 to 54 percent. Further runs were underway to define the effects at lower concentrations and to establish target levels for regenerated melt. Higher ash contents and higher carbonaceous residue contents were shown to cause higher melt viscosities.

Writing continues on the final report of bench-scale work on the economic study to compare gasoline costs via direct coal feeding versus feeding of SRC-I to zinc chloride processing.

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XII. FLUID COKING OF COAL LIQUEFACTION RESIDUES

EXXON RESEARCH AND ENGINEERING
COMPANY

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

Contract Funding: \$1,312,470

INTRODUCTION

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

liquids by methods which utilize 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.

Four samples of coal liquid residues produced in DOE-sponsored coal liquefaction projects will be analyzed and processed using bench-scale equipment. A Continuous Stirred Coking Unit (CSCU) will be used to obtain quantitative yield data for fluid coking of the coal liquefaction residues. This type of pilot unit was utilized successfully in scale-up of yield and quality data for petroleum. CSCU simulates the steam fluidization of a commercial fluid coker by means of a mechanical agitator. A schematic is shown in Figure XII-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. Analytical characterization were completed by the end of 1977 on three samples from DOE-sponsored liquefaction processes. A fourth sample was received for analysis in December. The samples received for analysis consisted of the following:

- Residue 1 - H-Coal Solvent Precipitation Underflow
- Residue 2 - Stripped SRC Coal Liquefaction Settler Underflow (Conoco)

Figure XII-1. FLUID COKING PROGRAM SCHEDULE

Figure XII-2. CONTINUOUS STIRRED COKING UNIT PROCESS SCHEMATIC

Residue 3 - Stripped SRC Antisolvent Deashing Underflow (Lummus)

Residue 4 - H-Coal Vacuum Bottoms

The Interim Technical Progress Report for First Residue was published in June 1977 and is available from NTIS.

By mid-1978, all analytical characterization and all experimental work on Residue 4, the H-Coal Vacuum Still Bottoms, was completed. Also, by mid-1978, nine runs were completed in the Continuous Stirred Coking Unit. Results showed that an increase in liquid yield resulted from increasing the injection steam rate from 15 to 30 percent on feed, while residence time had no observable effect.

PROGRESS DURING JULY-SEPTEMBER 1978

This project will terminate on December 31, 1978. During this third quarter of 1978, no work was scheduled on the Analytical Characterization of Coal Liquefaction Residues (Task 1 of the contract). Task 2, which concerned Bench-Scale Unit Tests, is at the stage where all experimental work on Residue #4, the H-Coal Vacuum Still Bottoms, has been completed. Compilation and interpretation of the data are in progress for inclusion in the third and Final Interim Report on Task 1 and Task 2. Task 3 was deleted. A Final Report is also being prepared.

XIII. UPGRADING OF COAL LIQUIDS

UOP, INC.

DES PLAINES, ILLINOIS

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

Contract Funding: \$945,584

INTRODUCTION

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

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

The objectives of this contract are to:

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

PROJECT DESCRIPTION

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

HISTORY OF THE PROJECT

The present contract is an extension of the program begun by UOP under contract E(49-18)-2010. Evaluations of the primary products of coal liquefaction generated by the Synthoil, Solvent Refined Coal (SRC), and H-Coal processes were completed in 1976, and the final report of the project was written.

The present program began in January of 1977 under contract EF-77-C-01-2566. Process variable studies on H-Coal gas oil began in February and studies on upgraded Exxon Donor Solvent gas oil began in October. The object of the studies was to arrive at optimum conditions for preparing feedstocks for subsequent hydrocracking. Second-stage hydrocracking studies of H-Coal hydrocrackate were completed in November.

Platforming of upgraded H-Coal naphtha was completed in April, and platforming of upgraded Exxon Donor Solvent (EDS) naphtha was completed in September. Hydrotreating process variable studies on EDS gas oil continued into 1978. Hydrotreating converted practically all heptane insolubles to distillate and substantially lowered the heterocyclics content. By mid-year, work on fluid catalytic cracking of EDS derived distillate feedstock was initiated.

PROGRESS DURING JULY-SEPTEMBER 1978

Series flow hydrocracking of a high-nitrogen EDS liquid to gasoline was started in Plant 601 (Run 777) in July, and the tests were concluded in September (Run 779).

In the series flow system, the first reactor was loaded with a UOP-DCB catalyst, and the second reactor was loaded with a UOP-HCA catalyst. The fractionator was operated at maximum gasoline yield mode with 375°F+ fractionator bottoms recycled back to the first reactor.

The final series flow hydrocracking run (Plant 601, Run 779) was started at 500 psig below base pressure, base fresh feed space velocity, and $1.18 \times$ base com-

bined feed ratio (CFR). Complete conversion of feed to gasoline was first attained when the temperature in the second reactor reached 80°C above base temperature with the first reactor at base temperature. At 307 hours on stream, the fresh feed space velocity was reduced from base to $0.5 \times$ base space velocity at otherwise identical pressure and CFR. Complete conversion to 375°F- distillate was again attained as the temperature in the second reactor reached base temperature with the first reactor operated at near-base temperature. Run 770 was terminated after 497 hours on stream. The series flow system remained reasonably stable during low pressure operation.

An EDS hydrocracked naphtha was reformed over a commercial Platforming catalyst. Experiments were carried out over a series of conditions to obtain data for a yield octane curve.

All hydrocracking runs on EDS derived feedstocks have been completed. Preparation of the interim report will be underway when all required analyses of the hydrocracked products have been completed and all product distribution data are obtained.

Preparation of an EDS derived feedstock for the final fluid catalytic cracking run has been started. FCC studies on this feed should be completed in October.

XIV. GASIFICATION OF RESIDUAL COAL LIQUEFACTION PRODUCTS

TEXACO, INC.

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

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

INTRODUCTION

Under this agreement, Texaco, Inc. will utilize its Texaco Synthetic Gas Generation Process (TSGGP) for gasification of residual material from coal liquefaction samples supplied by DOE. The objective of the contract is to evaluate materials from several coal liquefaction pilot plants to determine their suitability as feedstocks for a coal gasification process. After initial laboratory testing and evaluation, promising materials selected by DOE will be tested in large scale pilot plant gasification runs. Provisions have been made for the three types of tests are shown in the following table:

Test Type	Description	Max. Sample Size	Min. No. of Samples	Max. No. of Samples
I	Laboratory Evaluation	20 lbs.	4	15
II	Preliminary Pilot Plant Evaluation	20 bbls.	0	4
III	Extended Pilot Plant Evaluation	200 bbls.	0	4

The Type I evaluation will determine the chemical composition and physical properties of each material. On the basis of these results the mode of feeding the material to the Texaco Gasifier will be selected and the gasification conditions, yields and product gas composition will be estimated. An assessment of the suitability of the material for further testing will be made.

The Type II evaluation will consist of a preliminary pilot plant test in which approximately 20 barrels of

the candidate feedstock will be gasified. This short pilot plant evaluation will confirm the operability of the process on the candidate feedstock and permit refining the estimates of preferred processing conditions, product gas yield and composition. Unexpected operation problems will be identified.

The Type III evaluation will consist of an extended series of runs in which approximately 200 barrels of the candidate feedstock will be gasified. Operating conditions will be varied to further define the optimum. Definitive heat and material balance data will be obtained.

All materials received will be given a Type I evaluation. Each succeeding type of evaluation will be performed only on the most promising materials from the preceding evaluation at the request of DOE.

Almost all coal liquefaction processes, which are being developed require hydrogen or synthesis gas to solubilize the coal. In order to obtain a favorable product yield in such a coal liquefaction plant it is necessary to produce the needed hydrogen or synthesis gas primarily from the residue fraction of the coal. This material, together with the inorganic ash and some fraction of the converted coal, may be recovered in various forms depending on the particular process. Some of these waste materials will make nonliquefiable feedstocks for gasification using the Texaco Coal Gasification Process which produces synthesis gas or hydrogen. Others are liquefiable and suitable for the Texaco Synthesis Gas Generation Process.

HISTORY OF THE PROJECT

The contract was awarded to Texaco in June 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 feedstock 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.

Type I evaluations to determine the chemical composition and physical properties of coal liquefaction residues were performed on six samples during 1977. A preliminary pilot plant evaluation was also completed on H-Coal Syncrude Vacuum Tower Bottoms.

By mid-1978, final reports on the Type II Preliminary Pilot Plant Evaluation of 20 barrels of SRC-II vacuum flash drum bottoms and Exxon EDS vacuum tower bottoms were completed and submitted to DOE. Also, a Type I laboratory evaluation of char obtained from short residence time liquefaction of Kentucky No. 9 coal in the Rocketdyne high-mass-flow pilot process development unit was completed. A total of nine Type I Laboratory Evaluations, three Type II Preliminary Pilot Plant Evaluations and one Type III Extended Pilot Plant Evaluation have been completed to date. The materials are listed in the following table.

COAL LIQUEFACTION RESIDUES
EVALUATED TO DATE

Material Tested	Original Charge Coal	Test Level	Report Fe-2247
H-Coal Syncrude Vacuum Tower Bottoms	Ill. No. 6	I	2
H-Coal Settler Stripped Underflow	Ill. No. 6	I	3
Synthoil Centrifuge Underflow	Ky. 9	I	5
USS Clean Coke Hydrogenation Vapor Stripper Bottoms	Ill. No. 6	I	6
H-Coal Syncrude Vacuum Tower Bottoms	Ill. No. 6	II	8
SRC-II Vacuum Flash Drum Bottoms	Ky. 9/14	I	9R
Exxon Donor Solvent Vacuum Tower Bottoms	Ill. No. 6	I	10
Kerr-McGee Ash Concentrate	—	I	12
SRC-II Vacuum Flash Drum Bottoms	Ky. 9/14	II	14
Exxon Donor Solvent Vacuum Tower Bottoms	Ill. No. 6	II	15
Rocketdyne Char	Ky. 9	I	17
SRC-II Vacuum Flash Drum Bottoms	Ky. 9/14	III	20

PROGRESS DURING JULY-SEPTEMBER 1978

A Type III Extended Pilot Plant Evaluation of SRC-II vacuum flash drum bottoms from the liquefaction of Kentucky No. 9/14 coal was successfully completed during this quarter.

A total of 31 tons of the SRC-II residue, which was prepared at the Ft. Lewis, Washington pilot plant, were gasified during eight pilot plant runs at 350 psig.

The runs lasted from 8.0 to 14.8 hours each and a total of 78 hours of on-stream time was accumulated. The SRC-II residue contained over 27 weight percent ash.

The residue throughput, the steam/residue ratio and the oxygen/residue ratio were varied to determine the optimum operating conditions. At an oxygen/residue ratio of 0.73/1.00, a 99 percent conversion of carbon to syngas was achieved. More oxygen made little improvement in conversion while less oxygen resulted in a rapid loss of conversion. At 99 percent conversion, approximately 29 standard cubic feet of hydrogen plus carbon monoxide were produced per pound of residue gasified. The steam/residue ratio controlled the gasifier temperature at a given oxygen and residue rate but had little effect on conversion.

The gasifier temperature varied between 2,500°F and 2,650°F, the conversion of carbon to syngas varied between 97.3 percent and 99.5 percent. The cold gas efficiency varied between 80 and 83 percent.

No major operating problems were experienced during any of the key phase of operation which included melting the residue, pumping molten residue to the gasifier, gasification, syngas quenching and scrubbing, slag removal and char recovery. An atmosphere of nitrogen gas was maintained above the molten residue to minimize oxidation and polymerization. The molten residue was pumped to the gasifier undiluted at an average temperature of 515°F.

No evidence of significant refractory damage or serious burner tip erosion was observed.

Data from the runs are still being analyzed. No significant operating problems were experienced during the recently completed Type III Extended Pilot Plant Evaluation. Material balance from the runs will be reported as soon as data analysis is complete.

XV. PRODUCTION OF CLEAN INDUSTRIAL AND TRANSPORTATION FUELS FROM COAL

THE LUMMUS COMPANY
BLOOMFIELD, NEW JERSEY

Plant Site: Bloomfield, New Jersey
Contract No.: EX-76-C-01-2514
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 XV-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 XV-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 XV-1. CLEAN FUELS FROM COAL PROGRAM DEVELOPMENT SCHEDULE

Figure XV-2. CLEAN FUELS FROM COAL PROCESS SCHEMATIC

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

The Lummus process uses a proprietary solvent de-ashing system which, unlike filters and centrifuges, does not contain costly and hard-to-develop mechanical equipment, thus simplifying scale-up. 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 gasification 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 sub-bituminous coals have been processed successfully in the bench-scale pilot plant.

The program was initially submitted to ERDA, now DOE, in 1975 as an unsolicited proposal. Following contract negotiations, a revised experimental program was authorized and a contract granted by ERDA on September 28, 1976. Modifications were started to convert the expanded-bed liquefaction test unit and its ancillary anti-solvent de-ashing module, a distillation unit, and two vacuum flashing units to permit integrated continuous operation. Startup of the hydroliquefaction unit began in August 1977 with operations during the remainder of the year directed toward the preparation of equilibrium recycle solvent.

Operations on catalyst life testing were concluded by mid-1978 and the hydroliquefaction unit was readied for operations in connection with the program involving process variable studies.

PROGRESS DURING JULY-SEPTEMBER 1978

During this quarter, the Process Development Unit was operated to obtain data covering a process variable study. The first process variable study run was com-

pleted at an 820°F reactor temperature, an operating pressure of 2,700 psig, a coal space rate of 1.6 pounds-per-hour per pound of catalyst, and utilizing a 35 weight percent coal concentration paste. The first of three catalyst activity runs was then performed at an 820°F reactor temperature, a 2,100 psig operating pressure, a space rate of 1.6 pounds of coal per hour per pound of catalyst and 30 weight percent coal paste concentration.

An improved hydrogen consumption determination method based on elemental analyses was initiated, resulting in a 2.25 weight percent hydrogen consumption in the variable study run, and a 2.91 weight percent hydrogen consumption in the catalyst activity study run.

Two additional variable study runs were completed during September. The operating conditions for the first run were an 850°F reactor temperature, an operating pressure of 2,700 psig, and a coal space rate of 2.45 pounds of coal per pound of catalyst. For this run, the hydrogen consumption was 3.46 pounds per 100 pounds of coal as charged.

In the second run, the respective temperature, pressure and coal space rate values were 820°F, 2,700 psig, and 2.42. The hydrogen consumption in this instance was 2.13 pounds per 100 pounds of coal.

The hydroliquefaction unit operated satisfactorily during both of the above runs with the exception of a brief period during the last day of operation on HDS-71, when the pressure control valve on the low-pressure letdown vessel preceding the atmospheric flash unit leaked over a period of several hours. This resulted in a pick-up of some solvent boiling-range material (subsequently recovered) in the light oil distillate during this period.

The deashing unit performed satisfactorily during this period, with the antisolvent-free overflow averaging less than 0.05 percent ash. The vacuum flash unit utilized for the stripping operations on the deasher underflow sustained the deep-stripped underflow softening point at the desired level of 360°F or higher during most of this period. During the course of completing the deashing/vacuum flashing operations associated with HDS-72 operations, some erosion-induced problems were becoming apparent, affecting the performance of several recirculating pumps in the system. Corrective maintenance action was in progress at the end of the reporting period.

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Table 1 presents the operating conditions, hydrogen consumption, gas make, and +500°F net paste oil make for the four runs to date which relate to the process variable study phase of this program.

Table XV-1. HYDROGEN CONSUMPTION, GAS MAKE, AND +500°F NET PASTE OIL MAKE FOR SEVERAL RECENT COAL HYDRO-LIQUEFACTION RUNS

Run Designation	67	68	71	72
Temperature, °F	820	830-835	850	820
Pressure, psig	2700	2100	2700	2700
Coal	Illinois No. 6			
Coal Content of Fresh Paste Feed, wt. %	35	30	35	35
Coal Space Velocity				
lb coal/hr./lb. catalyst	1.64	1.47	2.45	2.42
Volumetric Liquid Recycle/				
Fresh Feed Ratio	1.0	71	.57	.57
Net Chemical Hydrogen Consumption,				
lb/100 lbs coal (as is)	2.25	2.91	3.46	2.13
Light (C ₁ -C ₄) Hydrocarbon Gas Make,				
lb/100 lb Coal (as is)	6.07	6.96	8.62	4.28
Net +500°F Paste Oil Make,				
lb/100 lbs coal (as is)	25.65	20.41	23.78	22.22

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; ° 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—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.