COAL LIQUEFACTION

QUARTERLY REPORT

OCTOBER – DECEMBER 1976

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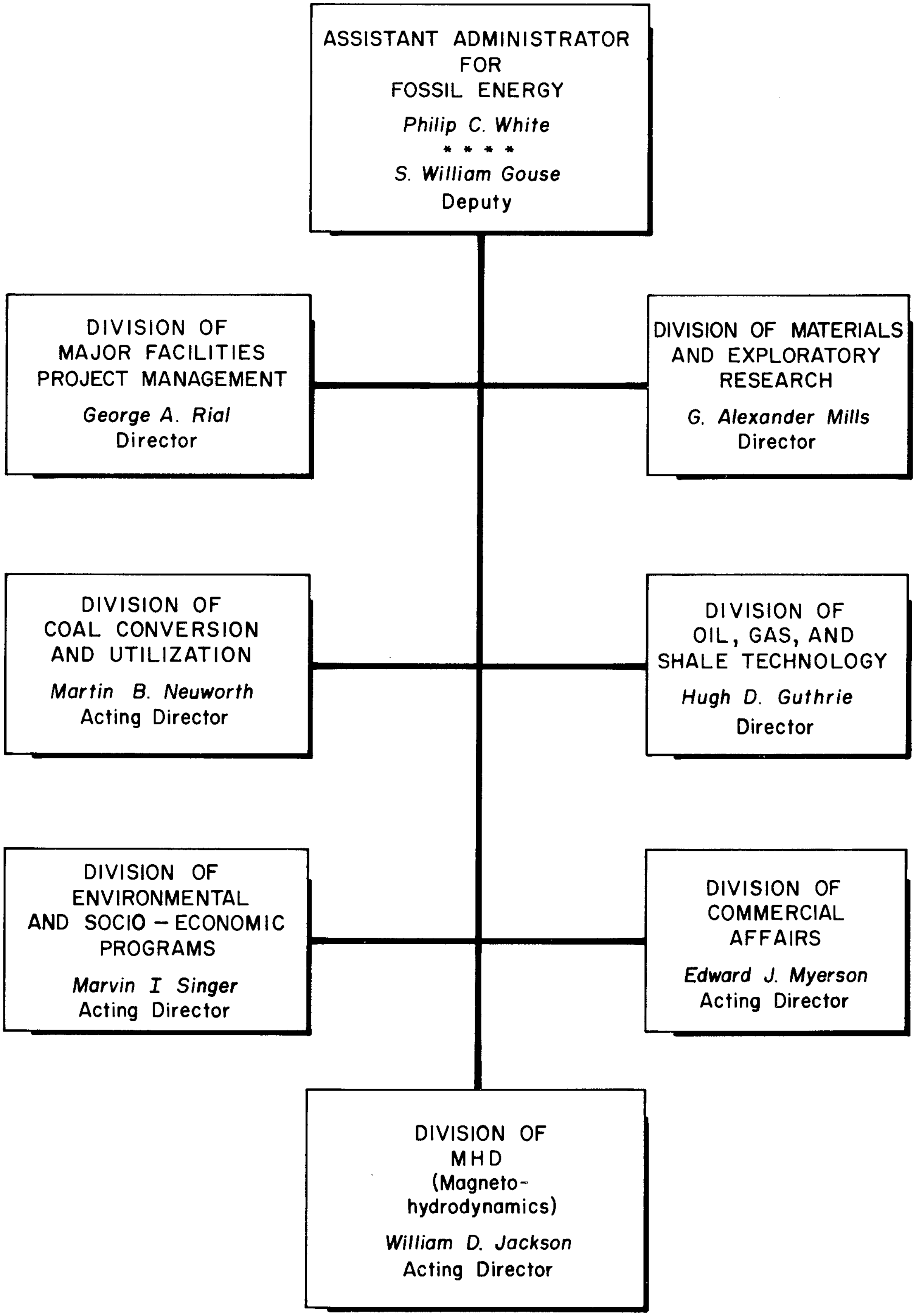
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EXECUTIVE SUMMARY

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

The technologies selected for development—gasification, liquefaction, and combustion—satisfy an urgent need for a particular type of fuel, are potentially feasible both technically and economically (in terms of the costs of research and development and the final product) and will not exceed air, water, and solid pollution standards established by the Environmental Protection Agency (EPA). The emphasis given each technology varies, depending on such considerations as technical complexity, development stage (laboratory research including bench-scale tests and experiments with process development units, and pilot plant design, construction, and operation), variety of uses for the fuel produced, and urgency of the need that the technology is designed to satisfy.

ERDA's program for the conversion of coal to liquid fuels was begun by two of ERDA's predecessor agencies: Office of Coal Research (OCR) in 1962, and Bureau of Mines, U.S. Department of the Interior, in the 1930's. Techniques for converting coal to synthetic liquid fuels, originally developed in Germany in the early 1930's, are being improved to increase the supply of easily transportable and usable liquid fuel. Current emphasis is on the development of fuels suitable for firing industrial and electric utility boilers and gas turbines.

Recent improvements have provided better catalysts, reactor designs, and construction materials, and have led to more attractive processing economics and lower capital investment. Coal liquefaction can now be achieved under more moderate processing conditions and more rapidly than was the case in the 1930's. The advantage of coal liquefaction is that the entire range of liquid products, including fuel oil, gasoline, jet fuel, and diesel oil, can be produced from coal by varying the type of catalysts and operating conditions used in the process. Furthermore, coal-derived liquid fuels also have the potential for use as chemical feedstocks.

To determine the most efficient means of utilizing coal resources, ERDA is sponsoring the development of several conversion processes that are currently in the pilot plant stage. Responsibility for the design, construction, and operation of the ***Solvent-Refined Coal (SRC) Process*** pilot plant at Ft. Lewis, Washington is assigned to Pittsburg and Midway Coal Mining Company. The plant produced a record of 500 tons of SRC during the quarter, and concluded a 3,000-ton production run begun late in 1975 for combustion testing of SRC by Southern Services Company's Georgia Power Company.

The ***H-Coal Project*** will culminate in the operation of a 600 ton-per-day coal liquefaction pilot plant at Catlettsburg, Kentucky to advance H-Coal technology to the level necessary to enable design and construction of a commercial plant. Hydrocarbon Research, Inc. (HRI) continued design and procurement of equipment for the H-Coal Pilot Plant during the quarter. Laboratory work supporting the design was completed by HRI and final reports were prepared. Laboratory work to support pilot plant operation was initiated and will continue through 1977 under a contract with HRI. Ashland Synthetic Fuels, Inc. (ASFI) is responsible for pilot plant construction and operation under a separate contract. Construction started in December 1976. HRI will provide engineering, design services, and field support as a subcontractor to ASFI during the construction and operation phases.

ERDA, together with the Electric Power Research Institute (EPRI), has contracted with Southern Services Company, Inc. for the ***Operation of a Solvent-Refined Coal Pilot Plant*** at Wilsonville, Alabama. Results of the plant's operation will be compared with the results from the Ft. Lewis, Washington SRC facility in order to improve the SRC process. Operation at the Wilsonville plant during the quarter concluded runs with Monterey (Illinois No. 6) coal and started tests with Amax coals.

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. An operability study in the fluid coking mode was successfully completed during the quarter in the two-barrel-per-day integrated coker/gasification plant, and operation of the one-ton-per-day and smaller scale bench liquefaction units continued to measure the process response for Western coals.

Cities Service Research and Development Company is conducting pilot plant studies of the ***Commercial Scale Ebullated-Bed Hydroprocessing of Solvent Refined Coal Extract.*** The last of the runs designed to determine the operating conditions and modifications necessary to process the SRC in an existing commercial LC-Fining unit were completed during this quarter.

Fluor Engineers and Constructors, Inc. is responsible for reactivating an ERDA-owned equipment test and evaluation facility in Cresap, West Virginia. Refurbishing activities at the ***Cresap Test Facility*** were approximately 99 percent complete by the end of the quarter.

Construction of a process development unit (PDU) is being managed by Foster Wheeler Energy Corporation to test the ***Synthoil Process***, which was originally developed by the U.S. Bureau of Mines. Associated laboratory research is being conducted by the Pittsburg Energy Research Center (PERC), Sandia Laboratories, Argonne National Laboratory, Battelle Memorial Institute (Columbus Laboratories), and Exxon Research and Engineering Company.

The ***Clean Coke Process*** is being developed by USS Engineers and Consultants, Inc., a subsidiary of United States Steel Corporation. The results of laboratory tests and data obtained from the PDU will be used for planned scale-up. During the quarter the flash vaporizer of the binder-preparation PDU was operated to process liquid blowdown from the carbonization PDU. An extended coal-conversion tests is planned for next quarter.

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. Final testing of a $^1/_4$-ton-per-hour system was completed during the quarter as was the design and fabrication of the hardware for a one-ton-per-hour system. Construction of the test facility was also completed in December, and initial preparations for testing were also completed.

Studies are being conducted by the Occidental Research Corporation to evaluate the commercial potential of the company's ***Flash Pyrolysis Coal Liquefaction Process***. Runs on the PDU during the quarter completed the first major program of the contract. Bench-scale runs confirmed the relatively high yield of tar which is characteristic of the process.

Research to improve solid-liquid separation is being conducted by Johns-Manville Sales Corporation in their ***Filtration Process and Equip-***

ment Studies. Filtration characteristics of SRC product from both Tacoma and Wilsonville as well as Synthoil product were determined and compared during the quarter.

Dow Chemical Company is conducting studies dealing with the production of ***Chemicals From Coal*** using the liquids from four coal liquefaction processes (COED, Synthoil, H-Coal, and SRC) in standard petrochemical processing experiments. During this quarter, the studies were concerned with the hydrotreatment of the hydrocrackate naphtha from the SRC II product, and the SRC II straight run naphthas.

Oak Ridge National Laboratory is providing ***R&D Engineering Service*** dealing with ***Hydrocarbonization*** and ***Carbonization of Residue*** from the SRC, H-Coal, and Consol Synthetic Fuel processes. Hydrocarbonization is a type of coal liquefaction process which combines high-temperature, high-pressure, and fluidized-bed operation using hydrogen-rich gas for fluidization, thus providing controlled product yields. During the quarter, three runs were completed in the 20-atmosphere bench-scale hydrocarbonizer in a recirculating fluidized-bed mode. Based on the design used in the hydrocarbonizer, a new rotary feeder was installed in the batch reactor used for the carbonization of residue, and eight carbonization runs were completed.

Continental Oil Company, with the participation of Shell Development Company, is developing the ***Zinc Halide Hydrocracking Process***, a technique designed to produce clean liquid and gaseous fuels from coal or coal extract. Batch kinetic studies of secondary recovery of zinc chloride from the cyclone ash continued during the quarter.

Exxon is studying the potential of ***Fluid Coking of Coal Liquefaction Residues*** to recover distillate liquids. Studies completed during the quarter included analytical tests on an H-Coal solvent precipitation underflow sample.

To facilitate the possible future upgrading of primary coal liquids using technology now available in the petroleum industry, UOP, Inc. is conducting a ***Characterization of Coal Liquids***, study, Processability characterization tests were performed during the quarter on the last three of eight ERDA coal liquids.

Texaco, Inc. is conducting a study of the ***Gasification of Residual Coal Liquefaction Products***. Materials from several coal liquefaction pilot plants will be evaluated to determine their suitability as feedstock for a proprietary Texaco coal gasification process. Studies during the quarter were performed on two possible candidate feedstocks obtained from the H-Coal liquefaction of Illinois No. 6 coal.

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

I. SOVENT-REFINED COAL (SRC) PROCESS

THE PITTSBURG & MIDWAY COAL MINING COMPANY

MERRIAM, KANSAS

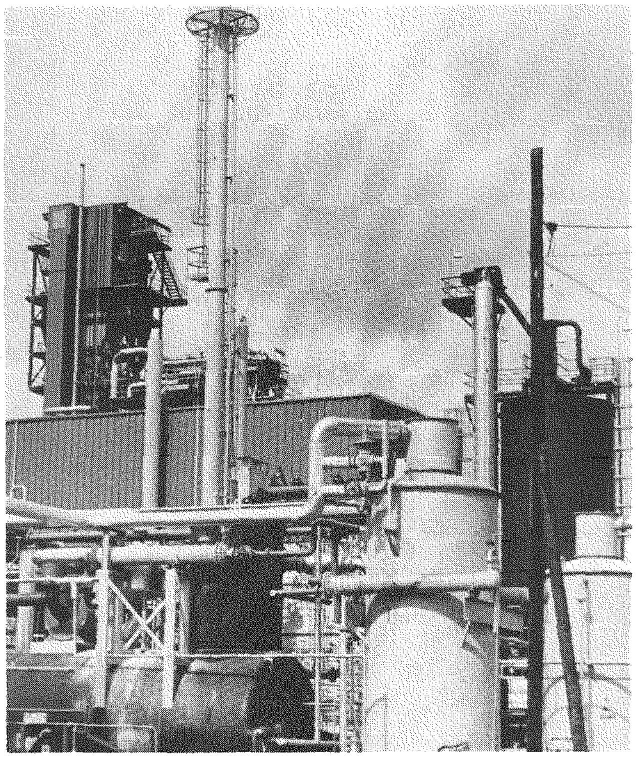
Plant Site:	Tacoma, Washington
Contract No.:	E(49-18)-496
Total Funding:	$82,098,707 *(100% ERDA)*

INTRODUCTION

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

PROCESS DESCRIPTION

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



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

The slurry of undissolved solids and the coal solution is then separated in a filtration unit. In a commercial-scale process, the solids would be sent to a gasifier-converter where they would react with supplemental coal, steam, and oxygen to produce hydrogen for use in

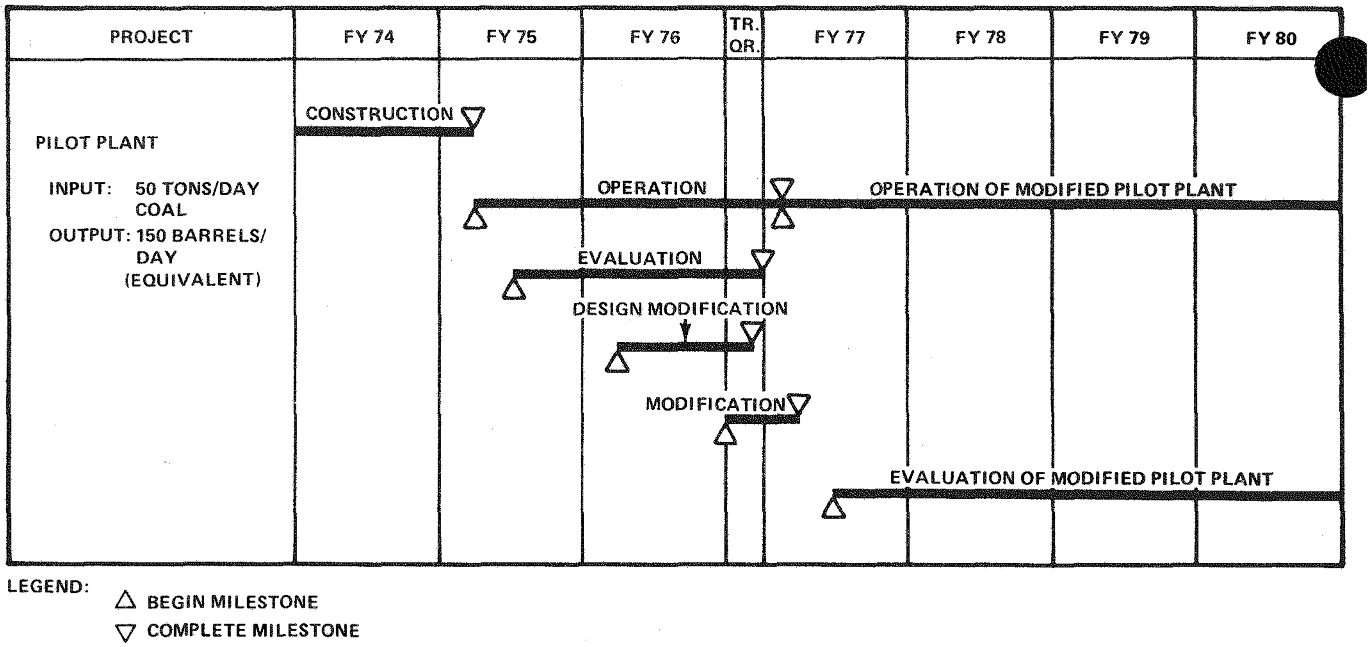


Figure I-1. SOLVENT-REFINED COAL PROGRAM SCHEDULE

the process. The coal solution passes to the solvent recovery unit and the final liquid product, solvent-refined coal, is produced. This material has a solidification point of 350° F to 400° F and a heating value of approximately 16,000 Btu per pound.

HISTORY OF THE PROJECT

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

To develop the SRC process further, a contract was awarded to P&M to design, construct, and operate a pilot plant that would be capable of processing 50 tons of coal per day. In 1969, Stearns-Roger Corporation completed the design for the pilot plant but funds to begin construction were not available until late 1971. In June 1972, OCR extended its contract with P&M to construct and operate the pilot plants. 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.

During 1975, eleven material balance runs were made on the SRC pilot plant in the distillate solvent mode in order to study the effects of different coal feed rates and dissolver temperatures. Five successful runs were also made during 1975 in the slurry recycle mode. Overall, the 50-ton-per-day pilot plant operated smoothly through 1975. In the latter part of the year, production of specification SRC product for use in a full-scale utility boiler combustion test began.

The laboratory unit was also run in both the distillate solvent mode and slurry recycle mode during 1975. The variables investigated included filtration rate, solvent recycle rate, slurry residence time, concentration of coal in the slurry, and temperature. Tests were also run to simulate operating conditions at the pilot plant.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

A record of 500 tons of SRC was produced in October, followed by the second largest monthly production

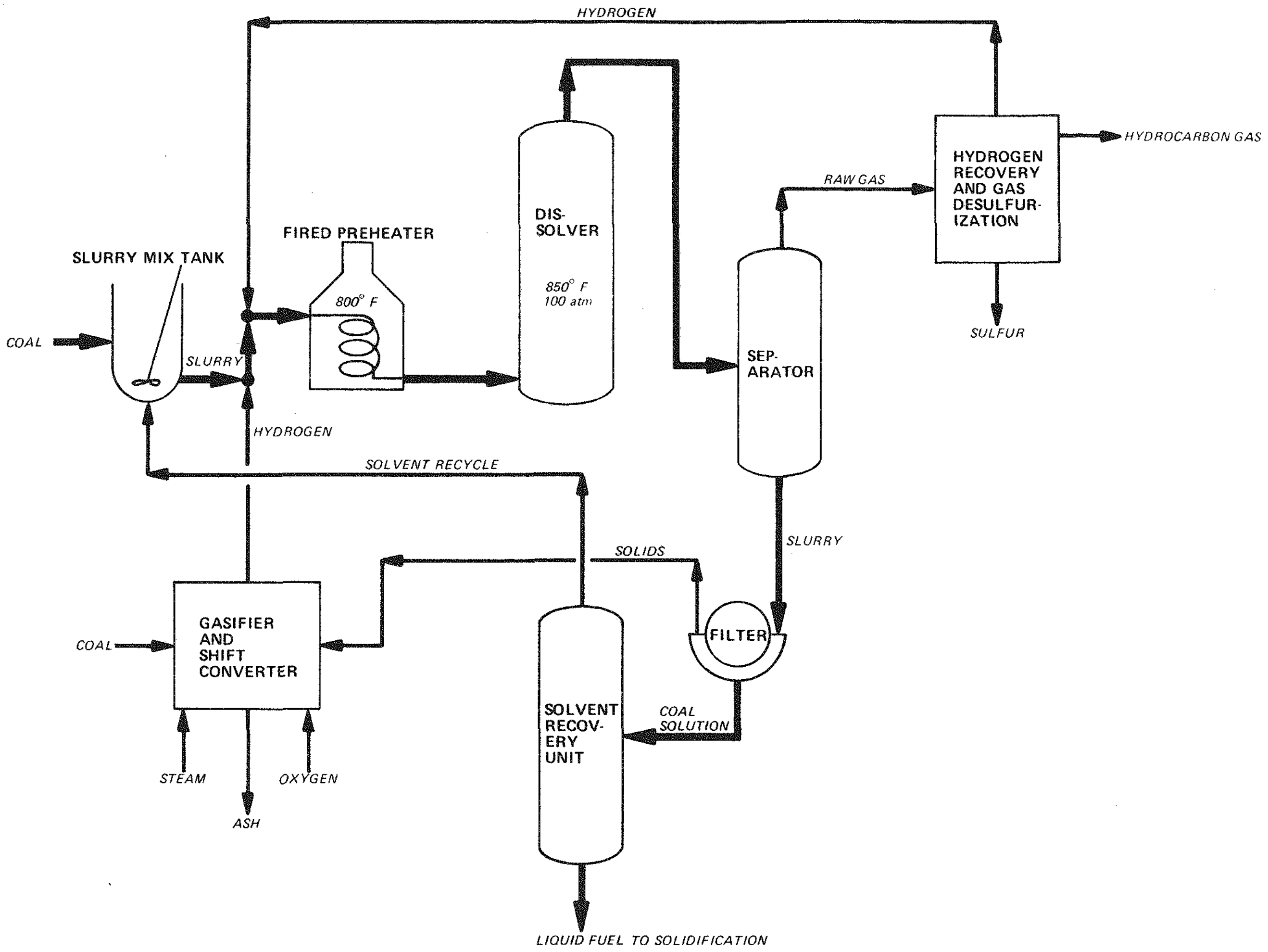


Figure I-2. SRC PROCESS SCHEMATIC

total of 484.2 tons. This concludes the 3,000-ton production run begun late in 1975 for combustion testing of SRC by Southern Services Company's Georgia Power Company. Record production was possible due to exceptional filter performance.

Following an extensive revamp of filtration instrumentation, an experimental program on filtration rates was started. The pilot plant was operated in December to make four filtration test runs. It was shut down on December 19 for a major maintenance turnaround and SRC-II piping tie-ins.

Actual field construction for the SRC II modification is about 30 percent complete, with the overall project being 60 percent complete. The scheduled completion date was set back one month to March 15.

At the Merriam Laboratory, nine experimental runs were completed in the series designed to develop the SRC II process (slurry recycle). The effect of process variables on SRC II operation was investigated.

Pilot Plant Operations

Coal was processed for 19 days in October, with SRC production setting a new monthly production record of 500 tons. A total of 876 tons were processed at feed rates of 2,500 to 4,500 pounds per hour and a solvent-to-coal ratio of 1.6. Operating conditions of 1500 psig and 850° F were maintained for one half of dissolver "A".

The filter operated exceptionally well during this period, requiring only preventive maintenance. Filter "A" produced 3.86 million pounds of filtrate for an average rate of 130 pounds per square foot per hour. Filter "B" produced 0.59 million pounds of filtrate at 166 pounds per square foot per hour. The mineral residue dryer operated with problems and was decoked.

The plant was shut down once during October for major repairs to the wash solvent column and reboiler. All but two of the 21 wash solvent column trays had collapsed and the wash solvent column reboiler was heavily coked. The light ends column reboiler also required repair during the quarter.

The typical analysis of on-specification SRC product during the month was:

Carbon	86.6 wt. percent
Hydrogen	5.77 wt. percent
Nitrogen	2.15 wt. percent
Sulfur	0.71 wt. percent
Oxygen (by difference)	4.63 wt. percent
Ash	0.14 wt. percent
Heating Value	15,844 Btu/lb. (Dulong)
Fusion Point	330° F (Gradient Bar)

The second largest production total of the year was recorded in November with 484.2 tons of specification SRC produced in 25 days of on-stream performance. A total of 814 tons of coal were consumed during the period. Coal feed rates varied from 2,500 to 4,500 pounds per hour at a 1.6:1 solvent-to-coal ratio.

The record production was made possible by the availability of Filter "A" throughout the entire month. Filter "A" produced 3.94 million pounds of filtrate at an average rate of 109 pounds per square foot per hour. Filter "B", operating with some difficulty due to a new screen, produced 1.09 million pounds of filtrate at an average rate of 138 pounds per square foot per hour. Following an extensive revamp of filtration instrumentation, an experimental program was initiated to study the effects of varying drum speed, knife advance rate, pressure drop, and precoat procedures.

The analysis of the SRC produced during November was:

Carbon	86.87 wt. percent
Hydrogen	5.96 wt. percent
Nitrogen	2.16 wt. percent
Sulfur	0.72 wt. percent
Oxygen (by difference)	4.14 wt. percent
Ash	0.15 wt. percent
Heating Value	16,039 Btu/lb. (Dulong)
Fusion Point	308°F (Gradient Bar)

The SRC pilot plant was on stream 13 days in December in order to make four filtration test runs on Filter "B". During this period, 421 tons of coal were processed to produce 231 tons of SRC. The plant was shut down on December 19 for a major maintenance turnaround and SRC II piping tie-ins. The mineral residue dryer was decoked early in the month and operated after the shutdown to process accumulated cake slurry.

A typical analysis of on-specification SRC during December was:

Carbon	87.08 wt. percent
Hydrogen	5.76 wt. percent
Nitrogen	2.12 wt. percent
Sulfur	0.78 wt. percent
Oxygen (by difference)	4.10 wt. percent
Ash	0.16 wt. percent
Heating Value	16,017 Btu/lb. (Dulong)
Fusion Point	330° F Gradient Bar)

SRC Modification

Actual field construction of the SRC II process modification is about 30 percent complete, with the overall project being 60 percent complete. Material delivery problems and a delay in approving the electrical subcontract resulted in a setback of the scheduled completion date by one month to March 15. This construction delay will result in a minimum plant down time.

Laboratory—Merriam, Kansas

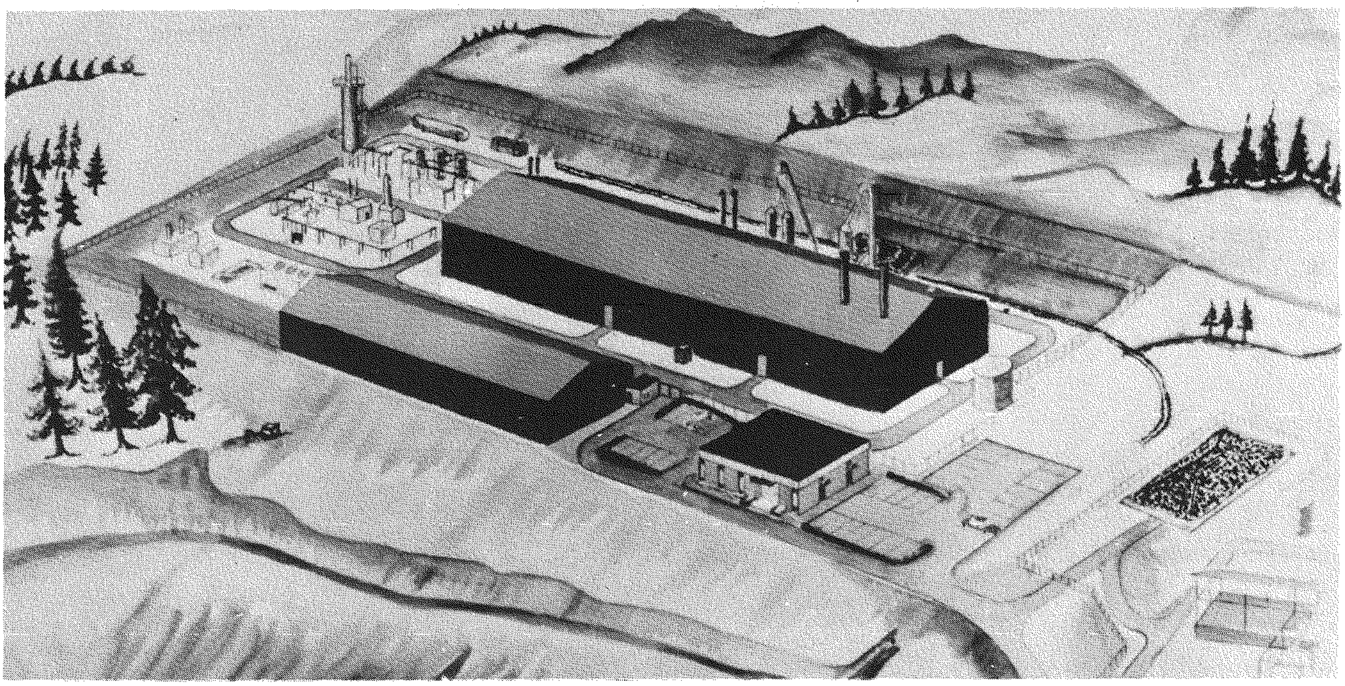
A total of nine SRC II (slurry recycle mode) runs were completed during the quarter. The runs in October were made to evaluate a new preheater designed to more closely match the flow parameters of the Ft. Lewis pilot plant preheater. It was concluded that no significant difference in the runs could be attributed to the variations in the preheater.

The runs in November were designed to determine the effect of increased hydrogen feed rate. It was determined that any improvement due to increased hydrogen availability is nullified by the decrease in actual liquid residence time due to the increased gas phase volume.

The investigation of the effect of process variables on SRC II operation continued in December. Lowering the dissolver temperature to 445° F had a substantial effect on product distribution. With the decrease temperature, oil yield decreased from 35.9 to 31.0

percent and solid SRC yield increased from 24.2 to 31.1 percent. The temperature decrease also resulted in the expected increase in hydrogen and sulfur levels of the heavy distillate product.

II. SYNTHOIL PROCESS



FOSTER WHEELER ENERGY CORPORATION

LIVINGSTON, NEW JERSEY

Total Funding: $24,425,776

SANDIA LABORATORIES

ALBUQUERQUE, NEW MEXICO

Total Funding: $1,636,000

ARGONNE NATIONAL LABORATORY

ARGONNE, ILLINOIS

Total Funding: $1,574,000

BATTELLE MEMORIAL INSTITUTE, COLUMBUS LABORATORIES

COLUMBUS, OHIO

Total Funding: $156,000

EXXON RESEARCH AND ENGINEERING COMPANY

BAYTOWN, TEXAS

Total Funding: $100,000

Plant Site: Bruceton, Pennsylvania

INTRODUCTION

Development of the Synthoil process, initiated by the U.S. Bureau of Mines, is currently being managed by ERDA through the Pittsburgh Energy Research Center (PERC) in Pennsylvania. The objective of this project is to determine the technical and economic feasibility of the process for scale-up to commercial use. Foster Wheeler Energy Corporation is responsible for designing and managing the construction of a ten-ton-per-day process development unit to test the Synthoil process. PERC is conducting support research for the design of the unit. In addition, PERC is monitoring laboratory research on various aspects of the Synthoil process being conducted by Sandia Laboratories, and Argonne National Laboratory. Research being conducted at Exxon Research and Engineering and Battelle Memorial Institute is being monitored by the Morgantown Energy Research Center (MERC) in West Virginia. The schedule for the Synthoil project is shown in Figure II-1.

PROCESS DESCRIPTION

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

Hydrogen propels the slurry so violently through the fixed-bed catalytic reactor that plugging of the bed by the mineral matter in the coal is prevented as the coal becomes liquefied. The turbulence of the slurry promotes mass and heat transfer in the slurry, which in turn promotes hydrodesulfurization and liquefaction.

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

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

HISTORY OF THE PROJECT

Work on this process began in 1969 at PERC. The initial work on the Synthoil process used a reactor with an internal diameter of 5/16-inch in a bench-scale plant that processed five pounds of slurry per hour. Experi-

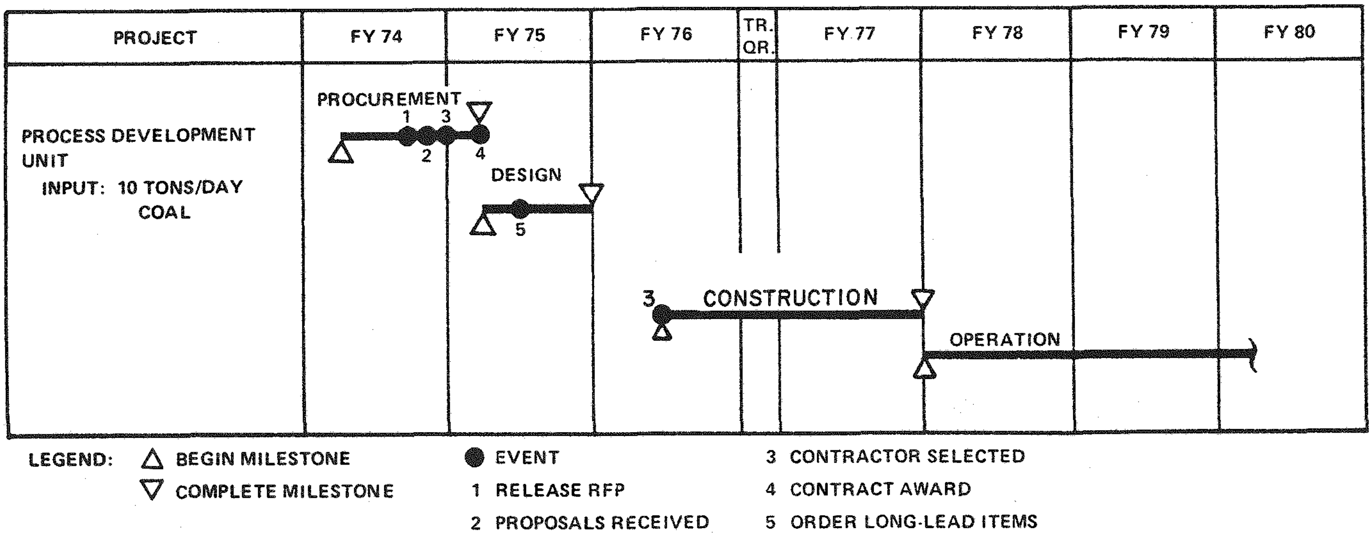


Figure II-1. SYNTHOIL PROGRAM SCHEDULE

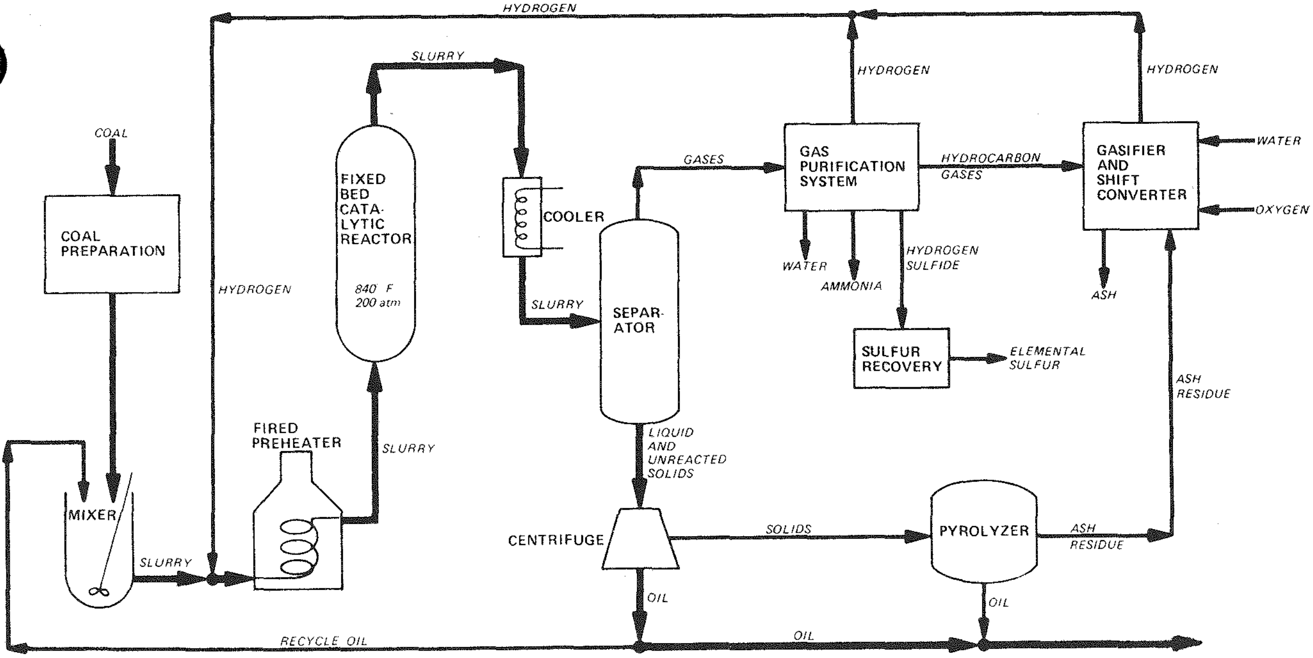


Figure II-2. SYNTHOIL PROCESS SCHEMATIC

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

To demonstrate the broad applicability of the process, a ½-ton-per-day slurry unit was constructed that used a reactor of 1.1-inch ID made of two interconnected stainless steel pipes that were each 14.5 feet long. The operations were carried out on several types of coal at reactor pressures of 140 atm and 280 atm and 840° F. High yields of low-sulfur and low-ash fuel oil were obtained. At the lower reactor pressure the calorific value, product oil yield, and hydrogen consumption was also lower. However, the sulfur and ash content of the product oil was higher than that of the oil produced at 280 atm. Operation at lower pressure would be favorable if the higher sulfur and ash content were environmentally acceptable. Economic analyses will decide the appropriate pressure for this process, considering the capital and operating costs for high-pressure and low-pressure operations, and the environmentally acceptable sulfur and ash contents of the product oil.

The overall design package for the process development unit was completed during 1975. The construction bid packages for both site preparation and plant construction were also completed during 1975. In addition, long lead equipment was placed on order. Actual field construction of the facility began during the fourth quarter of 1975.

III. H-COAL PROCESS

HYDROCARBON RESEARCH, INC.

TRENTON, NEW JERSEY

Plant Site: Catlettsburg, Kentucky

Contract No.: No. E(49-18)-1544 and
EF-77-C-01-2547

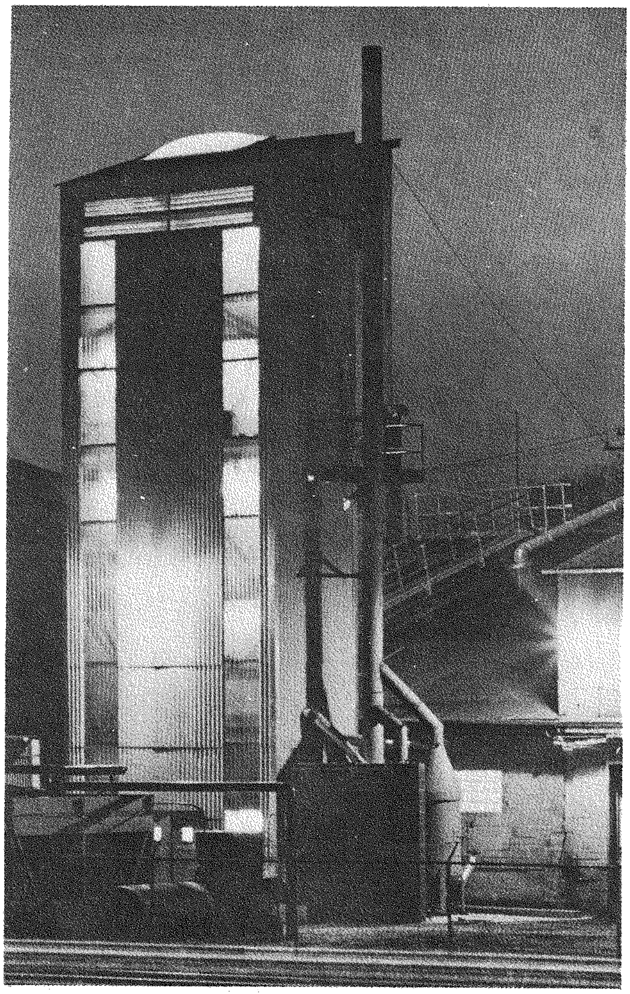
Total Funding: $44,351,586
ERDA: $32,692,102
Industry: $11,659,484

INTRODUCTION

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

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

Ultimately, the pilot plant will be constructed and operated to provide data for further evaluation of the [illegible]ocess and design of a full-size commercial plant. The [illegible]erall program plan is shown in Figure III-1.



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 III-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

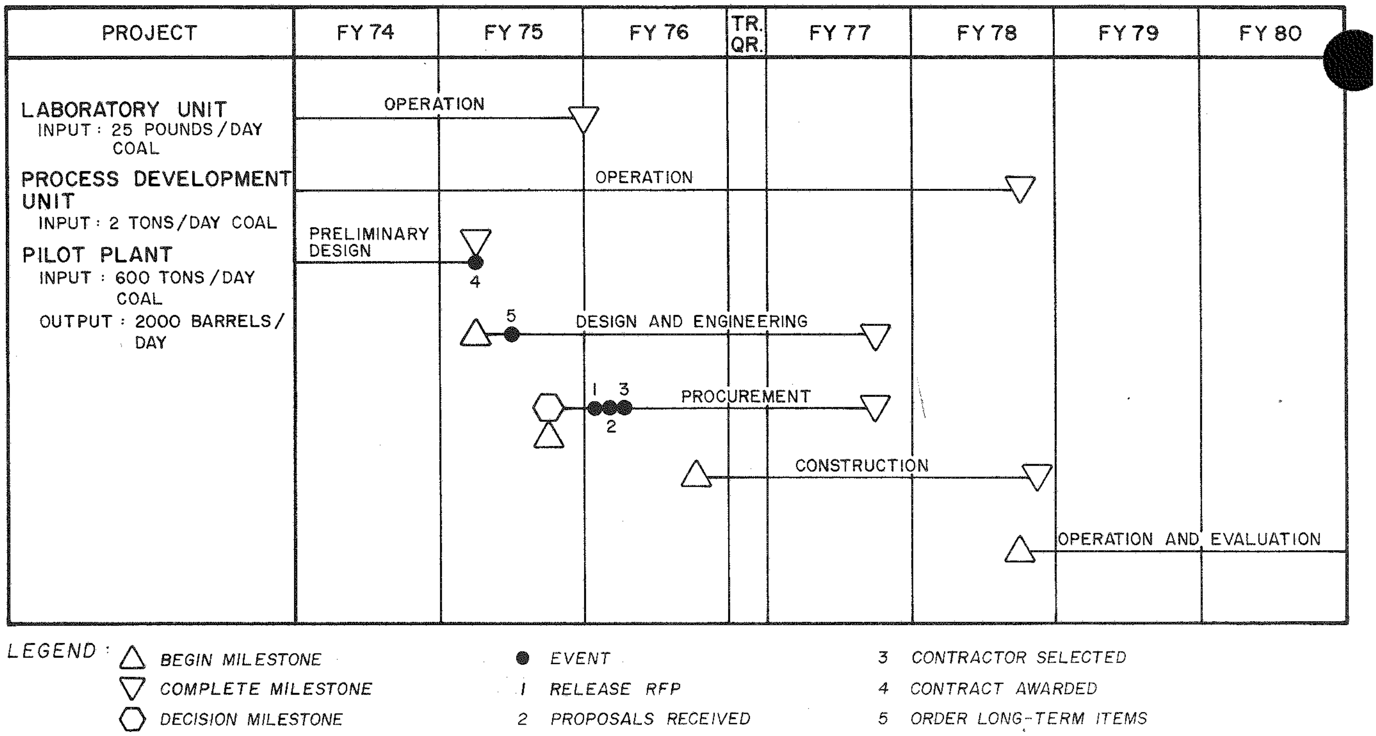


Figure III-1. H-COAL PROGRAM SCHEDULE

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 product, 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 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 as major products clean fuel gas and low-sulfur residual oil, the temperature and pressure in the ebullated-bed reactor are lowered, and less hydrogen is required.

Before the H-Coal process can become commercially and economically competitive, an adequate supply of hydrogen must be generated from the process itself. The H-Coal process requires between 14,000 and 20,000 standard cubic feet of hydrogen for each ton of coal processed, depending on the type of oil produced. In the pilot plant some of the hydrogen required has been obtained from the gas produced by the ebullated-bed reactor. Additional hydrogen has been purchased to

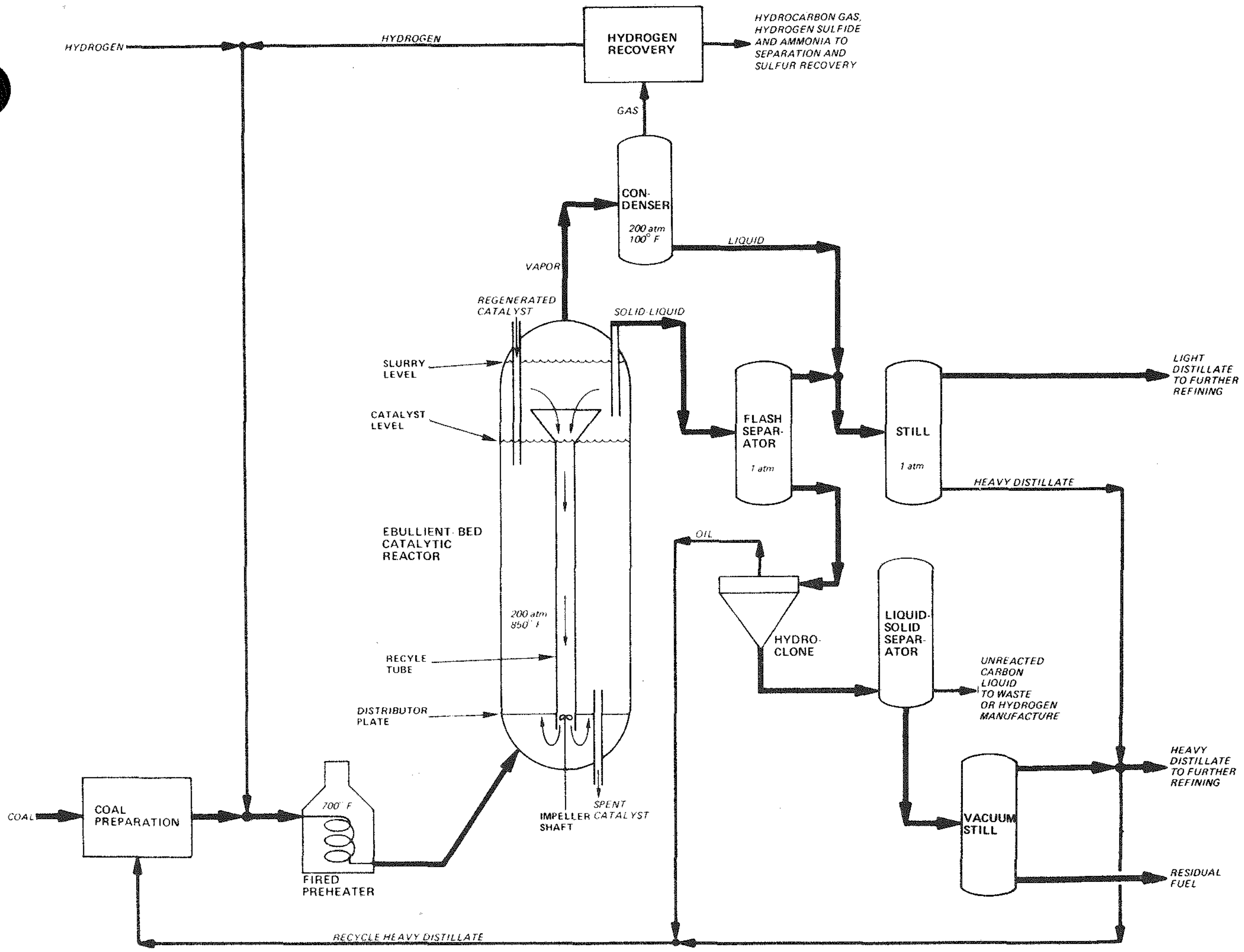


Figure III-2. H-COAL PROCESS SCHEMATIC

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 so that the liquids can be recycled for slurrying the coal. HRI has also been investigating other methods of separating solids from liquids, such as magnetic separation, filtration, centrifugation, and solvent precipitation. Magnetic separation has shown only limited effectiveness. Separation of the solids by filtration has not been conclusive, although filtration rates of 150 pounds per hour per square foot and relatively dry filter cakes have been achieved by using continuous drum pressure filters. Solvent precipitation appears to be the method having the most potential for commercial operation.

HISTORY OF THE PROJECT

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

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

Based on the data obtained from the bench-scale and process development units, design and engineering of the pilot plant began under the current contract 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.

The laboratory research in 1974 concentrated on bench-scale operation, liquid-solid separation, and process development unit modifications. A number of bench-scale runs were conducted to test regenerated, high-density, and aged catalysts. Tests were also conducted to investigate the mechanism of catalyst deactivation and the effects of low hydrogen partial pressure and high gas velocities between the bench-scale and process development unit. The process development unit wa[illegible] modified to ensure effective and safe operation, but the unit was not operated until 1975.

Pilot plant design and engineering, as well as laboratory research, was conducted during 1975. During that time the decision was made to construct the pilot facility adjacent to an Ashland Oil, Inc. refinery in Catlettsburg, Kentucky. Except for the steam supply, this arrangement would allow the utility system of the pilot plant to be shared with that of the refinery. All process descriptions, equipment specifications, and planning drawings were completed for the coal preparation section. Work in the hydrogenation area involved the modification and rearrangement of major equipment to conform to the physical characteristics of the new plant site. Preliminary process designs were also completed for the solid-liquid separation techniques of filtration, centrifugation, and solvent precipitation.

Laboratory research during 1975 concentrated on catalyst performance, hydroclone performance, and the production of low-sulfur fuel oil. Laboratory research was also conducted on the three solid-liquid separation techniques mentioned above.

PROGRESS DURING OCTOBER-DECEMBER 1976

The last quarter of the year was a period of relative inactivity on the process development unit (PDU), as final reports were being prepared. Reports were issued on costs, engineering, and laboratory research. Efforts will continue during 1977 under a new contract, EF-77-C-01-2547. Engineering functions will be conducted under a separate contract with Ashland Oil.

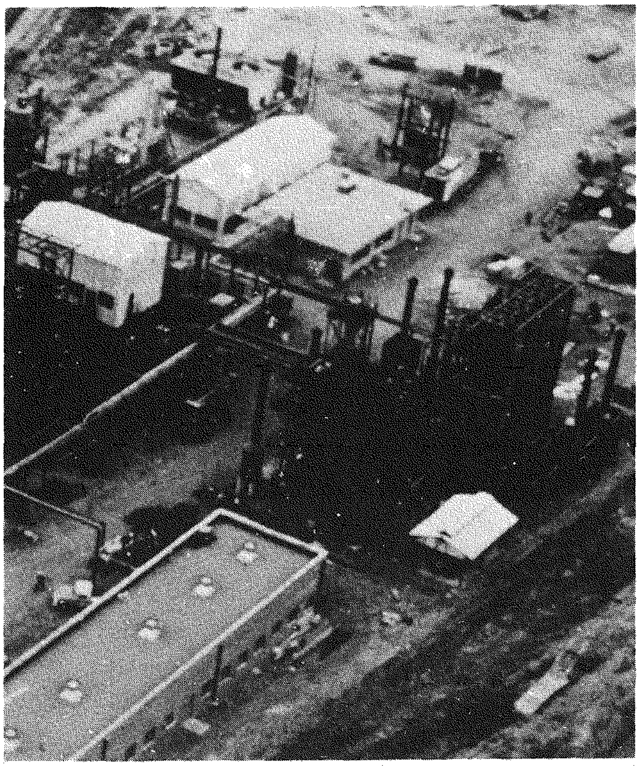
IV. CRESAP TEST FACILITY

FLUOR ENGINEERS AND CONSTRUCTORS, INC.
LOS ANGELES, CALIFORNIA

Project Site: *Cresap, West Virginia*

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

Total Funding: $45,699,780
ERDA: $43,042,140
Industry: $2,657,640



INTRODUCTION

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

PROCESS DESCRIPTION

In this process, shown schematically in Figure IV-2, coal is converted to low-sulfur liquid fuel. The coal is first crushed to 100-mesh and then combined with aromatic solvent in a mixer. The coal-solvent slurry is then pressurized and preheated. The slurry passes to an extractor where coal dissolution occurs. The product stream is then treated in a liquid-solid separation unit where the coal extract and solvent are removed, leaving a thickened slurry. This stream is sent to a carbonizer where the material is converted to char, oil, and gas. The oil is recycled to the liquid-solid separation unit and the char and gas are recovered.

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

The coal extract and solvent leaving the liquid-solid separation unit are heated and fed into a flash still where

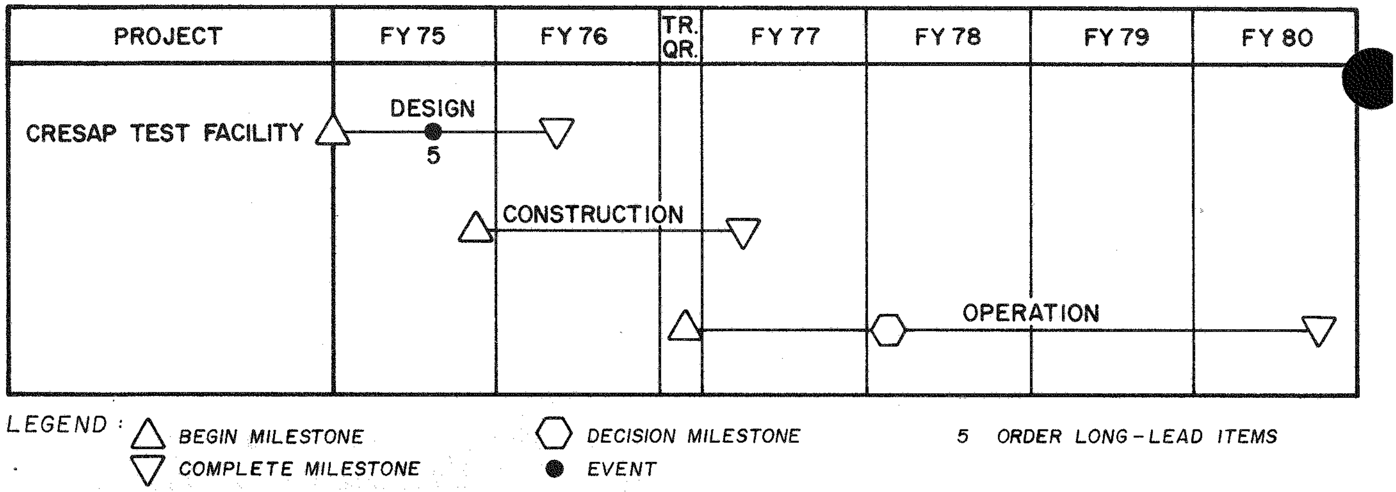


Figure IV-1. CRESAP TEST FACILITY PROGRAM SCHEDULE

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

HISTORY OF THE PROJECT

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

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

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

By the end of the quarter refurbishing activities were approximately 99 percent complete. The engineering and design work for the testing phase of the project was 79 percent complete and procurement was approximately 86 percent complete. Construction for Phase I of the test program was also finished during the quarter. Air leaks were repaired in the coal feed preparation equipment and the low temperature carbonization unit was checked out with ethylene glycol solutions in the liquid systems. Staffing was also nearly completed by the end of the quarter.

The results of 26 solvent de-ashing tests were evaluated during October and November. Continuous bench-scale extraction/settling tests were conducted in December and the results of the first four runs were evaluated. Studies also continued on the solids-liquids mechanical separation systems. The Krauss-Moffei and Chemapec Funda filters were delivered and data were compiled on the rotary drum, horizontal leaf, and crossflow filters.

Refurbishing and Start-up

Refurbishing activities at the Cresap Test Facility were approximately 99 percent complete by the end of the quarter. Only three process units were still to be completed by January 14, 1977. The engineering and design work for the testing phase of the project was 79

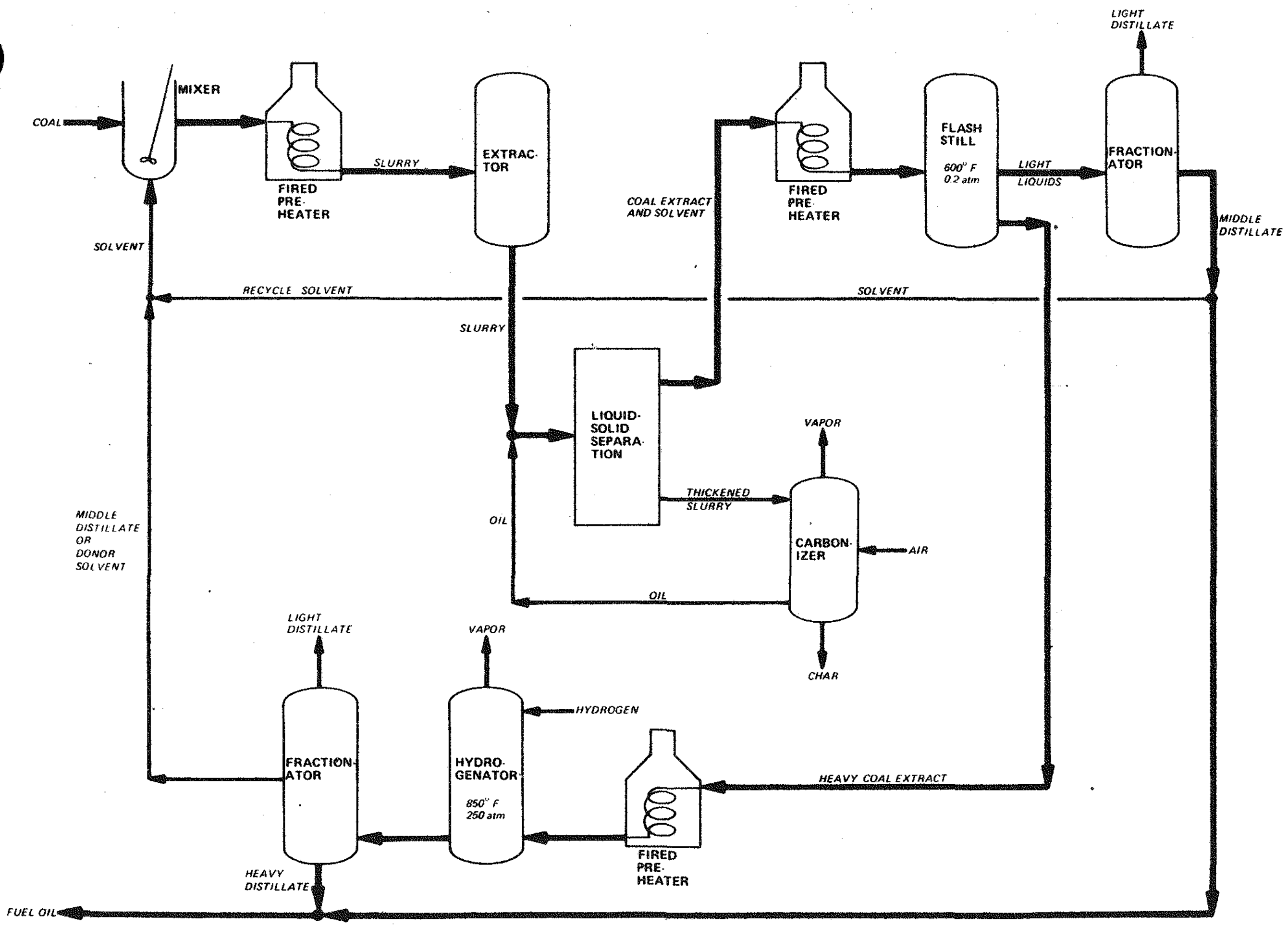


Figure IV-2. LIQUID FUEL FROM COAL PROCESS SCHEMATIC

percent complete by the end of the quarter, procurement was 86 percent complete, and construction for Phase I of the test program was complete.

The development of start-up plans continued, but operations were severely hampered by unusually cold weather. During the period, care/custody/control of the hydrogen generation unit was taken and line flushing was completed. Corrections were made to the coal feed preparation unit to eliminate air leaks. Some adjustment in procedures is also indicated to provide assurance that startup oxygen levels will be below the required 15 percent. The low temperature carbonization unit was in mechanical check out with ethylene glycol solutions being used in the liquid systems.

Staffing continued toward completion, with the only positions left to be filled being one process engineer, a test engineer, a plant inspector, and an instrument technician.

Test Program

One of the major process areas of investigation in the laboratory test program being conducted by the Conoco Coal Development Company (CCDC) was that of solvent de-ashing.

The objective of the test program was to determine the process design for solids-liquids separation using the solvent de-ashing concept developed by CCDC with verification on the bench-scale basis.

The results of 26 batch autoclave settling tests were evaluated during October and November. The tests were part of the SRC batch program developed to determine the settling rates for eight anti-solvents. The batch program was designed to guide the continuous bench-scale Pamco work.

During December a continuous bench-scale extraction/settling program was initiated and the results of the first four runs were evaluated. The antisolvents used in the batch autoclave programs were analyzed for Kauri-Butanol values and Fluorescence Indicator Adsorption (FIA). The antisolvent efficiency expressed as initial settling rate was correlated with Kauri-Butanol and FIA. Results indicated that FIA analysis was not as good a characterization of antisolvent efficiency as Kauri-Butanol values.

Studies dealing with solids-liquids mechanical separation were designed to:

- Determine maximum filtration rates with various candidate filtration equipment.
- Evaluate mechanical design, configuration, and reliability.
- Analyze the operations economics of the various filtration schemes with various coal liquefaction process streams.

During the quarter the Krauss-Moffei and Chemapec Funda filters were delivered and set in place. All drawings and documents related to the procurement and installation of the rotary drum, horizontal leaf and crossflow filters were collected, compiled, and reviewed.

Work also continued during the quarter for improved design of the following items:

- Pumps
- CSF process extractor
- Flow meters
- Control, letdown valves, and expansion chambers
- Valves and piping
- Corrosion and erosion prevention
- High pressure closures
- Vessels
- Heaters
- Heat exchangers
- Process definition and development

V. CLEAN COKE PROCESS

USS ENGINEERS AND CONSULTANTS, INC.
UNITED STATES STEEL CORPORATION

PITTSBURGH, PENNSYLVANIA

Plant Site: Pittsburgh, Pennsylvania

Contract No.: E(49-18)-1220

Total Funding: $11,792,329
ERDA: $8,254,630
Industry: $3,537,699

INTRODUCTION

The Clean Coke process is eing developed by USS Engineers and Consultants, Inc., a subsidiary of United States Steel Corporation, under the sponsorship of ERDA. The contract was initiated in 1972 under the auspices of the Office of Coal Research (OCR, now a part of ERDA). The objective of the project is to design a pilot plant 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 V-1.

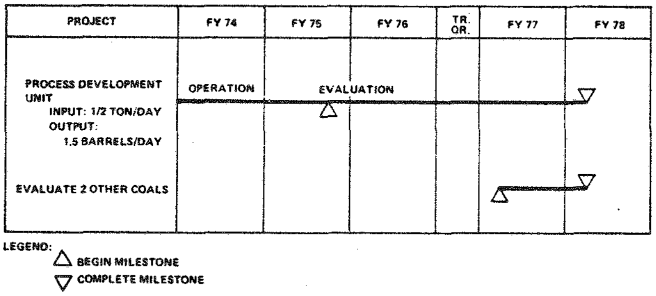
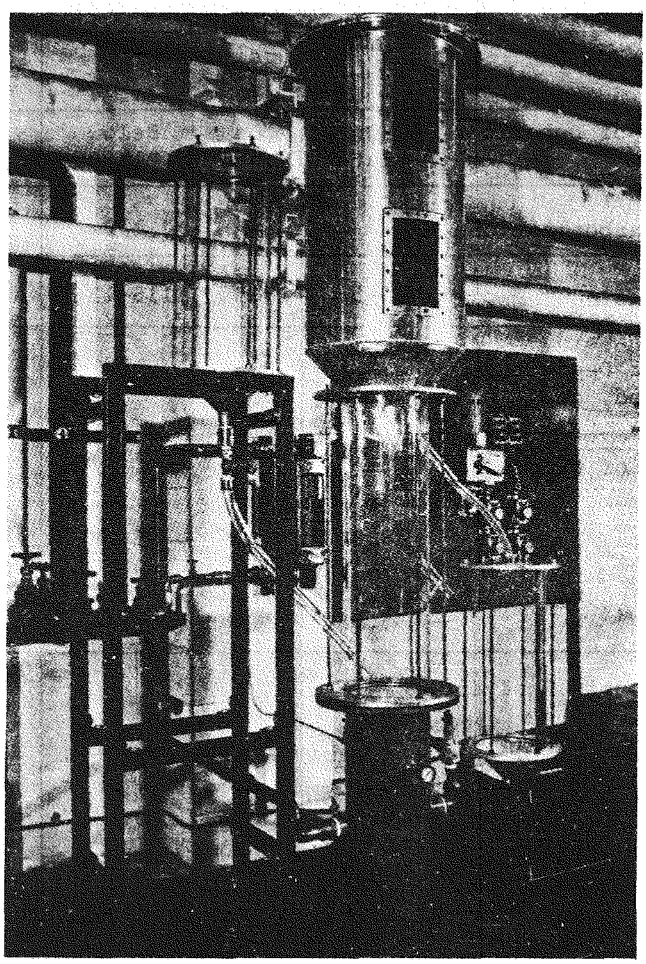


Figure V-1. CLEAN COKE PROCESS SCHEDULE



PROCESS DESCRIPTION

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

The feed coal is first pulverized and classified into two portions. One portion is sent to the carbonizer.

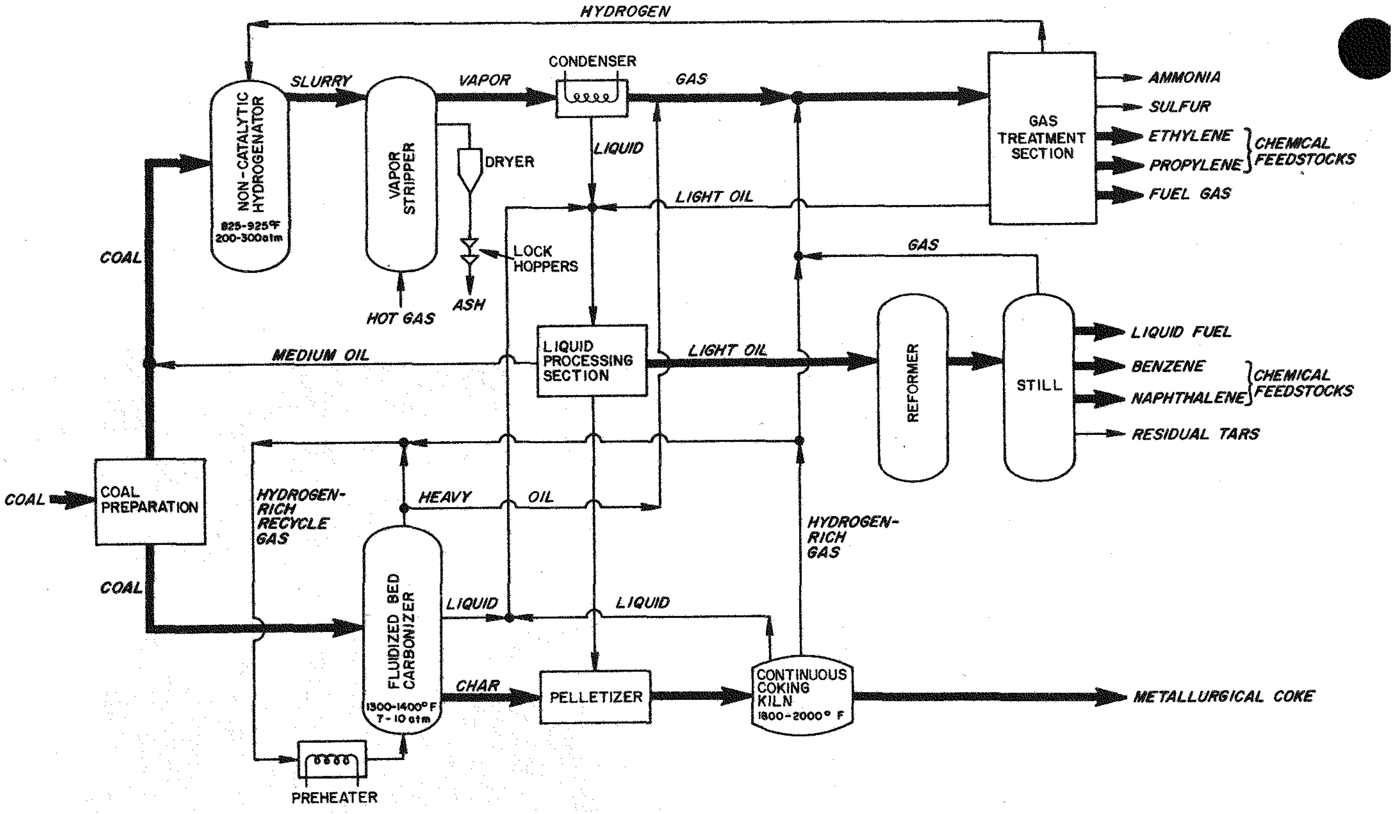


Figure V-2. CLEAN COKE PROCESS SCHEMATIC

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

The other portion of the coal is fed to the hydrogenation unit where it is slurried with a medium-weight oil recycled from the liquid processing section. This slurry is reacted with hydrogen recovered from the gas treatment section. The non-catalytic hydrogenation process is conducted at approximately 900° F and 270 atmospheres.

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

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

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

Two major advantages of the Clean Coke process design are that no hydrogen is produced for the liquefaction portion and that metallurgical-grade coke is a product from high-sulfur, high-ash coal.

HISTORY OF THE PROJECT

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

Under contract to ERDA since 1972, U.S. Steel is operating the process development units. The carbonizer is currently operating and the variables affecting carbonization are being tested. Initial testing has begun on the hydrogenation process development unit.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

Work during the quarter consisted of process development unit (PDU) tests at 1400°, 1450°, and 820° F. Total PDU operating time to date is 3,667 hours. This includes 2,498 hours at 1250° to 1450° F and 1,169 hours at 800° to 840° F.

Bench tests were made to develop treatments for rendering West Kentucky and Pittsburg seam coals nonagglomerating during pyrolysis. The flash vaporizer of the binder-preparation PDU was operated to process liquid blowdown from the carbonization PDU. A new feed heater was installed for the flash vaporizer. A new feed heater is also being designed for the continuous distillation column of the binder-preparation PDU.

In the hydrogenation area, two coal-conversion runs were made. The catalytic hydrogenation unit was operated to supply a quantity of slurry oil for an extended coal-conversion test in January.

Carbonization Process Development Unit

Work during the first two weeks of the quarter included general maintenance on the carbonization PDU and oxidation of 5,000 pounds of Illinois coal as carbonization feed. A ten-day, 1450° F carbonization PDU run was completed in which 1,511 pounds of char, containing 0.3 weight percent sulfur, was produced in 186 hours of operation. System pressure was 165 psia, and residence time was 104 minutes.

Tests at 1450° F indicated that char yield and concentrations of hydrogen and sulfur remaining in the char product decreased with increased temperature, and the volume of gas produced, as well as the hydrogen content of the gas, increased with temperature. Problems developed during the run in the main recycle-gas heater and wash oil recirculating pump.

An 820° F carbonization run was completed in November to confirm the material balance data obtained in a previous low temperature operation. During the run, 3,240 pounds of oxidized Illinois coal was processed. The runs were made with approximately the same operating conditions, and resulted in only slight variations in the analyses of the semichar product and recycle gas.

A 1400° F carbonization PDU run was completed in December using as feed the char previously produced in the carbonization PDU at a bed temperature of 1400° F with 50 ppm hydrogen sulfide in the recycle gas. The operation of a deep commercial fluid bed was simulated by using hydrogen sulfide concentrations of 1200 ppm in the fluidizing gas. Results show that though desulfurization may not be as efficient, an acceptable sulfur level can be obtained.

Carbonization Bench-Scale Studies

Carbonization studies to date have been made with Illinois No. 6 seam coal. As tests scheduled in 1977 will use Western Kentucky No. 9 seam and Pittsburgh seam coal, samples of both coals were acquired for testing in the 1.25-inch continuous bench-scale carbonizer to determine optimum processing parameters, prior to processing the coals in the three-inch bench unit or the PDU.

Binder-Preparation Process Development Unit

Maintenance work was completed on the binder-preparation PDU, and a bypass flush system was installed to enable the feed pump to be flushed at the end of each day. The flash vaporizer was operated to process liquid blowdown from high-temperature carbonization PDU runs. When the runs were completed, the feed heater was removed because of poor heat transfer and a new one installed. Work also continued on developing an operating procedure for the continuous distillation column.

Hydrogenation Process Development Unit

The heating scheme for the coal-conversion equipment of the hydrogenation PDU was modified to prevent overtaxing the slurry preheater. Coal-conversion runs were made with slurry oils containing 7.97 and 7.73 percent hydrogen, about one percent higher than in the oils used in previous runs. Analyses indicated that conversion of coal to oil was as high as 78 percent, compared with 58 percent conversion in a previous run with slurry oil containing 6.95 percent.

A coal-conversion run with raw coal was successfully completed in the PDU in November. Raw Illinois coal, pulverized to minus 100-mesh, was used instead of the washed Illinois coal used in previous tests.

An extended run of the hydrogenation PDU is planned. In order to produce sufficient slurry oil for the extended run, the catalytic hydrogenation unit was operated continuously during the last part of the quarter.

VI. LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

ROCKETDYNE DIVISION
ROCKWELL INTERNATIONAL CORPORATION
CANOGA PARK, CALIFORNIA

Plant Site: Canoga Park, California

Contract No.: E(49-18)-2044

Total Funding: $994,375
(100% ERDA)

INTRODUCTION

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

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

Upon completion of Phase I, the results of the cold-flow tests were submitted to ERDA for a decision on proceeding with Phase II. Upon Phase II authorization, small-scale reactor testing was initiated to evaluate the effects of reactor configuration and operating conditions. Based on these results, a larger reactor, with diverse injection methods and a coal flow rate of approximately one ton per hour, will be built and tested. Test results and analyses will be evaluated to determine the feasibility of this process. The schedule for the development of this process is shown in Figure VI-1.

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

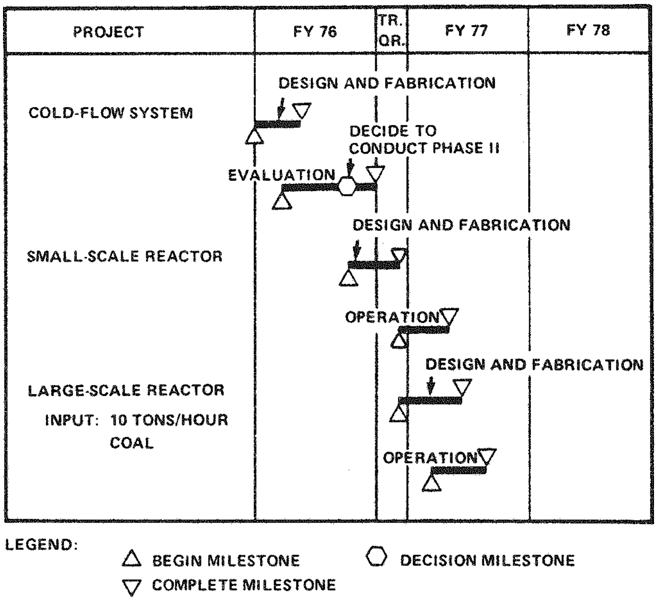


Figure VI-1. PROGRAM SCHEDULE FOR LIQUEFACTION OF COAL BY DIRECT HYDROGENATION

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 VI-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 atm and a temperature of approximately 1800° F.

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

HISTORY OF THE PROJECT

Development of a process for the liquefaction of coal by direct hydrogenation was started in August 1975. Work to date has focused on a series of tests designed to determine the most effective means of transporting coal to the point of injection into the reactor. These tests were conducted in both a transparent feeder mechanism and a cold-flow unit. Design of a one-ton-per-hour reactor also began during 1975.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

During the quarter, final testing of the ¼-ton-per-hour system was completed. Based on preliminary results, a reactor 36 inches long and 0.995 inch in

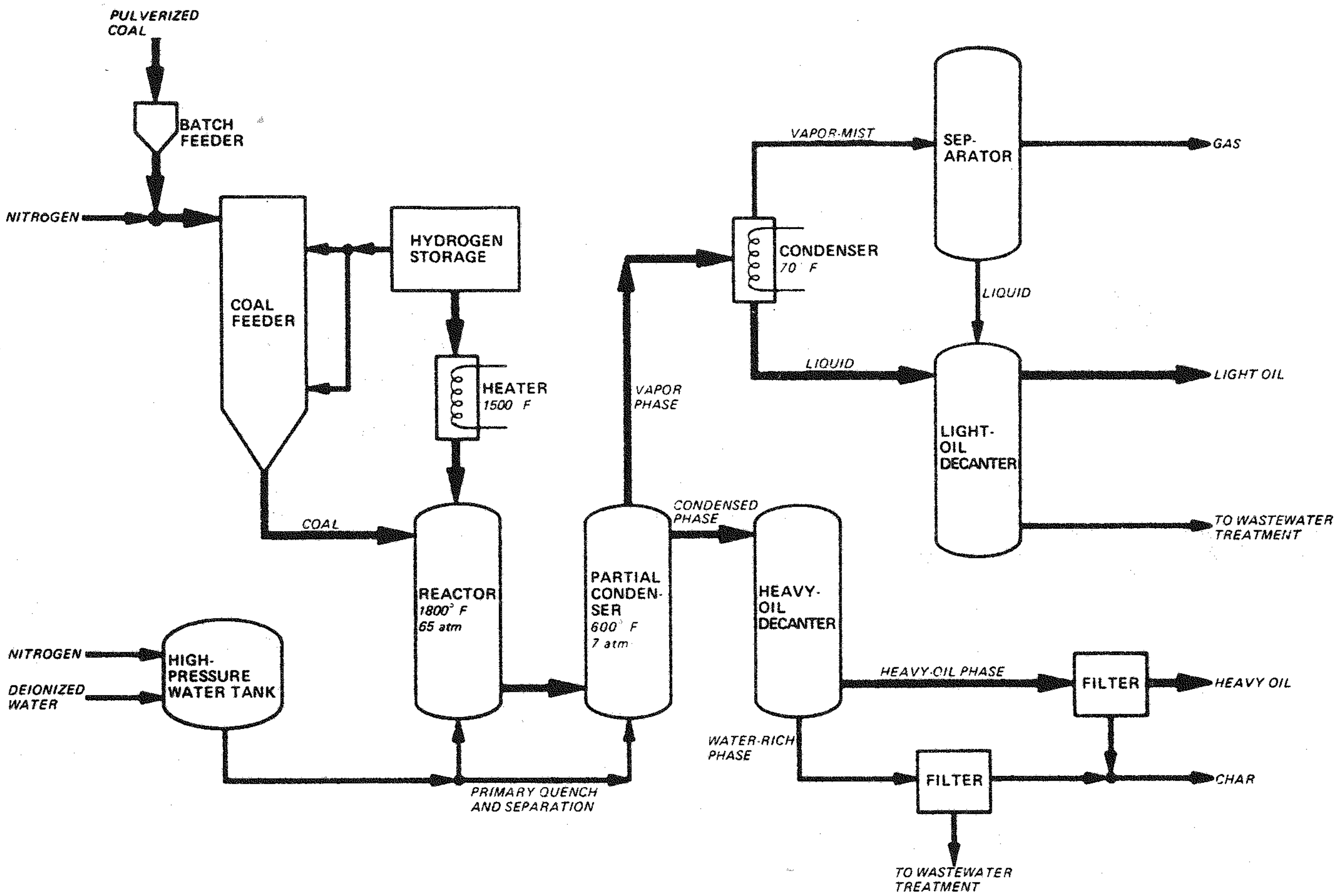


Figure VI-2. SCHEMATIC OF LIQUEFACTION OF COAL BY DIRECT HYDROGENATION diameter was used with the four-on-one injector. Oxygen was added in the preburner to obtain the desired temperatures. Results from this test were consistent with the trends shown by previous results.

Design and fabrication of the hardware for the one-ton-per-hour system was completed. Construction of the facility was completed in December, as were the initial preparations for testing.

Discussion of Activities

Based on preliminary results, a reactor 36 inches long and 0.995 inch in diameter was selected and used with the four-on-one injector for the final tests of the ¼-ton-per-hour system. Oxygen was added in the preburner to obtain the desired temperatures.

The test results indicated greater overall conversion with increasing residence time and velocity. The influence of these parameters on liquid yield is less certain, but it does appear that liquid yield is proportional to residence and velocity. Additional testing at the one-ton-per-hour level will be required to further define these effects.

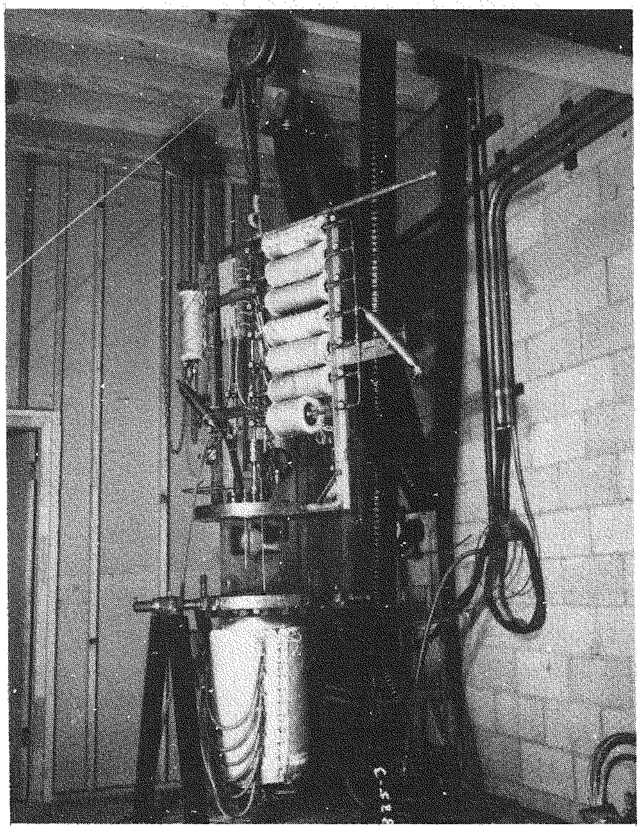
Design and fabrication of the reactor assembly for the initial one-ton-per-hour system was completed. The reactor was installed in a pressure vessel made from 14-inch diameter stainless steel pipe. The assembly includes a preburner to facilitate the addition of oxygen to the hydrogen stream prior to the main injector. A five-element oxygen injector is used in this preburner. A single-element coal/hydrogen injector is used. Oxygen injection orifices are also included to allow oxygen addition to the reactor if desired. Preparation of the one-ton-per-hour system for initial tests was completed in December.

A few exploratory cold-flow tests were conducted to evaluate a char pressure-letdown concept. These tests were made to evaluate the possibility of removing the char through a sonic orifice. A very dense flow of pulverized coal was readily achieved through a ⅛-inch orifice with a transport gas requirement corresponding to the gas in the interstices of the particles, as found in the earlier subsonic flows. A somewhat larger orifice was required to achieve sonic flow with char. A rough screening of the char showed that approximately ten weight percent of the char had particle sizes greater than ¼-inch. A ⅜-inch orifice was used to achieve sonic flow. This technique for char removal appears feasible and this is significant because of the possible applications of the system to a variety of coal conversion processes.

VII. FILTRATION PROCESS AND EQUIPMENT STUDIES

JOHNS-MANVILLE SALES CORPORATION
DENVER, COLORADO

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



INTRODUCTION

Under contract to ERDA, Johns-Manville Sales Corporation is conducting research to improve solid-liquid separation, a critical step in all coal liquefaction processes. The first phase of this program was initiated in May 1975. Johns-Manville is studying rotary drum pressurized precoat filtration. The objective of the research program is 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, reduce filter aid usage, and demonstrate the results on a pilot-plant scale.

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 the product flow from an existing liquefaction pilot plant. Phase I is being funded entirely by ERDA and Phase II is being supported by the Electric Power Research Institute (EPRI) in cooperation with the ERDA effort. One of the criteria established for a viable system is the capability of filtering in excess of 20 gallons of solid-liquid mixture per square foot per hour. Data obtained from operation of the pilot scale filtration unit will be used to design filters for commercial plants.

PROGRAM DESCRIPTION

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 atm. 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 process liquids are to be used: solvent-refined coal (SRC) from both the Wilsonville and Tacoma pilot plants and Synthoil boiler fuel supplied by the Pittsburg Energy Research Center.

Upon completion of these tests, the apparatus may be moved to a Pittsburg and Midway (P&M) facility at DuPont, Washington. Here it would be used to establish the relationships between test results obtained on fresh filter feed and those on oil products that have been cooled and tested in Denver. 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 ERDA-sponsored coal liquefaction plants. Johns-Manville is also responsible for assisting in the selection of a suitable precoat filter for the Cresap Test Facility in Cresap, West Virginia. This investigation will include (1) the feasibility of modifying the existing filtration equipment to accommodate the experimental work and (2) assistance in the start-up and establishment of operating conditions for the filter system selected.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

The filtration characteristics of Tacoma Filter Feed (TFF), uncentrifuged Synthoil product, and Wilsonville Filter Feedstock were studied in 44 runs made in the laboratory-scale rotary leaf, pressure precoat filter during this quarter.

Dependence of the TFF filtration rate upon blade advance was studied in much greater detail than in previous investigations and additional detail on pressure dependence was obtained.

The filtration characteristics of the relatively viscous Synthoil product were found to be somewhat different from the TFF stock. The Wlsonville Filter Feed was somewhat easier to filter than TFF.

Tests begun in December on hot, fresh samples of Tacoma Filter Feedstock. These tests indicated filtration rates equivalent to those obtained from previous samples, but with a different knife blade advance dependence.

Filtration Tests

Filtration characteristics of Tacoma Filter Feed (TFF) and uncentrifuged Synthoil product were studied in twelve runs made in the laboratory-scale rotary leaf, pressure precoat filter in October. The dependence of the TFF filtration rate upon blade advance was studied in much greater detail than in earlier research, and additional detail on pressure differential dependence was obtained. Maximum rates near 400 pounds per hour per square foot at 40 percent submergence were attained at 600° F with five-second immersions, pressure differentials of 35 to 40 psi, and large blade advances.

The filtration characteristics of the relatively viscous Synthoil product were found to be somewhat different from those of the TFF. It appeared that both pressure differential dependence and temperature dependence were greater for Synthoil than for the TFF. Blade advance dependence was greater at low pressure differential, but smaller at high pressure differential, as compared with TFF. For the Synthoil, rates as high as 269 pounds per hour per square foot at 40 percent submergence were obtained with acceptable filtrate quality.

In November and December, 32 experimental runs were conducted on Wilsonville filter feedstock as well as TFF. Filtration rate dependence of TFF upon blade advance, using Celite AF6 and AF7 as the precoat materials, was determined. Results were inconclusive due to a breakout of precoat material, however, there appeared to be no overall difference in filtration rates between AF6 and AF7. Maximum rates of 410 to 466 pounds per hour per square foot at 40 percent submergence were obtained.

Six of the runs made in December were on Wilsonville Filter Feed using Celite AF6 to confirm the results of tests conducted in November. In general, it was found that the filtration rate increased approximately two-fold upon increasing blade advance from two mils per cycle to ten mils per cycle. The filtration rate does not increase significantly upon increasing the pressure differential from 20 to 40 psi. There was some indication that the filtration rate may even be reduced slightly at the increased pressure differential. There appeared to be a slight reduction in filtration rate when the material was agitated to the maximum extent possible.

The Wilsonville Filter Feed appears to be somewhat easier to filter than TFF. A rate in excess of 400 pounds per hour per square foot at 40 percent submergence was obtained both in November and December.

Hot, fresh samples of Tacoma Filter Feed were originally scheduled to be tested at Ft. Lewis, Washington. Due to the time and expense of shipping the laboratory equipment to Ft. Lewis, arrangements were made to ship the hot samples to the Johns-Manville facilities in Denver. Testing of the hot samples began in December. The sample was obtained at 245° F and was not allowed to cool below 200°F. Because the solvent refined coal (SRC) and solvent mixture tends to undergo some chemical and/or physical changes if maintained at elevated temperatures for extended periods of time, Ft. Lewis personnel stated that the sample should not be allowed to cool below 150° F, nor should it be held at temperatures near 600° F.

The confirmation tests on the hot, fresh sample of TFF indicated filtration rates equivalent to filtration rates obtained on samples of TFF previously tested, but with a different knife blade advance dependence.

VIII. CHEMICALS FROM COAL

THE DOW CHEMICAL COMPANY
MIDLAND, MICHIGAN

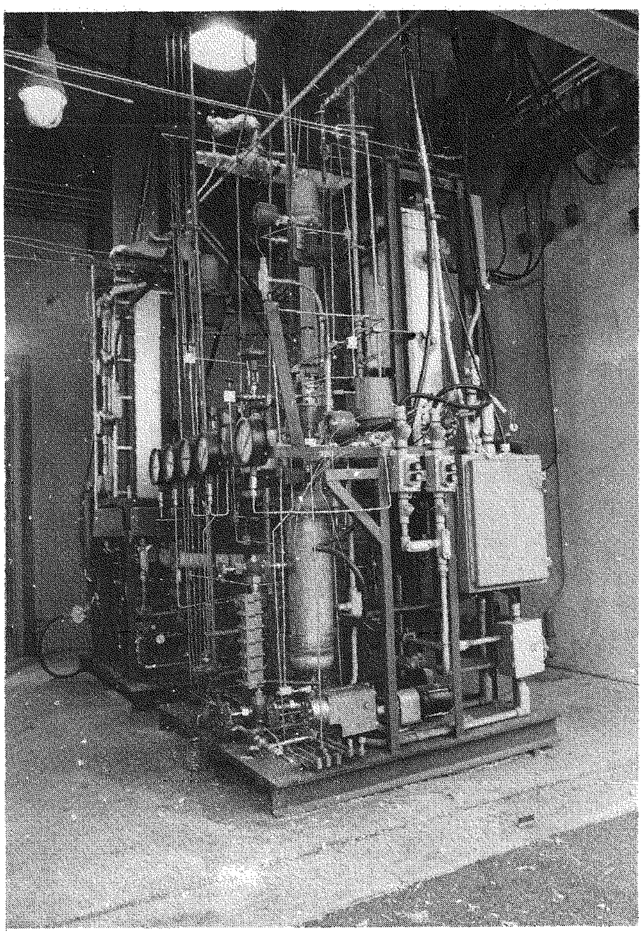
Project Site: Midland, Michigan
Contract No.: E(49-18)-1534
Total Funding: $342,896
(100% ERDA)

INTRODUCTION

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

PROGRAM DESCRIPTION

The liquids produced by the four processes are tested in standard petrochemical processing operations to determine the advantages and disadvantages of using coal-derived liquids for petrochemical feedstocks. The experimental work is being conducted in small units capable of operating unattended at pressures up to 204 atm., temperatures up to 1300° F, and feed rates from 25 to 400 milliliters per hour.



The processing sequence used for all of the samples is:

(1) Distillation into nominally three cuts- straight-run naphtha (350° F or less), middle distillate (350° F to 650° F), and heavy gas oil (650° F to 950° F).
(2) Hydrocracking of the middle-distillate and heavy gas oil to reduce molecular weight.

(3) Hydrotreating of the straight-run naptha and hydrocracked naptha.
(4) Reforming of the hydrotreated naphthas to maximize aromatics.

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

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

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

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

Hydrotreatment of the hydrocrackate naphtha from the SRC II product was completed this quarter. The hydrotreated product was extracted with hydrochloric acid to remove the pyridinic nitrogen present and dried over a molecular sieve to remove the water introduced with the acid. The naphtha was then reformed under the same conditions that were used on the other coal liquid hydrocrackate naphthas.

Hydrotreating experiments on the SRC II straight run naphthas were also completed this quarter. Hydrotreating this material proved to be very difficult and rapid deactivation of the catalyst occurred.

SRC Product Studies

Hydrotreating experiments on the SRC 11 hydrocrackate naphthas were completed this quarter. A final run was conducted at 2500 psig reactor pressure rather than the 1500 psig used in previous experiments. There was no improvement in the level of heteroatom removal as a result of the change in pressure. The most effective means of removing the resistant nitrogen in the material proved to be acid extraction.

The hydrotreated hydrocrackate naphtha was reformed over the same catalyst loading as the Synthoil naphtha. In the case of the feeds, the SRC II naphtha contained a much larger fraction of aromatics in the C_6-C_9 product than the hydrocrackate naphtha from either the COED, Syncrude, Synthoil, or H-Coal oil. The C_1-C_5 product yield was much lower for the SRC II reformate than for the others.

First and second pass hydrotreating experiments on the straight run naphtha were also completed this quarter. It was evident from the data presented that the naphtha contained some highly resistant species of sulfur heteroatoms which were not observed in the hydrocrackate naphtha. Deactivation of both cobalt molybdenum (HT-400) and mixed molybdenum (HDS-9A) catalysts occurred during the first pass experiments. These catalysts normally have superior desulfurization and denitrogenation activity with a high resistance to deactivation.

IX. R&D ENGINEERING SERVICES-HYDROCARBONIZATION

OAK RIDGE NATIONAL LABORATORY
OAK RIDGE, TENNESSEE

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

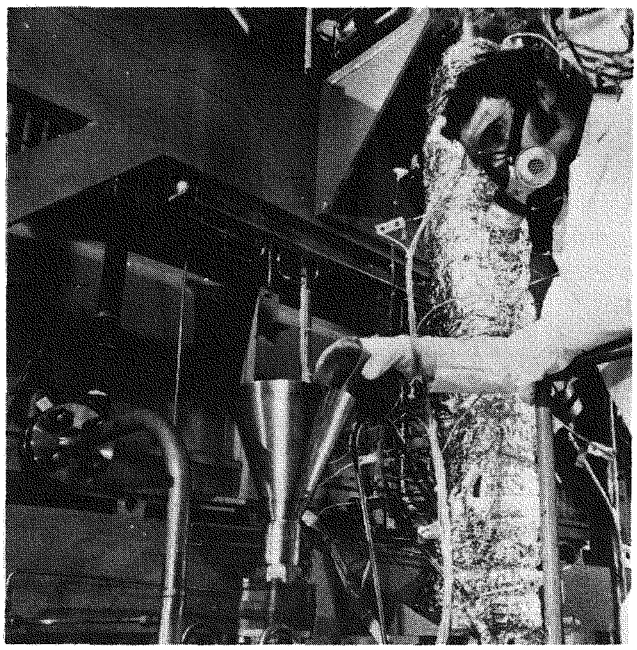
INTRODUCTION

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

PROCESS DESCRIPTION

Hydrocarbonization is an important type of coal liquefaction process which combines high-temperature, high-pressure, and fluidized-bed operation using hydrogen-rich gas for fluidization. The hydrocarbonization process can produce controlled yields of desulfurized char, liquid fuels, and substitute natural gas. The product char is 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 IX-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.

An intensive literature survey conducted during 1975 led to the preliminary conclusions that very little reaction takes place below 750° F and the yield of both oil and gas increases directly with temperature. A maximum oil yield was noted at 1,020° F to 1,110° F, however gas production increased proportional to temperature and therefore did not show an optimum over the temperature range studied. Two lucite mock-ups were constructed to study the mechanics of hydrocarboniza-

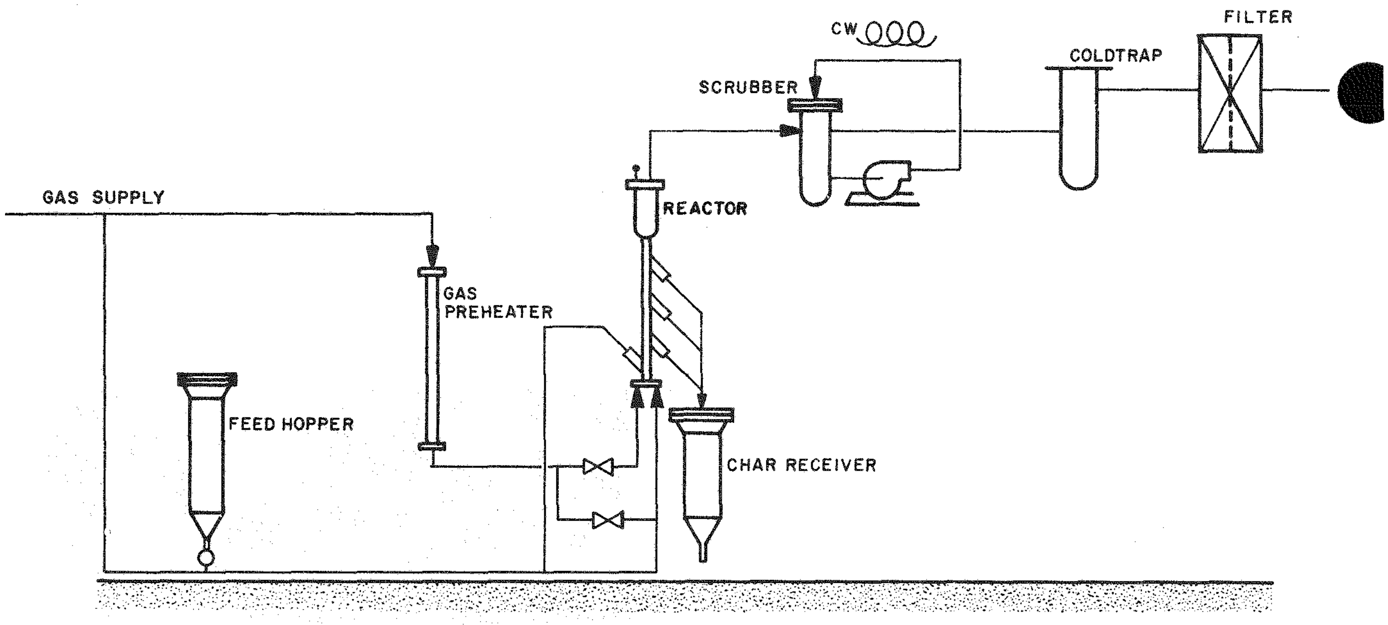


Figure IX-1. FLOW DIAGRAM OF BENCH-SCALE HYDROCARBONIZATION SYSTEM

tion processes and the ten-pound-per-hour bench-scale unit was built. The overall performance of this unit proved satisfactory.

PROGRESS DURING OCTOBER-DECEMBER 1976

Installation of equipment was completed in the solids handling facility at the beginning of the quarter. An inert gas purge system permitted grinding and sizing to be performed without exposure to the air.

Run HC-8 was completed in October with the 20-atm bench-scale hydrocarbonizer in a recirculating fluidized-bed mode. Runs HC-1 through HC-7 used Wyodak coal prepared at Morgantown Energy Research Center. The coal for Run HC-8 was prepared in the ORNL solids handling facility.

As particle size and flow properties are important in feeding coal by pneumatic transport, the larger particle size for Run HC-8 suggested that more gas was required to transport it. In Run HC-8, coal was fed at a rate of 13.6 pounds per hour for 6.2 hours at 800° F and 300 psig hydrogen pressure. In Run HC-9, Wyodak coal was fed at a rate of 6.8 pounds per hour for 4.8 hours at 950° F and 300 psig hydrogen pressure.

In Run HC-8, low liquid and gas yields resulted from the low temperature of 800° F, with the principal gas components being methane and carbon monoxide. Analyses of Run HC-9, at the higher temperature of 950° F, showed an increase in methane and carbon monoxide levels and a decrease in carbon dioxide content.

In Run HC-9, closure of the overall mass balance was poor due to the influx of water from the scrubber pump seal pot to the scrubber water reservoir. Operation of the bench-scale hydrocarbonizer has been impaired because of recurrent problems with the seals. Replacement seals are on order.

At the end of the quarter work began on an improved method of measuring bed recirculation rates by following a radioactively tagged particle in the moving bed.

X. R&D ENGINEERING SERVICES—CARBONIZATION OF RESIDUE

OAK RIDGE NATIONAL LABORATORY
OAK RIDGE, TENNESSEE

Contract No.: E(49-18)-1740
Total Funding: $325,000
(100% ERDA)

INTRODUCTION

The Oak Ridge National Laboratory (ORNL) is conducting several research and development projects in support of the increased utilization of coal as a source of clean energy. Under this contract, begun in November of 1975, ORNL is to:

- Review the experience with the low-temperature carbonizer at the Cresap Test Facility.
- Modify the existing atmospheric batch reactor to operate continuously on solids-laden residue from ERDA processes.
- Operate the above reactor with material from the H-Coal, Solvent Refined Coal (SRC), and Consol Synthetic Fuel (CSF) processes.

PROCESS DESCRIPTION

The process to be used by ORNL to evaluate the residual materials from the various liquefaction processes is shown schematically in Figure X-1. In the process, the residual materials will be converted to char by carbonization. The feedstock will be transported from the feed pot to the reactor by a stream of inert gas. Additional inert gas will be preheated and fed to the reactor for fluidization. The fluidized-bed reactor will be operated at temperatures up to 1,200° F. The char level in the reactor will be maintained by a char overflow pot nnected to the reactor by two overflow tubes. Inert gas passing up through the central draft tube will transfer the product char from the reactor to a cyclone where the gas and solid phases are separated. The gaseous effluent will be subsequently scrubbed and filtered prior to being discharged.

A literature review has been conducted and the bench-scale batch reactor was modified to convert it from batch feed to continuous feed with respect to char. In addition, a second feed system was designed, involving the mixing of the residue with toluene to form a slurry which can be injected into the reactor as a spray.

PROGRESS DURING OCTOBER-DECEMBER 1976

Based on the design used in ORNL's 20-atm hydrocarbonizer, a new rotary feeder for the residue carbonization experiments was fabricated and installed in October. Following installation of the feeder, two runs, RC-15 and RC-16, were conducted using a four-to-one ratio of Wyodak char to residue as feed and a reactor temperature of 1130° F. Run RC-15 was interrupted by plugged effluent lines and difficulty was encountered in maintaining a steady feed rate during Run RC-16. Material received from Run RC-16 was of a different texture than material recovered in previous runs.

Three residue carbonization runs were conducted during November, with equipment changed to facilitate operation. All three attempts were made with a four-to-one char residue feed mixture and resulted in tar prod-

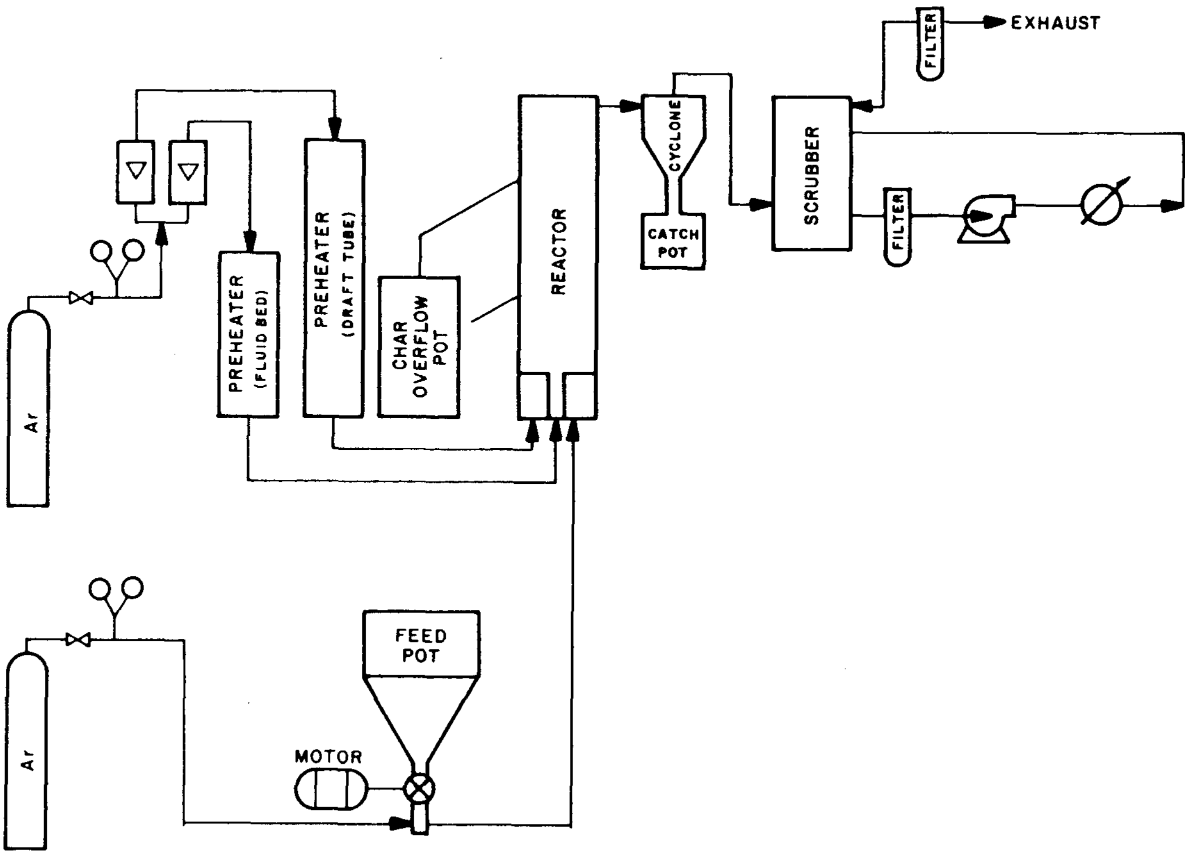


Figure X-1. FLOW DIAGRAM OF EVALUATION PROCESS

uct from the scrubber. Plugging of the feed nozzle and effluent lines caused interruptions in the experiments.

The ash content of the tars recovered from the scrubber in Run RC-16 and filter housing in Run RC-17 was lower than the level in the feed. The amount of volatile material in the tars was more concentrated than that in the feed.

In December, three residue carbonization runs were conducted using a feed mixture of four parts char to one part residue and with a methyl-naphthalene scrub liquid. The change from a water scrub liquid prevented plugging of the filter and subsequent aerosol emissions. Plugging of the feed line and the reactor to scrubber line continued to be a problem.

XI. ZINC HALIDE HYDROCRACKING PROCESS

CONOCO COAL DEVELOPMENT COMPANY
LIBERTY, PENNSYLVANIA

Project Site: Liberty, Pennsylvania
Contract No.: E(49-18)-1743
Total Funding: $6,450,335
ERDA: $5,650,335
Industry: $800,000

INTRODUCTION

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

Specifically, experimental studies will be performed involving both continuous bench-scale and process development unit work dealing with the investigation of zinc halide as a catalyst for the hydrogenation and hydrocracking of coal extract and subbituminous 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 program is shown in Figure XI-1.

PROCESS DESCRIPTION

The zinc halide hydrocracking process is designed to maximize the conversion of subbituminous 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.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

During the quarter, the bench-scale regenerator was temporarily converted for continuous tests of secondary recovery of zinc from the coal ash recovered in primary regeneration. Two test runs were conducted in October in continuation of the batch kinetics studies of secondary recovery of zinc chloride from the cyclone ash.

In November, a variable study with Colstrip subbituminouos coal was begun on the bench-scale hydrocracking unit. Five runs during the month included two blank runs with vehicle only, a test of the stability of heavy naphtha as vehicle, and two coal runs.

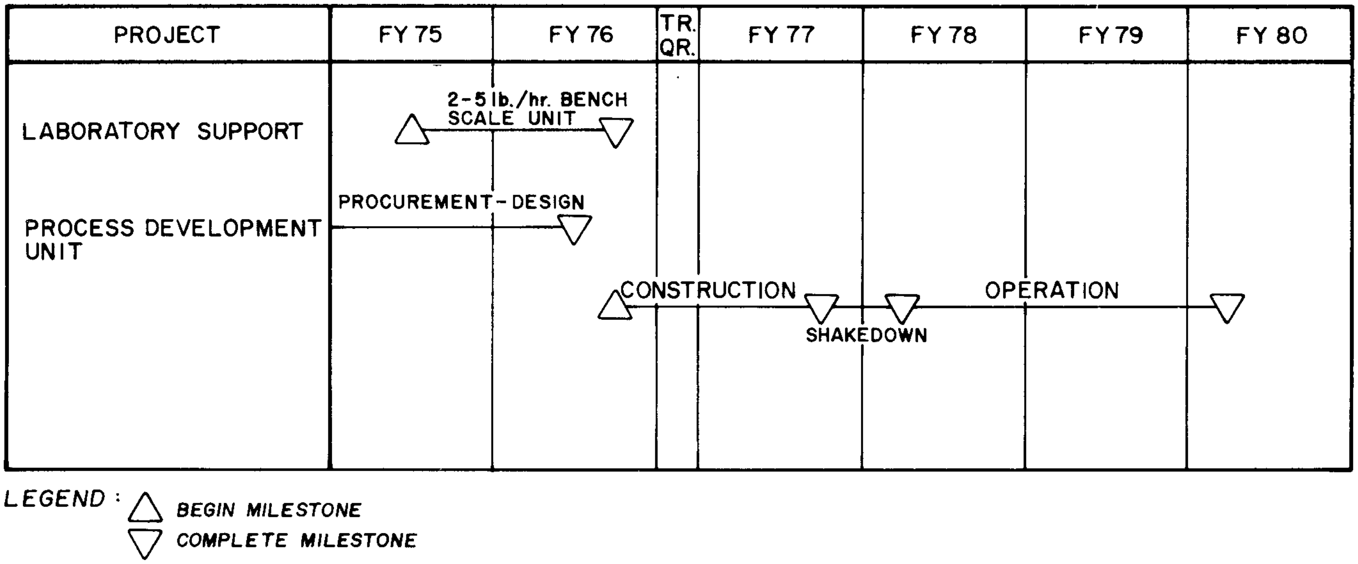


Figure XI-1. ZINC HALIDE HYDROPROCESSING PROJECT SCHEDULE

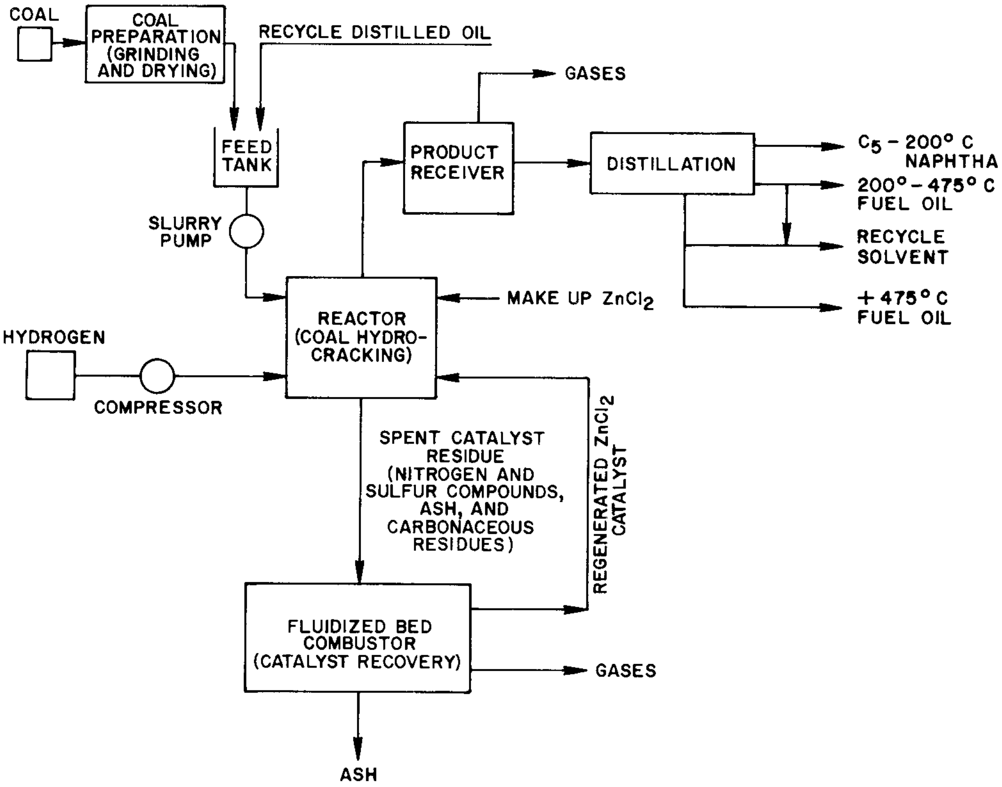


Figure XI-2. ZINC HALIDE HYDROCRACKING PROCESS FLOWSHEET

Two balance runs were completed in December. Supporting batch tests on process distillates to be used as recycle vehicle indicated that a middle distillate fuel oil and gasoline are likely to be produced from this coal.

Bench-Scale Studies

Material balance calculations were completed for five Colstrip coal hydrocracking runs, and coal-derived product yields were obtained. Conversion to 475° C^- products of up to 81 percent of the MAF coal were obtained at 775° F and 3500 psi total pressure.

A program was initiated to study the effect of operating conditions on coal hydrocracking kinetics. The bench-scale regenerator was modified for the continuous studies on the kinetics of secondary zinc recovered from cyclone ash. A stability test of spent catalyst showed that it could be stored for several weeks, either as a liquid or solid, and still be fluid enough to pump.

A series of runs to determine the effect of operating conditions on the liquefaction of Colstrip subbituminous coal were begun in November on the bench-scale hydrocracking unit. Two blank runs were conducted to define yields from the slurry vehicle at both severe and mild operating conditions. One run fed heavy naphtha from previous Colstrip liquefaction tests to establish the extent of hydrocracking which might be obtained if this portion were to be recycled as a vehicle for feeding coal. Two runs initiated operation with coal slurry.

A total of four runs were made in the bench-scale hydrocracker in December. Two runs were terminated late in their material balance periods by failure of the coal slurry feed pump. Difficulty was encountered due to high viscosity melts which were produced at lower conversion levels and mild conditions. A higher zinc chloride to coal ratio will be used in future runs at milder conditions of temperature and pressure.

Laboratory Studies

Two batch autoclave hydrocracking runs were completed in October. One run demonstrated that regenerated catalyst is as active as fresh catalyst. During the other run, heavy naphtha from the zinc chloride liquefaction of Colstrip coal was used as feedstock. Results of this test showed that there was only slight cracking of the material; and it, therefore, could be recycled as a coal slurry vehicle if heavier oils are not available.

Two supporting batch autoclave runs were made in December. The first run was made to test the reactivity of the 475° C^+ residue generated in the continuous hydrocracker to further hydrocracking with the zinc chloride catalyst. The second run was made to determine the reactivity of distillate products to further hydrocracking. The tests indicated that a middle distillate fuel oil as well as gasoline products would result from hydrocracking Colstrip coal slurried in recycle process solvent.

Process Development Unit

The preliminary operating manuals for the process development unit (PDU) were completed. Construction of the PDU continued, with the detailed engineering design progressing rapidly. Piping and electrical layouts within the building were nearly completed. All major equipment was on order and the mounting of vessels began.

XII. FLASH PYROLYSIS COAL LIQUEFACTION PROCESS

OCCIDENTAL RESEARCH CORPORATION
LA VERNE, CALIFORNIA

Plant Site:	La Verne, California
Contract No.:	E(49-18)-2244
Total Funding:	$3,780,000 *(100% ERDA)*

INTRODUCTION

The Occidental Research Corporation (ORC), a wholly-owned subsidiary of Occidental Petroleum Corporation, will evaluate the commercial potential of its flash pyrolysis coal liquefaction process under the sponsorship of ERDA. Figure XII-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.

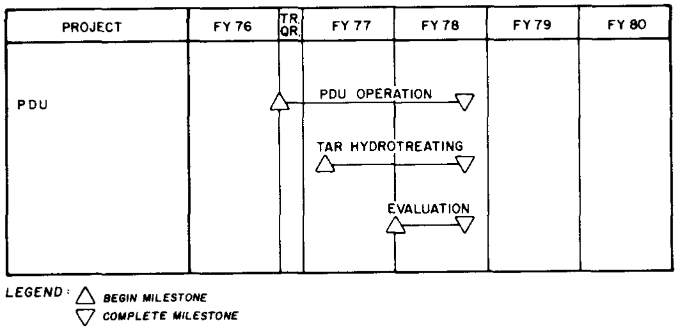


Figure XII-1. FLASH PYROLYSIS PROJECT SCHEDULE

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 XII-2 provides a schematic flow diagram of the process.

After the coal is extensively milled and screened, it is transported to the pyrolysis reactor by heated nitrogen at rates of up to 300 pounds per hour. In the reactor, the coal is mixed with recycled hot char which rapidly heats the coal to a pyrolysis temperature in the range of

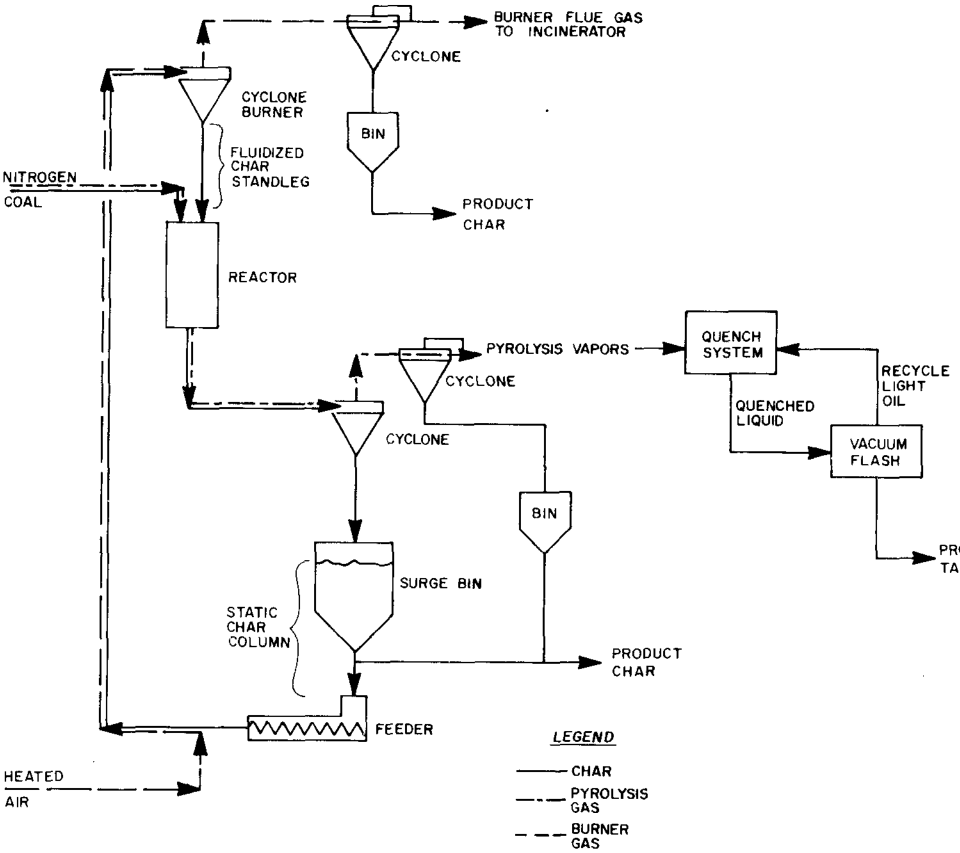


Figure XII-2. FLASH PYROLYSIS PROCESS FLOWSHEET

950° to 1350° F. The char circulation rate ranges from five to ten times that of coal.

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

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

HISTORY OF THE PROJECT

ORC constructed their three-ton-per-day PDU for development of other pyrolysis processes, and revised and improved the unit for use in this contract. 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 using the process.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

The second process development unit (PDU) shakedown run with subbituminous coal was completed in November. The new tar collection system and recent revisions made in the pyrolysis system were tested and operated without problems. Quenching, water/light oil separation, and the vacuum flash unit all functioned according to design.

The third and final PDU shakedown run in December completed the first major program of the contract. All of the objectives of the run were achieved. During the last seven days of the run, 16 tons of coal were processed at a rate of 2.4 tons per day. During one twelve-hour period, a rate of 3.5 tons per day was achieved.

Bench-scale runs in October confirmed the relatively high yield of tar which is characteristic of the flash pyrolysis process. Runs made in November indicated that tar yield is highest at about 1200° F, with residence time of 1.5 seconds. The runs also confirmed that the maximum tar yield for the PDU occurred over the temperature range 1000-1200° F. Tar composition appeared to be relatively independent of temperature, but varied significantly with residence time.

Work on coking of hydrotreating catalysts to determine equilibrium levels was also continued during the quarter.

Process Development Unit

The second process development unit shakedown run (Run No. 119-76) began on October 18 and ended November 31. The purpose of the run was to evaluate the new tar collection system and the recent revisions made in the pyrolysis system. During two stable periods of operation, 8,600 pounds of subbituminous coal were processed. A coal rate of 200 pounds per hour was sustained for 23 hours. The new bin activator was effective in preventing rat-holing, thereby improving char flow control.

The new tar collection system also operated without problems. In the first-stage recovery section, the primary scrubbers efficiently quenched the pyrolysis gases to within 10° F of the quench liquid temperature. The second recovery stage also functioned to design expectations. The main decanting vessel was effective in separating the pyrolytic water from the light oils. The recovered light oils had a specific gravity of 1.05 and settled in the decanter. No problems were experienced with water-and-oil emulsions. A successful test of the new vacuum flash unit was made using creosote. The feed system, heater, vacuum flash unit, vacuum pump, and control components functioned well. A problem of maintaining the circulating char inventory with fine feed coal occurred, and revisions were made to solve the problem prior to the next run.

PDU shakedown Run 120-76 was successfully completed during December, achieving all of the run objectives, including the evaluation of the revised feed coal preparation system. The run completed the first major program of the contract, the PDU shakedown. The PDU was operated from December 13 through December 20 for extended periods under stable process conditions. Sixteen tons of subbituminous coal were fed at an average rate of 203 pounds per hour. During one 72-hour period of operation, a rate of 224 pounds per hour was maintained, with only two minor interruptions due to mechanical problems. The design capacity of three tons per day was maintained for a twelve-hour period. Pyrolysis at temperatures of 960° and 1200° F were demonstrated, thus establishing the practical range for this key process variable. The tar collection system operated smoothly during the run, and about 3000 pounds of tar were recovered amounting to a nine to ten percent recovery in the tank, based on coal as-fed.

Bench-Scale Activities

A bench-scale pyrolysis run in October at 1200° F and a 1.5 second residence time yielded 16.7 percent tar, moisture and ash free basis, from subbituminous coal, confirming the relatively high yield of tar characteristic of the flash pyrolysis process. The sensitivity of tar yield to pyrolysis temperature and residence time was demonstrated by the lower tar yield of 9.7 percent (MAF basis) for the run performed at 1400° F with a three-second residence time.

Six bench-scale pyrolysis runs were completed in November, with maixmum tar yield obtained at a pyrolysis temperature of 1200° F and a residence time of 1.5 seconds. Increasing the residence time to three seconds resulted in a slight decrease in tar yield, but improved properties. The oil content of the tar was relatively independent of pyrolysis temperature but was affected by residence time. The oil content increased from an average value of 43 to 54 percent when the residence time was increased from 1.5 to three seconds, with a corresponding decrease in the preasphaltene content.

Work on coking of sulfided catalysts for bench-scale hydrotreating to determine equilibrium coke levels continued. Tests showed that equilibrium levels of nine to ten percent coke are obtained in 30 minutes at 400° C and 2,000 psi hydrogen pressure.

XIII. OPERATION OF SOLVENT-REFINED COAL (SRC) PILOT PLANT

SOUTHERN COMPANY SERVICES, INC.
BIRMINGHAM, ALABAMA

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

INTRODUCTION

The operation of the solvent refined coal (SRC) pilot plant at Wilsonville is jointly sponsored by ERDA and the Electric Power Research Institute (EPRI). The schedule for the project is shown in Figure XIII-1. The objectives of the project are to:

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

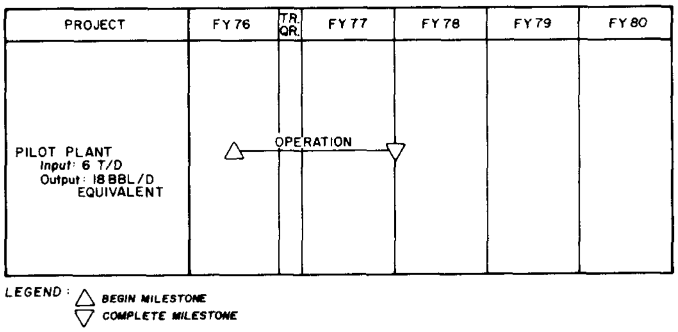


Figure XIII-1. SRC PILOT PLANT OPERATION SCHEDULE

PROCESS DESCRIPTION

The SRC process converts high-sulfur, high-ash coals to a clean distillate fuel and a clean high-Btu gas. A schematic of the process is shown in Figure XIII-2. The pulverized coal is mixed with two to three parts process-derived solvent. Hydrogen is then added to the coal-solvent slurry. The mixture is pumped to a preheater and then fed into a single-stage reactor or dissolver which operates at 800°-875° F, and a total pressure of approximately 1700 psig. Under these conditions, approximately 93 percent of the carbonaceous material in the coal feed is dissolved during the residence time of approximately 45 minutes. The product gases from the dissolver are separated and sent to a clean-up system to remove the hydrogen sulfide (about 60 percent of the organic sulfur in the coal is converted to hydrogen sulfide) and convert it to elemental sulfur. The liquid slurry is then subjected to a mineral separation step wherein the undissolved solids are removed. Process solvent is recovered from the coal solution by distillation and recycled to slurry the coal feed. The residue which remains is the solvent refined coal product, which solidifies at about 350° F.

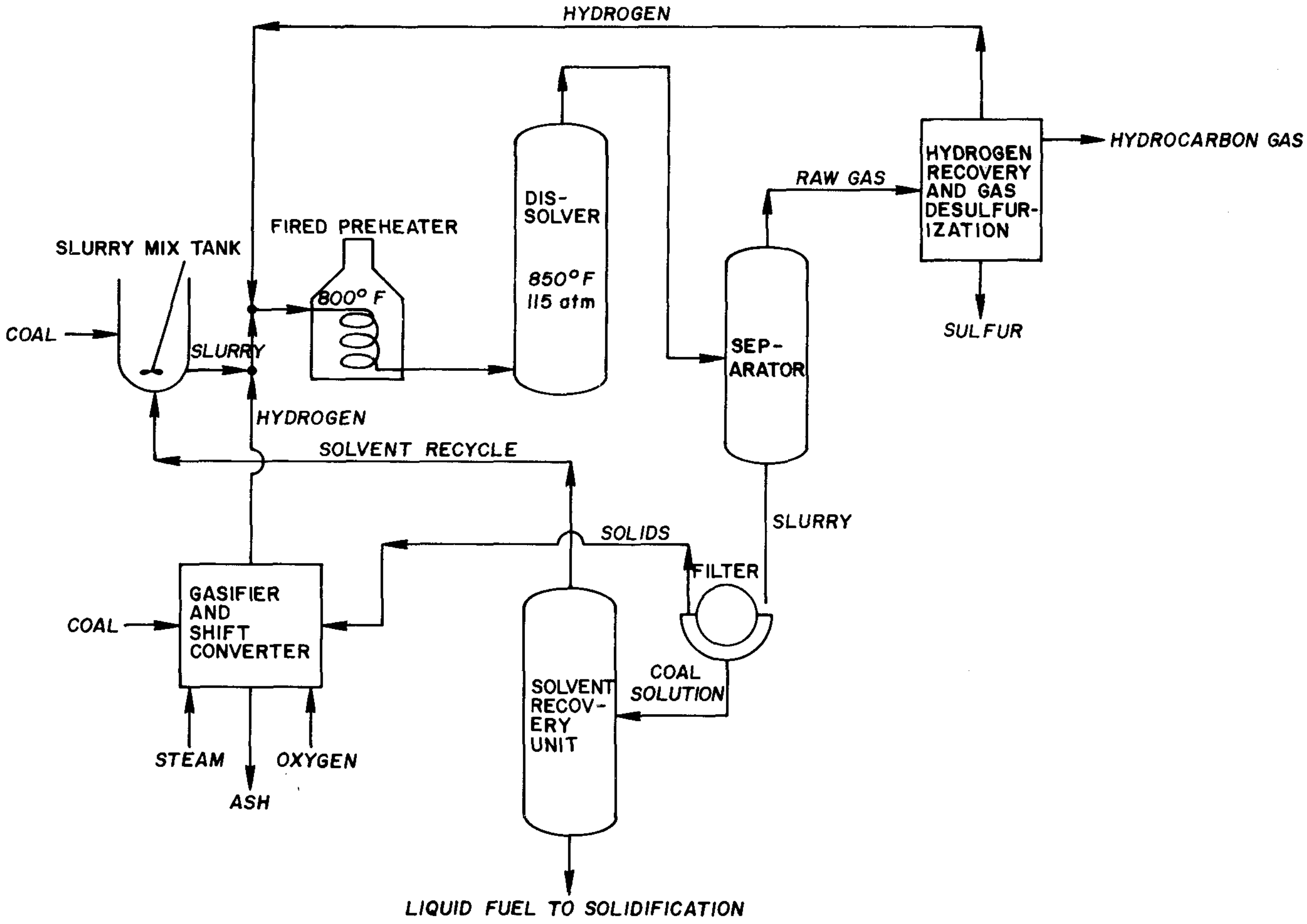


Figure XIII-2. SRC PROCESS FLOW DIAGRAM

HISTORY OF THE PROJECT

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

The pilot plant began operation in January 1974, and by the end of 1975, had been in operation for more than 7,800 hours, including periods of 45 and 75 days of sustained operation. One subbituminous and four bituminous coals have been tested. SRC product meeting plant specifications of 0.16 percent maximum ash and 0.96 percent maximum sulfur has been produced from each coal. A fuel meeting these specifications and having a heating value of 16,000 Btu per pound could be used to achieve compliance with most emission limits and EPA's "new source performance standards."

In January 1976, ERDA joined EPRI as a cosponsor of the pilot plant operation. To simulate operation at the Ft. Lewis SRC pilot plant, a mixture of coals from the Kentucky No. 9 and 14 seams were used for plant operation. Coal feed rates as high as 75 pounds of coal per hour per cubic foot of dissolver volume, almost three times the design feed rate, were achieved. Material balance data for ten runs were obtained, allowing correlation of the results from both the Wilsonville and Ft. Lewis pilot plants. Empirical models to aid in scaling up to larger plants were developed for predicting conversion, sulfur removal, and filtration rates for operation with Kentucl No. 9 and 14 coals.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

During October, the Wilsonville solvent refined coal (SRC) plant operated for 602 hours and was on stream 81 percent of the time. Monterey (Illinois No. 6) coal was used in the plant all of September, October, November, and continued until early December. In October, plant operations were interrupted and delayed by equipment failures, line plugging, and power failures.

In November, the plant operated for 608 hours and was on stream 84 percent of the time. Material balances for three runs were completed during the month.

The plant operated for 460 hours and was on stream 62 percent of the time during December. One run with Monterey coal and two with Amax coal were completed. Solids buildup was not as great as when Amax coal was used previously in November 1975.

Additional data were accumulated in November and December to relate pressure drop across the dissolver to solids buildup in the dissolver.

Pilot Plant Operations

At the beginning of the quarter, Run 94 was in progress with Monterey (Ill. No. 6) coal. Conditions duplicated Run 50 conditions with the exception that Run 50 used 350° F+ boiling range solvent, and Run 94 used 450° F+ boiling range solvent. More than the normal time was spent completing the run due to equipment failures, line plugging, and power failures. Following is a comparison of the two runs:

	Run 50	Run 94
Conversion, % MAF coal	95	95
Hydrogen consumption, % MAF coal	3.0	3.4
SRC yield, % MAF coal	52	50
Gas Make, % MAF coal	13	14
Sulfur in SRC, wt. %	0.95	1.0

During November, Run 95 was conducted for 36 hours and Runs 96 and 97 were conducted for 48 hours. Run 95 used the full dissolver volume and Runs 96 and 97 used one-half the dissolver volume.

Comparison of the data follows:

	Run 95	Run 96	Run 97
Conversion, % MAF coal	94	94	94
Hydrogen consumption, % MAF coal	2.8	2.9	2.1
SRC yield, % MAF coal	59	52	59
Gas Make, % MAF coal	15	13	10
Sulfur in SRC, wt. %	1.0	1.2	1.4

Additional data were accumulated to relate pressure drop across the dissolver with solids buildup in the dissolver. The accumulated solids in the dissolver were 0.2 percent of moisture-free coal for Run 95 and 0.1 percent for Runs 96 and 97.

Run 98 was in progress at the beginning of December. The run bypassed the dissolver and concluded that test program for Monterey coal. Amax coal from the Bell Ayr mine in Wyoming was used for the next series of runs using the full dissolver volume. Run 99 was an attempt to compare the difference between 350° F+ boiling range process solvent with Run 57 using 450° F+ boiling range solvent. Coal concentration in the feed slurry was increased from 20 weight percent in Run 99 to 25 weight percent in Run 100, while other process conditions remained unchanged. Comparison of the runs follows:

	Run 98	Run 57	Run 99	Run 100
Conversion, % MAF coal	89	85	90	91
Hydrogen consumption, % MAF coal	1.8	3.2	3.4	3.8
SRC yield, % MAF coal	76	40	46	42
Gas Make, % MAF coal	4	17	17	12
Sulfur in SRC, wt. %	2.1	0.2	0.2	0.2

Solids buildup did not present the problem which was previously encountered using Amax coal in November 1975. The filtration rates for Amax coal were three times those for Monterey coal. Filter screen blinding did not occur while using Amax coal.

XIV. DONOR SOLVENT LIQUEFACTION PROCESS

EXXON RESEARCH AND ENGINEERING
COMPANY

BAYTOWN, TEXAS

Contract No.: E(49-18)-2353

Total Funding: $12,740,360

INTRODUCTION

Under the terms of this contract, details of prior development work on the Exxon Donor Solvent (EDS) process conducted between 1966 and January 1976 are confidential. The program schedule for this project is shown in Figure XIV-1. The objectives of this contract are to:

- Expand the process data base from experimental units of up to one-ton-per-day to confirm the design for the construction of a 250-ton-per-day pilot plant.
- Provide data on the compatibility of coal-derived products with petroleum liquids, and on the storage behavior and end use potential of raw and hydrotreated coal liquids as well as blends of coal liquids and petroleum liquids.
- Provide data for the design of large scale coking and gasification units integrated with the EDS process.
- Provide economic estimates for conceptual commercial plants.
- Provide a data base to improve the design scale-up capability of critical components and plant sections.

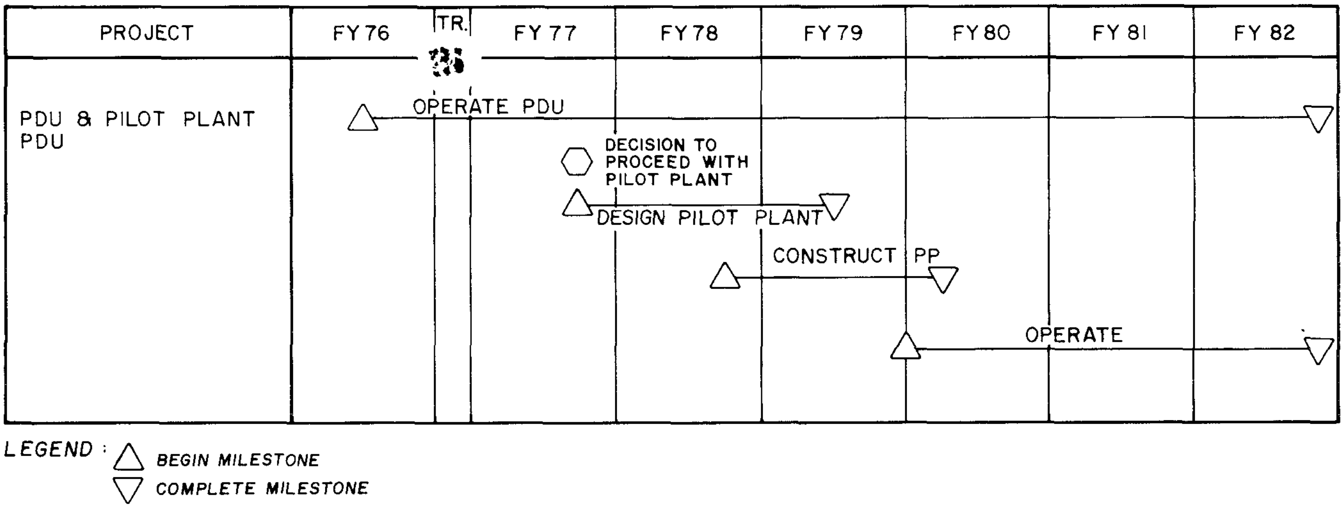


Figure XIV-1. DONOR SOLVENT PROJECT SCHEDULE

PROCESS DESCRIPTION

Steps in the Donor Solvent Process involve engineering and design technology similar to that practiced in the petroleum industry. The process was designed to permit process control, allow for feeding different coals, and allow product distribution to vary based on market demand. In this process, the donor solvent provides a mechanism to allow intimate contact between hydrogen and dispersed coal fragments. The donor hydrogen content of the spent solvent is restored after liquefaction in the solvent hydrogenation reactor. This step also reduces the sulfur, nitrogen, and oxygen contents of the recycle solvent. The donor hydrogen content is the major parameter used to evaluate the effectiveness of various catalysts and processing conditions to produce good quality donor solvent. A schematic of the process is provided in Figure XIV-2.

The coal is dried and ground to 30-mesh and mixed with recycle solvent to form a slurry. Hydrogen is normally preheated separately and combined with preheated slurry at the reactor inlet. An alternate mode of operation is to combine the hydrogen with the slurry before preheating.

After liquefaction, the product goes to the first stage of separation. Water is removed, hydrogen is recycled, and heavy liquid products are sent to the vacuum separation section.

The primary vacuum flash tower removes the mineral matter and unreacted coal. The secondary vacuum tower is used to remove a heavy distillate which becomes the endpoint of the recycle solvent. This overhead from the secondary vacuum flash tower is then combined with the lighter liquid stream recovered from the liquefaction reactor separator system and fed to the solvent hydrotreating section, consisting of fixed-bed catalytic reactors. The solvent and naphtha from the solvent hydrotreating section are then separated by fractionation. Most of the solvent is recycled, as is the hydrogen, after undergoing cleanup in a monoethanolamine (MEA) scrubbing system.

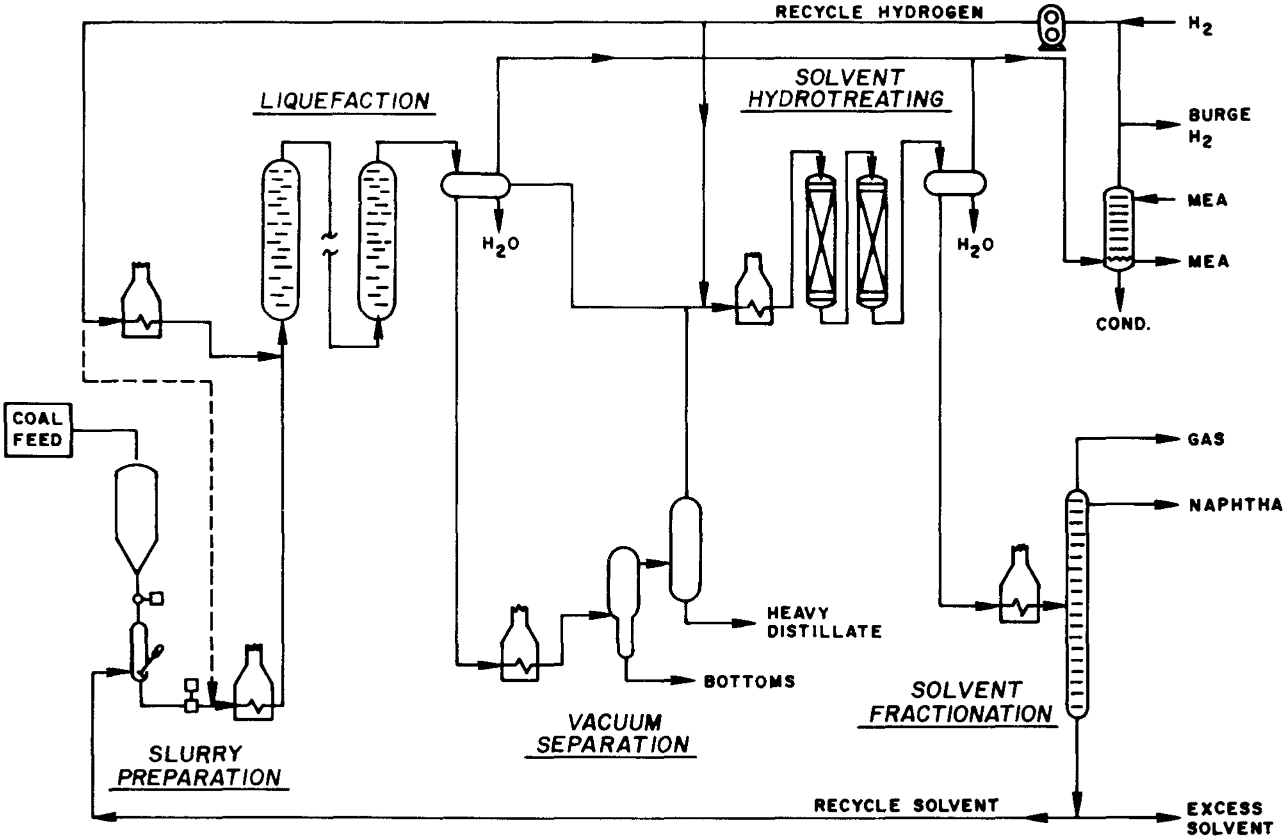


Figure XIV-2. DONOR SOLVENT LIQUEFACTION PROCESS

HISTORY OF THE PROJECT

Exxon's research on the EDS process began in 1966 and has resulted in a commercial study design. A semi-empirical kinetic model of liquefaction yields from Illinois coal has been expanded to include recent results from high-severity liquefaction experiments. Progress has been made in the development of a computer model of the entire integrated EDS process. The model will simulate, and link together in a single interfacing package, the six major processing sections: liquefaction, solvent hydrotreating, coking, cryogenic hydrogen recovery, steam/methane reforming, and final product separation.

Evaluation of several process alternatives have begun, including development of a computerized process alternative model to be used as a basic tool for the process engineering and economic studies which are planned. Engineering studies have been initiated to identify and develop equipment and engineering data needed for a safe, operable, and reliable EDS commercial plant.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

Lignite testing in the Recycle Coal Liquefaction Unit was completed during the quarter, and process variable studies of Wyoming subbituminous coal began. Runs in in the unit at standard conditions on Wyoming coal resulted in higher 1000° F$^-$ conversion than from either Illinois No. 6 bituminous coal or North Dakota lignite. Tests on the Wyoming coal showed that calcium carbonate formation and deposition in the liquefaction reactor was essentially eliminated with sulfuric-acid-treated coal.

After a scheduled six-month downtime of the Coal Liquefaction Pilot Plant, operations of the unit were begun in November. Trial feasibility operation processing Wyodak coal were completed. The studies showed that the same configuration used successfully for Illinois No. 6 coal can also be used for processing Wyodak coal.

An operability study in the fluid coking mode was successfully completed during the quarter in the two-barrel-per-day integrated coker/gasification pilot plant G). Comparison of IKG operability on coal lique- ion bottoms to IKG resid operability supports the feasibility of commercial scale operation of fluid coking on coal liquefaction bottoms.

Laboratory Studies

The lignite screening study was completed in the 50-pound-per-day Recycle Coal Liquefaction Unit (RCLU) during October. A run was made with North Dakota lignite at 800° F, 1500 psig, and 63 percent of standard space velocity. The run provided additional information on solids accumulation rates and scale buildup on the walls of the reactor.

During the last half of October, an RCLU study was initiated to determine the effects of processing conditions on conversion and yield structure of Wyoming subbituminous coal and to develop additional data on Wyoming coal operating difficulties experienced earlier.

In two yield periods, performed at 840° F, 1500 psig, and standard space velocity, liquefaction of the Wyoming coal resulted in six percent higher 1000° F$^-$ conversion than from Illinois No. 6 bituminous coal, and three percent higher conversion than for North Dakota lignite.

The C_4-1000° F liquid product yield from Wyoming coal was about the same as for Illinois No. 6 coal and five percent greater than for lignite. The C_1-C_3 gas yield was the same for all three coals. As with earlier Wyoming coal experiments, this operation was terminated due to plugging in the reactor.

Runs in November were also made at standard conditions of 840° F, 1500 psig, and typical space velocity as well as at 2500 psig. Results of the runs indicated that increased pressure did not alleviate the calcium carbonate deposition problem experienced with processing of subbituminous coal.

Ten RCLU runs were also made in November on Wyoming coal, pretreated with sulfuric acid. In contrast to the runs with untreated coal, very limited amounts of calcium carbonate were present in the accumulated solids. Early operational problems associated with Wyoming subbituminous coal were attributed to calcium carbonate deposition in the liquefaction reactor. The calcium carbonate accumulated in the reactor as a wall scale and as large free-flowing agglomerates, causing problems in the reactor and downstream equipment. Wyoming coal, pretreated with sulfuric acid, appeared to be a solution to calcium carbonate formation. The acid pretreatment converted calcium humate in the coal to calcium sulfate prior to liquefaction and, therefore, essentially eliminated calcium carbonate formation in the reactor.

Testing in December also investigated a second problem associated with processing western subbituminous coal. The heavy, partly liquefied bottoms are highly viscous and accumulate in the smalll lines associated with the liquefaction reactors and eventually cause plugging. In an attempt to alleviate the plugging, the concentration of coal in the slurry was decreased by 35 percent, and a run at 840° F, 1500 psig, and a typical space velocity was completed with no operational problems. The more dilute slurry appeared to alleviate the problem of the highly viscous bottoms.

Pilot Plant Operation

Startup of the one-ton-per-day Coal Liquefaction Pilot Plant (CLPP) was well underway at the beginning of the quarter. In October, the pre-startup turnaround was completed, and the operators were brought onto the unit. All the safety valves were renewed and all necessary instrument calibrations were completed.

During the last half of October the solvent hydrotreating, solvent fractionation, and high-pressure gas scrubbing/compression sections were brought onstream. Operation of the sections produced 4,000 gallons of hydrogenated creosote oil, used as startup solvent for the CLPP, from raw creosote oil. Each of the sections performed well during the hydrogenated creosote oil production run.

The new solvent-quench-oil system in the hydrotreating section operated successfully, maintaining a more uniform temperature profile in the solvent hydrotreater than previously.

During November, the primary emphasis was on checking out the modifications made prior to startup. Coal feed was started to the unit in November. The service factor on coal feed was 65 percent, representing an improvement over the 35 percent service factor experienced during the original startup.

During December, Illinois No. 6 and Wyoming coals were processed in the pilot plant. Verification operations were completed with Illinois No. 6 coal. The two primary objectives of the operations were to re-establish material balance closure and confirm the product yields from the unit following modifications. Three yield periods were completed at typical operating conditions. Material balances for these yield periods ranged from 98 to 101 percent.

Wyoming coal feasibility operations were conducted to determine whether or not any unit modifications were necessary before more extensive studies were conducted on the coal. Results from the operations showed that no major modifications were required, however, operating procedures will be changed to more accurately control the liquefaction temperature and viscosity of vacuum tower bottoms. Yield data comparison show good agreement between the CCLP and the RCLU.

The two-barrel-per-day coking/gasification pilot plant (IKG) was operated in the fluid-coking mode for the entire month of October. The operations demonstrated the IKG operability on EDS liquefaction bottoms, and observations and samples for Phase III-A deliveries were secured. The comparison of IKG operability on coal liquefaction bottoms to IKG reside operability supports the feasibility of commercial scale operation of fluid coking on coal liquefaction bottoms.

XV. FLUID COKING OF COAL LIQUEFACTION RESIDUES

EXXON RESEARCH AND ENGINEERING COMPANY

Contract No.: E(49-18)-2422

Total Funding: $11,312,470

INTRODUCTION

Exxon Research and Engineering Company, under the sponsorship of ERDA, will study 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 residues from one selected coal liquefaction process. The program schedule for this project is shown in Figure XV-1.

PROGRAM DESCRIPTION

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

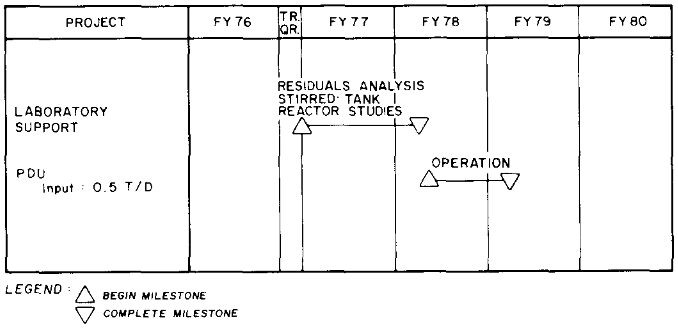


Figure XV-1. FLUID COKING PROGRAM SCHEDULE

Three samples of coal liquid residue produced in ERDA-sponsored coal liquefaction projects will be analyzed and processed using bench-scale equipment. A Continuous Stirred Coking Unit (CSCU) will be used to obtain quantitative yield data for fluid coking of the coal liquefaction residues. This type of pilot unit was utilized successfully in scale-up of yield and quality data for petroleum. CSCU simulates the steam fluidization of a commercial fluid coker by means of a mechanical agitator. A schematic is shown in Figure XV-2. One of the selected coal liquid residues will be processed in a nominal two-barrel-per-day fluidized-bed pilot unit.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

Analytical tests for the H-Coal solvent precipitation underflow sample were completed during the quarter. Distillation and viscosity tests were run on the sample and the specific gravity and Conradson Carbon values determined.

Bench-scale studies in the one-gram test unit on coking yields for the H-Coal sample were completed

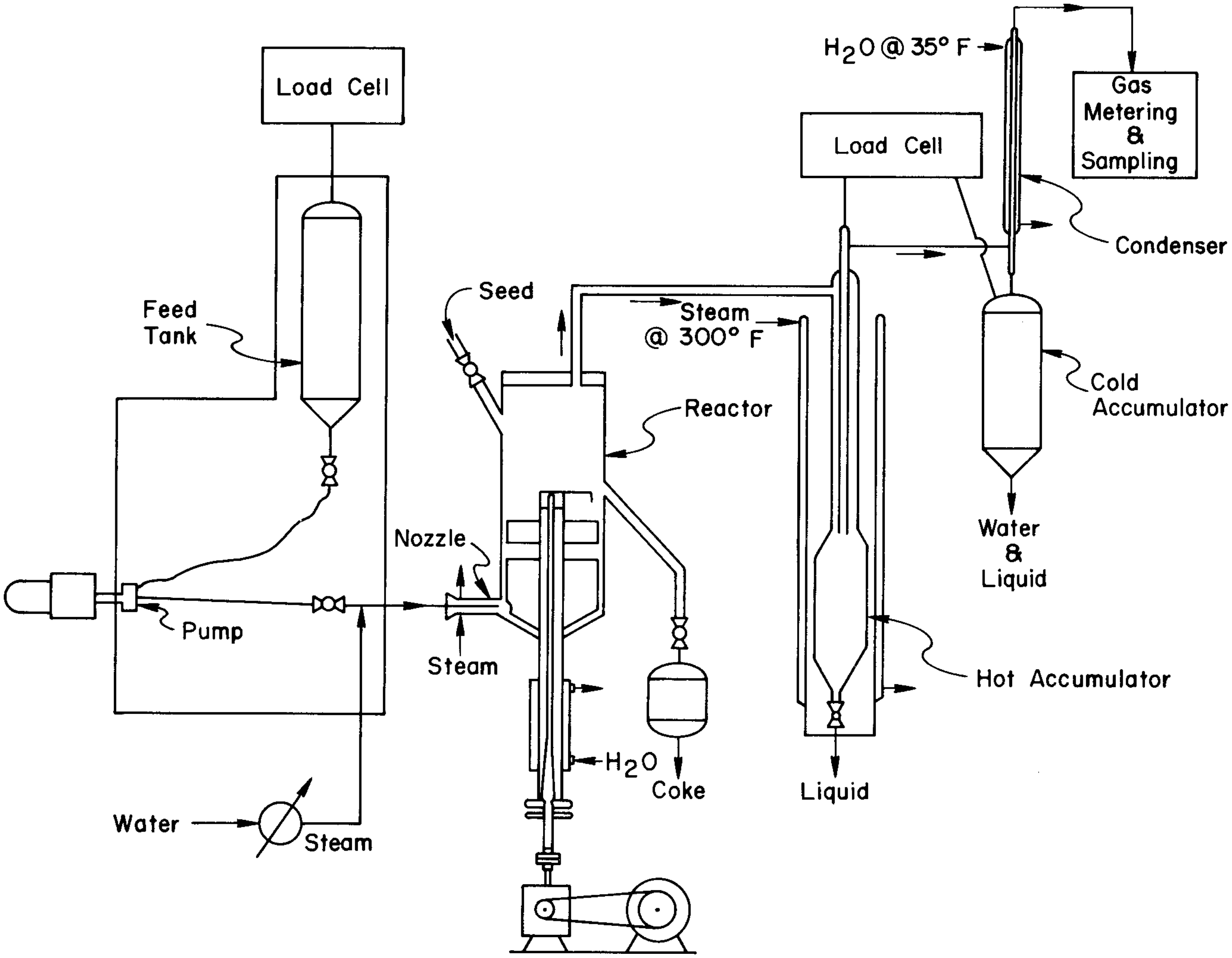


Figure XV-2. FLUID COKING PROCESS SCHEMATIC

and tests began in November on the Continuous Stirred Coking Unit (CSCU).

Analytical Characterization

During the quarter, analytical tests for the H-Coal solvent precipitation underflow sample were completed. The sample was handled as a composite liquid phase and solid phase sample.

Results from the distillation tests, however, were based on cumulative liquid and solid phase results in order to be more representative than the composite sample distillation results. The cumulative amount of 1000° F⁻ material in the sample based on weight fraction of the liquid and the solid was approximately 42 percent.

Viscosity test results indicated the sample contained a large fraction of light material. A five-day thermal stability test was conducted on the H-Coal sample at 400° F. The rate of increase of viscosity was approximately one poise per day. Viscosity measurements ranged from 13 poise at 200° F to 0.2 poise at 500° F. Viscosities ranging from 3.5 to 19 poise were also determined on the solid phase alone over the temperature range of 300°-500° F.

The specific gravity of a composite sample was found to be 1.28. The Conradson Carbon value was found to be 50.5 weight percent.

Bench-Scale Tests

Bench screening studies in the one-gram test unit coking yields for the H-Coal sample were complete during the quarter. The maximum liquid yield obtained was approximately 47 percent at 1100° F. Liquid and gas yields increased at the expense of coke formation up to 1100° F.

Tests began in November on the Continuous Stirred Coking Unit (CSCU) in order to obtain yield and quality data not provided by the bench screening tests. The CSCU program for H-Coal is designed to investigate coking product yield and quality as a function of process variables for two steam rates, low and high-vapor residence times, and three temperatures in the 900-1100° F range selected as a result of the bench screening tests.

Two runs were completed in November on the CSCU with the H-Coal sample. To ensure representative sampling of the solid phase of the H-Coal sample, the material was cored vertically from the barrel. The solid phase was blended with an appropriate quantity of liquid phase material and homogenized by feed case pump-around prior to coking.

A third run was completed in December and a fourth was stopped due to pressure buildup in the reactor during the run. Examination of the reactor showed that 689 grams of coke had been deposited on the top walls of the reactor and on the outlet filter. The next two runs were terminated due to plugging of the nozzle and hot box feedlines.

XVI. CHARACTERIZATION OF COAL LIQUIDS

UOP, INC.
DES MOINES, ILLINOIS

Contract No.: E(49-18)-2010
Total Funding: $369,914

INTRODUCTION

UOP, Inc. is under contract to ERDA to demonstrate the nature and degree of possible upgrading of primary coal liquids using technology now available in the petroleum refining industry. The objectives of this contract are to:

- Evaluate the processability of the primary products from each of the current coal liquefaction processes.
- Estimate the optimum conditions for processing coal liquids in current hydrorefining units.
- Correlate bench-scale and analytical data in order to estimate processability on the basis of analytical data alone.
- Assess the economic viability of feeding raw coal liquids to existing refineries in order to produce a full slate of finished products.
- Identify a preferred method for processing coal liquids in an existing refinery.
- Identify areas where research and development appear to be essential in order to realize commercial utilization of coal liquids as refinery feedstocks.

PROJECT DESCRIPTION

Product liquids from coal liquefaction plants differ m petroleum crude in that they are deficient in hydrogen, have a higher polynuclear aromatic content, higher level of inorganic impurities, and are generally heavier stocks.

The response of existing full-scale refineries to almost any petroleum crude feedstock can be accurately predicted from chemical and physical characteristics of that stock. These characteristics, along with bench-scale refining unit test data, have been corroborated by full scale refinery experience. This approach, formally termed "characterization", will be used to estimate the result of processing coal liquids in existing oil refineries.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

Processability characterization tests on the last three of the eight ERDA coal liquids were completed during the quarter. Solvent refined coal (SRC) without the addition of extraneous solvent, upgraded SRC, and H-Coal process hydroclone underflow were the three liquids evaluated.

The SRC was catalytically hydrotreated without addition of extraneous solvent by recycling hydrotreated product as solvent. The yield of product from SRC was 98^+ weight percent at a hydrogen consumption rate of 3800 standard cubic feet per barrel.

A sample of H-Coal process hydroclone underflow was the seventh of the eight coal liquids to be characterized. A portion of the sample was batch filtered in the laboratory to obtain a substantially de-ashed charge stock. This material was then hydrotreated at four sets of process conditions.

The last of the coal liquids to be characterized was an upgraded SRC feedstock using the SRC previously evaluated in the sixth run. The upgraded SRC, containing 9.8 percent heptane-insolubles and 70.1 percent 650° F^{+} bottoms, was processed over a commercial hydrocracking catalyst at four sets of conditions. Low yields of 650° F distillate was noted during the tests, but this was attributed to the presence of high-molecular-weight asphaltic materials.

Bench-Scale Operations

The sixth of eight ERDA coal liquids to be characterized was Solvent Refined Coal (SRC) from the pilot plant in Ft. Lewis, Washington. The SRC was hydrotreated without addition of extraneous solvent.

The objective of the study was to establish a product distribution for hydrotreating SRC undisturbed by the addition of extraneous solvent. This was accomplished by recycling hydrotreated product as solvent at a combined feed ratio of 2.0 to 2.5. Two continuous runs were made to study the effect of changes in operating conditions. A third run of extended duration was made at a single set of conditions to provide feedstock for subequent hydrocracking as the last of the coal liquids. A 90^{+} percent conversion of heptane-insolubles was attained under the most severe operating conditions. The product slate, in which the products were assigned identity according to their boiling range, was:

Gas	1.5 weight percent
Gasoline	5.2 weight percent
Kerosene	5.6 weight percent
Light Gas Oil	32.7 weight percent
Heavy Gas Oil	14.2 weight percent
850° F^{+} bottoms	40.8 weight percent

The seventh of the coal liquids to be characterized was a 113-kilogram sample of H-Coal process hydroclone underflow. Laboratory batch filtration of a portion of the sample at 200° C and 500 psig yielded 24 kilograms of hydrotreater feedstock containing 0.12 percent ash, 49 percent heptane-insolubles, and 29 perc benzene-insolubles.

Bench-scale, continuous hydrotreatment of the filtrate was conducted at four process conditions, followed by a catalyst activity check at base conditions. At these base conditions, the heptane-insoluble conversion was initially 84.8 percent. Decreasing space velocity to one-half base conditions increased conversion to 93.0 percent. Response to a 10° C temperature increase was minor. The final return to base conditions gave only 76.7 percent heptane-insolubles conversion, indicating appreciable catalyst deactivation. The run was terminated voluntarily at 185 hours. Pressure surges indicated incipient bed plugging, probably due to residual ash.

In addition to studying the effects of variables, the sixth run was also a hydrotreating run which produced 4.5 gallons of upgraded SRC for use as the last feedstock in the hydrocracking study. The experiments were conducted at four sets of hydrocracking conditions. At base case space velocity, 10° C above base temperature, and 200 psig above base pressure, conversion to 650° F^{-} distillate was 11.8 volume percent and heptane-insolubles were reduced to 5.36 percent from 9.75 percent in the feed. Raising the temperature 10° C, at otherwise identical conditions, did not improve conversion or lower the heptane-insolubles content in the liquid product. When the temperature was lowered to 10° C above base temperature, while other processing variables remained unchanged, conversion to 650° F^{-} distillate decreased to 6.0 percent while heptane-insolubles content increased to 6.57 percent. The run was concluded with a catalyst activity check at the initial conditions. Results showed that after 119 hours of processing, conversion to 650° F^{-} distillate decreased from 11.8 to 2.0 volume percent, the product hydrogen content dropped from 9.32 to 8.83 percent, the product nitrogen content increased from 0.153 to 0.377 percent, and the API gravity decreased from 5.0 to 3.2. The data showed that, as in hydrocracking Synthoil product, the conversion to 650° F^{-} distillate was very low because of the presence of high-molecular-weight asphaltic materials.

XVII. COMMERCIAL SCALE EBULLATED-BED HYDROPROCESSING OF SOLVENT-REFINED COAL EXTRACT

CITIES SERVICE RESEARCH AND
DEVELOPMENT COMPANY
CRANBURY, NEW JERSEY

Plant Site: Lake Charles, Louisiana
Contract No.: E(49-18)-2038
Total Funding: $591,122

INTRODUCTION

Cities Service, under ERDA sponsorship, is determining the operating conditions and modifications necessary to utilize an existing LC-Fining unit (ebullated-bed hydroprocessing unit) to upgrade a production quantity of Solvent Refined Coal (SRC) extract. The specific objectives of this contract are to:

- Determine the ebullated-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 ebullated-bed hydroprocessing equipment.
- Demonstrate that the SRC process and ebullated-bed hydroprocessing can be combined to provide a valuable liquid fuel or refinery feedstock from coal and thereby accelerate commercial utilization of the SRC process.

PROCESS DESCRIPTION

Solvent Refined Coal (SRC) extract is a hydrocarbon ixture containing significant quantities of oxygen, trogen, 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 finished products. Ebullated-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 XVII-1.

The SRC extract, together with hydrogen, is fed upward through the reactor as a liquid/gas mixture at

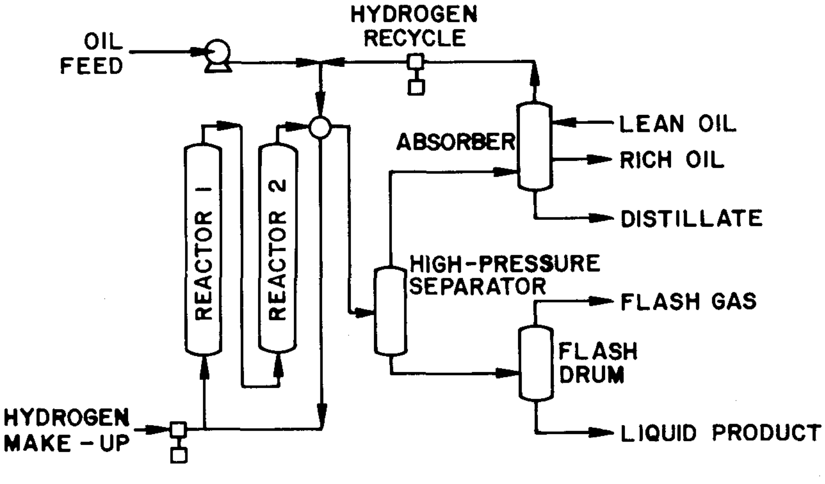


Figure XVII-1. EBULLATED-BED HYDROPROCESSING PROCESS SCHEMATIC a velocity such that the catalyst is in continuous motion. Part of the reactor effluent is recycled back through the reactors to maintain the necessary liquid velocity through the reactor system (the ebullated-bed). Because of the movement 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.

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 an ebullated-bed unit to process a specified quantity of SRC extract.

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

The last of the experimental runs designed to determine the operating conditions necessary to process solvent refined coal (SRC) in the commercial LC-Fining units were completed during the quarter. A blend of 78.0 volume percent SRC and 22.0 volume percent Koppers heavy residue creosote oil was the feed for Run LC-22. Conversion of 850° F plus material averaged 32.9 volume percent. Hydrogen consumption averaged 2,445 standard cubic feet per barrel.

The first 30-day catalyst aging run, LCF-24, processing a blend of 67.0 weight percent SRC and 33.0 weight percent Koppers heavy residue creosote oil was completed. Conversion of the 850° F+ material averaged 45.9 volume percent.

Engineering studies were temporarily suspended during the quarter as data from the catalyst aging runs are necessary to complete the studies to be conducted at Lake Charles.

Bench-Scale Operations

The last of the experimental runs, LCF-22, designed to determine the operating conditions necessary to process SRC, was completed during the quarter. The run was terminated after 240 hours at the same operating conditions as the preceding runs. Feed for th run was a blend of 78.0 volume percent SRC and 22.0 volume percent Kopper heavy residue creosote.

Data from the four runs provided the following information:

- SRC can be redissolved in native process solvent and selected foreign solvents without adverse effects.
- Gas yields vary according to the elemental analysis of the feed; i.e., the yield of water varies with the oxygen content of the feed blend. The yield of light hydrocarbons was in the range and ratio expected, except for the methane yield in Run LCF-19 which was lower than the methane yield expected.
- The yield of the liquid fractions varies according to the composition of the feed blend and level of conversion, indicating that the cracking rates for a given boiling range of material is independent of the solvent used to redissolve the SRC.
- The non-coal-derived solvent (Neville LX-745 Aromatic Oil) provided higher conversion of the 850° F+ material at a given level of SRC concentration than the two coal-based solvents.
- A drop in the amount of 850° F+ material converted to lighter boiling material occurred with increasing amounts of SRC in the feed blend.
- Coal-derived solvents produced blends with lower viscosities and lower amounts of insolubles than the petroleum-based Neville oil.

The first 30-day catalyst aging run, LCF-23, began during the quarter. The run was terminated due to an operational upset which caused both reactors to plug. The catalyst in both reactors was changed and Run LCF-24 began in November. Both runs processed a blend of 69.5 volume percent SRC and 30.5 volume percent Koppers heavy residue creosote oil which is the designated feed blend for the commercial scale processing of SRC extract at Lake Charles. Run LCF-24 was terminated after a total of 496.5 hours of operation using the commercial feed blend.

Conversion of 850° F+ material averaged 32.9 volume percent (ranged from 30.8 to 36.2 volume percent) and hydrogen consumption averaged 2445 standard cubic feet per barrel (ranged from 1965 to 2890 standard cubic feet per barrel).

XVIII. GASIFICATION OF RESIDUAL COAL LIQUEFACTION PRODUCTS

TEXACO, INC.

Contract No.: E(49-18)-2247
Total Funding: $929,840
(100% ERDA)

INTRODUCTION

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

PROCESS DESCRIPTION

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

PROGRESS DURING OCTOBER-DECEMBER 1976

Summary

Tests during the quarter were performed on two possible candidate feedstocks obtained from the H-Coal liquefaction of Illinois No. 6 coal. The "Syncrude Vacuum Tower Bottoms" appeared to be a suitable feedstock for the Texaco coal gasification process, while the "Settler Stripped Underflow" seemed a poor candidate because of its physical properties.

Laboratory Tests

Testing began on both samples of H-Coal residues received to determine the chemical composition and physical properties of each material. On the basis of these results, the method of feeding the material to the generator 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 "Syncrude Vacuum Tower Bottoms" appeared to be an excellent candidate feedstock for the Texaco process. A temperature of 480°-500° F will be required to pump this residue into the gasifier. At ambient temperatures it was a brittle solid which can be charged easily into the melt tank.

The "Settler Stripped Underflow" appeared to be made up of fine particles pressed together into random sized hard lumps which could not be melted at 600° F. All attempts to break apart the hard lumps failed. Because of these poor physical characteristics, the material seemed to be a poor candidate for the Texaco process.

It was recommended that a Type II pilot plant evaluation be performed on the "Syncrude Vacuum Tower Bottoms." The Type II evaluation would consist of a preliminary pilot plant test in which approx mately 20 barrels of the candidate feedstock would b gasified. The evaluation would 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 operating problems could also be identified.

XIX. PRODUCTION OF CLEAN INDUSTRIAL AND TRANSPORTATION FUELS FROM COAL

THE LUMMUS COMPANY
BLOOMFIELD, NEW JERSEY

Plant Site: Bloomfield, New Jersey
Contract No.: E(49-18)-2514

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-sufficient, 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 bench-scale 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 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.

PROCESS DESCRIPTION

The Lummus Clean Fuels From Coal (CFFC) Process is based on direct catalytic hydroliquefaction in a multistage, plug-flow ebullated-bed reactor. The process employs a proprietary catalyst having a substantial denitrogenation capability and an integrated solvent deashing 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. Approximately 25 percent of the effluent from the last reactor is recycled to the first, to aid in the maintenance of the ebullated bed and ensure emergency operation in the event of a feed slurry pump failure. 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.

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

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

HISTORY OF THE PROJECT

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

Lummus also performed exploratory work on denitrogenation catalysts. A highly active catalyst was formulated in late 1974 and a patent application on the catalyst was filed.

The proposed program was initially submitted to ERDA in 1975 as an unsolicited proposal. Following contract negotiations, a revised experimental program was authorized and a contract granted by ERDA on September 28, 1976.

PROGRESS DURING OCTOBER-DECEMBER 1976

Effort during the quarter was directed at the necessary modifications to convert the expanded coal 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. Bid packages covering the mechanical and electrical sub-contractor installation work were prepared and issued, and bids received. By the end of the quarter, the bids had been evaluated. Procurement activities continued throughout the reporting period.

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

$$\text{density of oils; } {}^\circ\text{API} = \frac{141.5}{\text{Specific gravity, } 60^\circ\text{F}/60^\circ\text{F}} - 131.5$$

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

ash—solid residue remaining after the combustion of coal.

ASTM—American Society for Testing Materials.

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

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

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

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

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

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

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

BTX—benzene, toluene, xylene; aromatic hydrocarbons.

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

nation—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 of crushed coke usually passing a ½-inch or ¾-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 $CaMg(CO_3)_2$.

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 yields from coal or oil shale; conducted in a retort und 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 suspen solids from a flowing liquid by means of the centrifu forces set up when the liquid is made to flow through a tight conical vortex.

drocoking—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 hydrogen 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 culcite 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.

anation—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—particlized 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 componds, hydrogen cyanide, and carbon dioxide are removed from the gas stream by the methanol at temperatures below 0° C.

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

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

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

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

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

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

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

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

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

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

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

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

slurry—a suspension of pulverized solid in a liquid.

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

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

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

solvent refined coal (SRC)—a coal extract derived by solvent extraction; a brittle, vitreous solid (m.p. 300° F to 400° F) containing about 0.1 percent ash and about 10 percent of sulfur in the original coal feedstock; calorific value is abo 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 moisture and 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, min 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 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:

$$CO + H_2O \rightleftarrows H_2 + CO_2.$$

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