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EXPLORATORY RESEARCH ON SOLVENT REFINED COAL LIQUEFACTION

Quarterly Technical Progress Report for April 1—June 30, 1979

July 1980

Work Performed Under Contract No. AC01-79ET14800

The Pittsburg & Midway Coal Mining Co.
Shawnee Mission, Kansas

U. S. DEPARTMENT OF ENERGY

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EXPLORATORY RESEARCH
ON
SOLVENT REFINED COAL LIQUEFACTION

QUARTERLY TECHNICAL PROGRESS REPORT
FOR THE PERIOD
APRIL 1, 1979 THROUGH JUNE 30, 1979

THE PITTSBURG & MIDWAY COAL MINING CO.
P. O. BOX 2970
SHAWNEE MISSION, KANSAS 66201

July 1980

PREPARED FOR THE
U. S. DEPARTMENT OF ENERGY
DIVISION OF COAL CONVERSION AND UTILIZATION
UNDER CONTRACT DEAC-0179-ET14800

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ABSTRACT

This report summarizes the progress of the Exploratory Research on Solvent Refined Coal Liquefaction project by The Pittsburg & Midway Coal Mining Co.'s Merriam Laboratory for the period April 1, 1979 through June 30, 1979. Experimental work during this period included a number of short residence time runs, but discussion of that work has been delayed until a later report. Experimental work reported focuses on an investigation of the decline in solvent quality experienced by the Wilsonville Pilot Plant during runs in support of the SRC I Demonstration Plant. A four run series was initiated with Wilsonville solvent; both the coal used at Wilsonville (Kentucky 6/11 - Pyro Mine) and Kentucky 9/14 coal from the Colonial Mine were used. The effect of pyrite addition to the Pyro Mine coal was investigated. No solvent quality or coking problems were experienced in the Merriam runs. Significant changes in solvent composition were apparent and equilibrated solvent samples were returned to Wilsonville for solvent quality testing.

I. SUMMARY

This is the first quarterly technical progress report to the Department of Energy in which work at the Merriam Laboratory on the Solvent Refined Coal (SRC) process is reported under separate cover. For this reason, a brief history of work at the laboratory has been included as well as an annotated bibliography of previously issued final and interim reports (Appendix A).

Work reported for this quarter focuses on an investigation of the decline in solvent quality experienced by the Wilsonville Pilot Plant during runs in support of the SRC I Demonstration Plant. The coal in use was Kentucky 6/11 from the Pyro Mine which was mistakenly supplied in place of the requested Kentucky No. 9 coal.

A four run series (DOE 301-304) initiated with Wilsonville solvent was made with the Pyro Mine coal and Colonial Mine coal (Ky 9/14) with and without added pyrite. No problems related to solvent quality or coking were apparent in the Merriam runs. The solvent received from Wilsonville was found to contain an unusually high hydrogen content (9.0%). This level decreased substantially during the two initial Merriam runs which were made under Wilsonville conditions. After the supply of Pyro Mine coal was exhausted, the feed was changed to Kentucky 9/14 coal from the Colonial Mine with little apparent change in results. In the final run in this series, the operating temperature was increased from the Wilsonville temperature of 441°C (826°F) to a temperature more typical of Merriam SRC I operations, 455°C (851°F). There was a further decline in hydrogen content of the recycle solvent and SRC and an improvement in desulfurization. Conditions and results for these runs are shown in Table B-1.

During the runs with Pyro Mine coal, ammonium chloride deposits formed in the vapor/liquid separation equipment. In addition, a chloride stress corrosion crack developed during the first run in this series in a line between the high temperature and intermediate temperature separators. This may have been due to use of the Pyro Mine coal or Pittsburgh Seam coal from the Powhatan No. 5 Mine which immediately preceded it.

A number of runs were also made at short residence times, both in the SRC I mode and with partial slurry recycle, but discussion of these experiments has been delayed until a later report.

II. INTRODUCTION

This report covers work at the Pittsburg & Midway Coal Mining Co.'s Merriam Laboratory on the Solvent Refined Coal (SRC) process during the period April 1, 1979 through June 30, 1979.

A. Reports of Previous Work

Early (prior to 1976) work on this project has been reported in a number of final and interim reports, a summary of which appears in Appendix A. Interim reports on selected topics are still being issued.

During the period January 1, 1976 through March 31, 1979, research on the SRC process at this facility has also been described in monthly, quarterly, and annual technical progress reports in conjunction with the Fort Lewis Pilot Plant activities. These have appeared most recently under DOE Contract EX-76-C-01-0496.

B. Current Contract Activities

The following report represents the first description of work under the new contract, DEAC-0179-ET14800, Exploratory Research on Solvent Refined Coal Liquefaction, in which the Merriam activities are reported separately.

The basic objectives of the contract research are: to improve the economics of the SRC process by developing substantial process improvements, to extend the potential application of the process, and to improve the understanding of process mechanisms. Specific objectives are as follows:

1. Evaluate the effect of recycle of selected coal solids or their products.
2. Determine the response, in both SRC I and SRC II operation, to the addition of specific chemicals and catalysts to the reactor feed.
3. Evaluate the potential usefulness of novel reactor configurations.
4. Evaluate a carefully selected group of coals in both SRC I and SRC II operation in a fully recycling unit to extend the understanding of the relationship between the physical and chemical properties of coals and the behavior of those coals under liquefaction conditions that effectively simulate large-scale operation.
5. Determine the effect of feedstock and process variables on preheater performance.
6. Explore the use of synthesis gas in the SRC II process.

The new contract involves a shift in emphasis toward basic research in coal chemistry rather than support of the Fort Lewis Pilot Plant. This change in direction may result in circulation of these reports to some people not familiar with past work at the laboratory. A brief history of the P&M Laboratory in solvent refining of coal is, therefore, included below.

III. HISTORY OF THE P&M MERRIAM LABORATORY IN SOLVENT REFINING OF COAL

The P&M Merriam Laboratory began bench scale work in the solvent refining of coal in 1962. Early work was to verify design concepts and operability for a planned one ton per day process development (PDU) which was built during 1963 and operated during much of 1964.

This was the Woodswether Road Plant in nearby Kansas City. Merriam personnel staffed the PDU facility which demonstrated the basic operability of the SRC I process on a variety of coals using anthracene oil as solvent.

In 1967 bench scale work at Merriam resumed, primarily to support the design effort for the 50 ton per day SRC Pilot Plant. Physical properties of reaction materials and products and heats of reaction were measured. Merriam staff provided the design basis for the planned SRC Pilot Plant and supervised the Stearns-Roger design effort.

From 1969 through 1971 Merriam staff also supervised the design of the PDU unit for Project Lignite, evaluated bids for construction of the unit at the University of North Dakota and administered the Project Lignite program. This program was transferred to the University of North Dakota in 1971.

Continuing bench scale liquefaction research during those years produced evidence of the importance of in situ catalysis of the SRC liquefaction process by coal minerals. This was reported in 1972 in a now much referenced paper by Wright and Severson¹.

Construction of the SRC Pilot Plant at Fort Lewis, Washington, began in 1972. The nucleus of the pilot plant staff was supplied from Merriam. Later, laboratory staff for the pilot plant were trained at the Merriam Laboratory.

Bench scale research during the period of pilot plant construction was directed toward methods for starting up the plant using anthracene oil, fluid catalytic cracker decant oil, or blends as the startup solvent. Processing techniques for equilibrating the solvent inventory were developed. Preliminary work indicating the potential usefulness of slurry recycle (SRC II) was done.

Through much of 1975 and early 1976, exploratory bench scale research into SRC II processing defined areas of operability and potential economic interest.

The later portion of 1976 and all 1977 was spent in process variable studies on several bituminous coals in the SRC II process. Operable and attractive process conditions were defined for the first three coals subsequently run in SRC II at Fort Lewis.

During 1978, the major effort returned to exploratory research aimed at potentially significant improvements in the SRC process. Construction and startup of a new, highly instrumented, continuous bench scale unit was completed.

IV. CURRENTLY REPORTED WORK

During the second quarter of 1979, activities at the Merriam Laboratory included:

- An investigation of the decline in solvent quality experienced at the Wilsonville SRC I Pilot Plant.

- An extension of earlier short residence time work to higher temperatures and shorter residence times.
- A series of eight runs investigating short residence time operation with partial slurry recycle.

Conditions and results of all runs reported this quarter are summarized in Table B-1.

V. DISCUSSION

A. Investigation of the Decline in Solvent Quality at the Wilsonville Pilot Plant

1. Background

Difficulties were experienced in runs made at the Wilsonville SRC Pilot Plant in support of the SRC I Demonstration Plant design. Solvent quality (as determined by microautoclave tests) continually declined and there was eventually coking in the preheater and dissolver.

Conditions in use at Wilsonville were as follows:

1/2 Dissolver	
Coal Rate (dissolver volume only)	50 lb/hr/ft ³
Dissolver Outlet Temperature	825°F (441°C)
Total Pressure	1700 psig
Gas Feed	20,000 SCF/ton of coal
Gas Feed Purity	85% hydrogen
Solvent/Coal Ratio	1.6-1.7
Solvent Distillation Range	450-900°F (232-482°C)
Recycle Solvent Hydrogen Content	9.0 wt %

Two things were noteworthy about these runs; the source of the coal and hydrogen content of the recycle solvent.

As the actual demonstration plant coal was not available, the coal chosen by Wilsonville was from a mine near the source for the demo plant, the Pyro Coal Company's Pyro Mine (Union County). This Kentucky No. 9 coal was expected to have a pyritic sulfur content of 1.9 wt. percent whereas the material received had only 1.1 percent. It was later learned that the supplier had substituted Kentucky 6/11 coal in place of the requested Kentucky No. 9.

The 9.0 wt. percent hydrogen level in the solvent supplied by Wilsonville appeared to be abnormally high compared to that in normal SRC I recycle solvents at Merriam and Fort Lewis. These typically contained 7.5-7.8 percent hydrogen. It was reported that the Wilsonville solvent had remained at this high level ever since a series of runs had been made earlier at 2400 psig.

2. Merriam Experiments

A series of four experiments was conducted to investigate the interaction of the Wilsonville solvent, Kentucky 6/11 or Kentucky 9/14 coals, operating temperature and pyrite addition. Run DOE 301 was made with Kentucky 6/11 coal, Wilsonville solvent and Wilsonville conditions to obtain a sample of equilibrated solvent. Run DOE 302 was made under the same conditions except for addition of pyrite to determine the effect of pyrite level on solvent quality. Run DOE 303 was made with Kentucky 9/14 coal from the P&M Colonial Mine to determine the effect of coal source, and run DOE 304 was made at the elevated temperature of 455°C which is more typical of SRC I work at Merriam. Samples of equilibrium solvent from all four runs were returned to Wilsonville for solvent quality testing.

In the initial run in this series, DOE 301, Kentucky 6/11 coal from the Pyro Mine, supplied by Wilsonville, was used. Although pyritic sulfur was not determined directly, iron analyses by our laboratory were indicative of a higher pyrite content (estimated pyritic sulfur level of 1.9%) than reported above. No problems related to solvent quality or coking were apparent during the run. Solvent was recycled and significant changes were apparent in both the standard solvent analyses and simulated distillation spectra (discussed below). Recycle solvent hydrogen content decreased from 9.0 to 8.5 percent during the run. Due to a limited supply of coal, the run was terminated before the solvent was completely equilibrated and it is likely that further changes in solvent composition would have been observed if the run had been continued.

Run DOE 302 was made under the same conditions but with pyrite added at a 0.94 wt. percent level based on coal. Again there were no difficulties related to solvent quality or coking. The hydrogen content continued downward reaching a level of 8.17 percent when the run was terminated due to depletion of the coal. The results (Table B-1) indicate increases in recycle solvent and total oil yields and a reduction in SRC yield with the pyrite addition.

The pyrite used in run DOE 302 was the same finely divided material (average particle size 0.6 μm) used in the additive studies reported last quarter².

As there had been no obvious differences in reactivity between this coal and our normal supply of Ky 9/14 coal from the Colonial Mine, run DOE 303 was made as a continuation of 302 with elimination of the added pyrite and substitution of Colonial Mine coal for the Pyro Mine coal. This run was continued for an additional two days with no apparent change in results. Specifically, there were no further changes in apparent recycle solvent composition. Yields obtained with the Colonial Mine coal were very similar to those obtained with the Ky 6/11 coal.

Because the hydrogen content of the recycle solvent had remained higher than was considered to be normal for SRC I, one further change was made. Operating temperature was increased from 411°C (825°F) to 455°C (851°F), a temperature more typical of Merriam SRC I work. This run was continued for an additional two days with compositions responding in the expected manner. There was a further decline in hydrogen content of the recycle solvent (to 7.97%) and SRC, and an improvement in desulfurization (Table B-1). There was a small loss of recycle solvent (1.1% based on MF coal), a small decrease in SRC yield, and an increase in hydrocarbon gas yield.

Semiquantitative saturate analyses indicated significant decreases in the level of saturates in recycle solvent obtained in all of the Merriam runs in comparison to the original Wilsonville solvent.

Material balances were calculated based on distillation of both filtrate and unfiltered coal solutions. Yields presented in Table B-1 are those determined by distillation of filtrate. SRC yields based on the two methods are compared below. The usual bias between the two methods is apparent with the SRC yield based on distillation of unfiltered coal solution averaging 2.4 percent (absolute) higher than that based on distillation of filtrate. The largest bias occurred in the run with added pyrite.

	SRC Yield		Distillation SRC(UFCS)-SRC(Filtrate)
	Based on Filtrate	Based on UFCS	
DOE 301	61.7	62.1	0.4
302	58.6	63.8	5.2
303	60.7	63.4	2.7
304	58.3	59.7	1.4

B. Short Residence Time SRC I at Higher Temperature and Shorter Contact Times

Discussion of these runs will be delayed until a later report.

C. Short Residence Time Operation with Partial Slurry Recycle

Discussion of these runs will be delayed until a later report.

D. Characterization of Solvents by Simulated Distillation

In section III-A it was reported that the solvent supplied by Wilsonville had a hydrogen level of 9.0 wt. percent compared to 7.5-7.8 percent in a typical SRC I recycle solvent at Merriam. It was also found that there was a significant decrease in the level of saturates in the recycle solvent obtained in all of the Merriam runs in comparison to the original Wilsonville solvent. The

simulated distillation technique (described in the first quarter 1979 report²) was used to examine further the difference between the Wilsonville solvent and equilibrated recycle solvents obtained during the runs at Merriam.

Boiling point distribution data determined by simulated distillation for the Wilsonville solvent and recycle solvents in runs DOE 301-304 are shown in Table B-2. These data are also plotted in Figure B-1. (The curves for the DOE 301-304 recycle solvents are averaged results for simulated distillations run on three or four individual recycle solvent samples taken during the latter parts of the runs). Boiling point distributions for the recycle solvents in runs DOE 301-304 are similar but there is a somewhat higher concentration of lower boiling material in the Wilsonville solvent.

Some of the spectra are reproduced in Figures B-2 through B-7. They provide evidence for solvent variations not readily apparent in tabular data summaries. The following spectra are included:

<u>Figure No.</u>	<u>Identification</u>
B-2	Saturates Extracted from Wilsonville Solvent
B-3	Wilsonville Solvent
B-4	DOE 301 - Product 51 Recycle Solvent
B-5	DOE 302 - Product 27 Recycle Solvent
B-6	DOE 303 - Product 38 Recycle Solvent
B-7	DOE 304 - Product 29 Recycle Solvent

Eight peaks are marked in each spectrum to aid in comparison. The retention times are printed on each spectrum in hundredths of minutes. Absolute retention times may vary due to column aging or use of different columns. The latter is responsible for the one minute decrease in retention times in Figures B-4 and B-5 compared to Figures B-2, B-3, B-6 and B-7.

The Wilsonville solvent used as initial feed solvent for the DOE 301-304 series has a rather different appearance (Figure B-3) than the recycle solvents obtained in the Merriam 301-304 series (Figures B-4 through B-7). In the Wilsonville solvent, peak No. 2 represents a major fraction of the material. The relative contribution of this peak is much less significant in the recycle solvent obtained in the Merriam runs. The depletion of this material may be due to reaction or its separation from the primary recycle solvent fraction in the Merriam product recovery system. By comparing the spectrum of the saturates (approximately 16%) extracted from the Wilsonville solvent (Figure B-2) to the original solvent (Figure B-3), it is evident that peak No. 2 does not represent saturated materials.

Peak No. 5 represents a minor fraction of the Wilsonville solvent. This is a much more significant component of all of the Merriam recycle solvents, however. The retention time of this peak corresponds to that of phenanthrene.

E. Maintenance and Modifications

1. Maintenance

- a. Run DOE 301 was interrupted when a crack in a 3/8 inch high pressure line between the high temperature separator (350-390°C) and the intermediate temperature separator (305-350°C) began leaking. The tubing was carrying vapor from the high temperature separator. Water was being injected above the high temperature separator to prevent plugging due to salt formation. The temperature of the line was controlled by heating tape wrapped around the tubing. The failure occurred at an area where the heating tape was not insulated because the tubing was passing through a wall and there was insufficient room for insulation.

The tubing which failed and the adjacent pieces of tubing were sent to Gulf Science & Technology Co. for failure analysis. X-ray fluorescent spectroscopy analysis showed that the tubing was Type 316 stainless steel. The three sections of tubing were examined with dye penetrants to determine if other cracks were present, but only the one area showed crack indications. Since this area did not have insulation, it is suspected that water vapor had condensed at this cooler area in the wall resulting in chloride stress corrosion cracking.

A cross section of the tubing was examined microscopically and it was found that cracking initiated at the I.D. and propagated toward the O.D. (Figure B-8). The cracking was finely branched and of a transgranular mode typical of stress corrosion cracking (Figure B-9). Scanning electron microscopy tests were conducted to detect chlorides but no chlorides could be detected in the cracks. It is believed that a high percentage of chlorides in the tubing were removed during cleaning of the tubing since these chlorides are water soluble.

It was concluded that chloride stress corrosion cracking is the probable cause of failure. The absence of insulation in the failed area probably allowed water to condense and concentrate chlorides. This was the first occurrence of stress corrosion cracking at the Merriam Laboratory.

A modification of heat tracing (see below) in this area has been performed to reduce the possibility of this problem recurring.

- b. During runs DOE 301 and 302, partial plugging in the line between the high temperature and intermediate temperature separators occurred frequently due to deposition of

ammonium chloride. This problem was overcome by intermittent water injection into the vapor line from the high temperature separator.

- c. Plugging of the on-line distillation column during runs DOE 301 and DOE 302 necessitated removal of the vessel and repacking of the column. The column apparently overheated causing coke to form in the bottom section of the vessel.
- d. The BLH Electronic's weigh system, used in formulation of feed slurry, became badly out of calibration during runs DOE 301-303 and was recalibrated during run DOE 304. Calculated slurry compositions were corrected to compensate for the poor calibration.

2. Modifications

- a. Heat tracing of an 18 inch section of line which passes through a concrete wall and connects the high temperature and intermediate temperature separators was modified to eliminate a cold spot. This is the line where water was injected to prevent ammonium chloride deposits and where the chloride stress corrosion crack mentioned above occurred. To prevent the cold spot, the line was heat traced with three heat tapes; one for each side of the wall and one in the wall.
- b. A Research Control valve was added to the liquid removal system on the refrigerated separator to eliminate the 50 to 100 psi pressure drop previously encountered while taking a sample. This control valve allows the 100 cc sample bomb to fill gradually with insignificant pressure loss to the system.

REFERENCES

1. Wright, C. H. and Severson, D. E., "Experimental Evidence for Catalyst Activity of Coal Minerals," ACS Division of Fuel Chemistry, Preprints 16, 2, 68 (1972).
2. Solvent Refined Coal (SRC) Process, Quarterly Technical Progress Report for the Period January 1, 1979-March 31, 1979, The Pittsburgh & Midway Coal Mining Co., to be published; FE/496-172.

A P P E N D I X A

Review of Merriam Laboratory Publications

Throughout the programs administered by the Office of Coal Research (OCR) and Energy Research and Development Administration (ERDA), the work of the Merriam Laboratory was reported from time to time by means of interim and final reports summarizing considerable time and effort in a single volume.

The initial development work for the SRC I process was done by the Merriam, Kansas Laboratory of the Spencer Chemical Company (which was acquired by Gulf Oil Company during the contract period) and the final report of this work was issued as Research and Development Report No. 9 to the OCR.

This contract was followed by a period of minimal activity. Work was later resumed to provide information leading to the design and eventual construction of the Solvent Refined Coal Pilot Plant at Fort Lewis, Washington. These activities are reported under R&D Report No. 53 which is divided into sections devoted to engineering, design and construction, and laboratory work. Volume II describes laboratory studies and a number of interim reports on this subject have been issued. When ERDA was established, the work continued to be reported in much the same way.

This laboratory has been directly responsible for work reported in certain interim reports and the laboratory management has supervised a number of contract research projects which have resulted in separate interim reports and a number of theses. A listing of all these reports is given in Table A-1.

The Merriam Laboratory issued Interim Report Nos. 6, 7, 8 and 21, as well as R&D Report No. 9, and the subject matter of these is summarized below.

Research and Development Report No. 9 describes the operation of a one ton per day continuous plant to produce Solvent Refined Coal. The Merriam Laboratory was involved in the analytical work required to operate the continuous unit and in the operation of a batch autoclave facility used in support of the effort.

When work was later resumed, these facilities were dedicated to studies necessary to support the design of the Fort Lewis Pilot Plant. This required a number of reaction studies which were done in a batch autoclave and which are reported in Interim Report No. 6. The objectives at this time were better characterization of materials and the development of background information leading to improved control chemistry for the proposed pilot plant. In addition, the use of various coals, solvents and reducing gas systems was studied.

Interim Report No. 7 describes the construction and operation of a laboratory scale continuous reactor which was used to follow up the autoclave work. Anthracene oil or hydrogenated anthracene oil was used as a solvent and several coals were reacted either with hydrogen or carbon monoxide as reducing gas. Some of the work was conducted in cooperation with the University of North Dakota which was interested in processing of lignite. Considerable emphasis was given to characterization of materials and analytical techniques to develop better methods for use at the pilot plant.

Interim Report No. 8 describes the operation of the laboratory reactor using a petroleum derived start up solvent. It was established in this work that the coal derived solvent would be less hydrogenated (more aromatic) than the catalytic cracker decant oil used as the start up solvent. Previous work had shown that starting with a very aromatic solvent, such as anthracene oil, would result in the solvent gaining hydrogen. Thus, the coal derived oil was shown to be of intermediate aromaticity between the coal pyrolysis products and petroleum derived materials.

Interim Report No. 21 was devoted to the development of several laboratory scale reactors and also to exploration of techniques using recycle of coal solution in various concentrations as a component of the feed slurry (SRC II). At the beginning of trials with recycle of unfiltered coal solution, the reactors available were not properly designed to allow a convincing steady state result. Considerable trial and error work was done to find a small, practical reactor for this purpose and to develop proper heat tracing and auxiliary separation equipment to allow these complex and time consuming experiments to be completed successfully.

Since January 1, 1976, the primary method of reporting has changed to a system of monthly, quarterly and annual reports. Until March 1979, these contained sections devoted to the work at the Fort Lewis (and more recently, Harmarville, Pennsylvania) pilot plant(s) and to the work at the Merriam Laboratory. A listing of these reports is given in Table A-2.

For further reference, a list of SRC papers presented by P&M personnel is included in Table A-3.

TABLE A-1

SOLVENT REFINED COAL FINAL AND INTERIM REPORTS

1. Solvent Processing of Coal to Produce a Deashed Product, R&D Report No. 9, Kloepper, et al, Gulf Oil Corp., Merriam, Kansas - NTIS #PB167-809.
2. Development of a Process for Producing an Ashless, Low-Sulfur Fuel from Coal.

OCR Research & Development Report No. 53, Contract No. 14-01-0001-496 (The Pittsburg & Midway Coal Mining Co., Kansas City, MO)

- a. Interim Report #1 (Vol. I - Engineering Studies - Part 1 Economic Evaluation of a Process to Produce Ashless, Low-Sulfur Fuel from Coal) GPO #163.10:53/INT 1, NTIS #PB234-040.
- b. Interim Report #2 (Vol. III, Pilot Plant Development Work, Part 1, Design of Pilot Plant) GPO #163.10:53/INT 2, NTIS #PB234-039.
- c. Interim Report #3 (Vol. I, Part 2, COG Refinery Economic Evaluation - Phase I) 1974, GPO #163.10:53/INT 3, NTIS #PB234-043.
- d. Interim Report #4 (Vol. I, Part 3, COG Refinery Economic Evaluation - Phase II), 1974, GPO #163.10:53/INT 4, NTIS #PB234-043.
- e. Interim Report #5 (Vol. I, Part 4, Impact of the SRC Process) GPO #163.10:53/INT 5, NTIS #PB234-038.
- f. Interim Report #6 (Vol. II - Laboratory Studies - Part I, Autoclave Experiments), 1975, GPO #163.10:53/INT 6, NTIS #PB236-305.

ERDA Contract No. E(49-18)-496 (Pittsburg & Midway Coal Mining Co., Kansas City, Mo.)

- g. Interim Report #7 (Vol. II - Laboratory Studies - Part 2, Continuous Reactor Experiments Using Anthracene Oil Solvent). Not available at this time. FE-496-T4.
- h. Interim Report #8 (Vol. II, Part 3, Continuous Reactor Experiments Using Petroleum Derived Solvents), NTIS #FE-496-T1.
- i. Interim Report #9 (Vol. III, Part 2, Construction of the Pilot Plant), NTIS #FE-496-T2.

- j. Interim Report #10 (Vol. IV - Product Studies - Part 2, An Annotated Bibliography on Mineral Fiber Production from Coal Minerals). Not available at this time.
- k. Interim Report #11, Vol. IV, Part 3, Products from Coal Minerals). Not available at this time.
- l. Interim Report #12 (Vol. IV, Part 4, Sulfur Removal from Coal Minerals). OCR R&D Report No. 53, NTIS #PB237-765.
- m. Interim Report #13 (Vol. IV, part 5, Developmental and Rate Studies in Processing of Coal Minerals). OCR R&D Report No. 53, NTIS #PB237-766.
- n. Interim Report #14 (Vol. IV, Product Studies, Part 5 - Establishment of Equipment and Its Operation for Hydro-treating of a Coal-Derived Liquid). FE-496-T5.
- o. Interim Report #15 (Volume IV - Product Studies - Part 6 - Hydrogenitrogenation of a Coal-Derived Liquid). FE-496-T7.
- p. Interim Report #16 (Volume IV - Product Studies - Part 7 - Effect of Catalyst Pore Size on Hydrodesulfurization of Coal-Derived Liquids). FE-496-T6.
- q. Interim Report #17 (Volume IV - Product Studies - Part 8 - A Preliminary Study of Non-Catalyzed Hydrodesulfurization of Coal-Derived Liquids). FE-496-T8.
- r. Interim Report #18 (Volume IV - Oklahoma State University, Ahmed's Thesis, Part 9). FE-496-T9.
- s. Interim Report #19 (Volume IV - Oklahoma State Final Report). FE-496-T10.
- t. Interim Report No. 20 (Volume IV, Part 11, Moh's Thesis - Washington State University), 1979. FE-496-T11.
- u. Interim Report No. 21 (Volume II, Part 4 - Lab Report, Wright), 1978. FE-496-T12.
- v. Interim Report No. 22 (Volume III, Part 3 - Pilot Plant Report Startup & Operation), 1977. FE-496-T13.
- w. Interim Report No. 23 (Volume IV, Part 12 - Biswas Thesis - Washington State University). FE-496-T14.
- x. Interim Report No. 24 (Volume III - Part 4 - Pilot Plant Industrial Hygiene & Medical Surveillance Program), 1978. FE-496-T15.

- y. Interim Report No. 25, (Volume III - Part 5 - Pilot Plant Environmental Studies - Baseline & Investigative, 1978. FE-496-T16.
- z. Interim Report #26 (Volume III - Part 6 - Fate of Trace Elements in the SRC Process), 1978. FE-496-T17.
- aa. Interim Report No. 27 (Vol. III: Pilot Plant Development Work - Part 5: Environmental Program), 1979. FE/496-T18.
- bb. Interim Report No. 28 (Vol. III: Pilot Plant Development Work - Part 4: Industrial Hygiene, Clinical and Toxicological Programs), 1979. FE/496-T19.

TABLE A-2

SRC QUARTERLY & ANNUAL TECHNICAL REPORTS

January - March 1976 Quarterly Report	FE-496-126
April - June 1976 " "	FE-496-127
July - September 1976 " "	FE-496-129
Annual Report 1976	FE-496-131
January - March 1977 Quarterly Report	FE-496-134
April - June 1977 " "	FE-496-141
July - September 1977 " "	FE-496-143
Annual Report 1977	FE-496-149
January - March 1978 Quarterly Report	FE-496-155
April - June 1978 Quarterly Report	FE-496-157
July - September 1978 Quarterly Report	FE-496-160
Annual Report 1978	FE-496-170
January-March 1979 Quarterly Report	FE-496-172

TABLE A-3

SRC Papers Presented by P&M Personnel

1. Techniques Employed in Designing a Process Development Plant to Produce Low-Ash Fuel From Coal - presented at Symposium on Coal Processing at the National Meeting of AIChE in Memphis in February, 1964 by D. L. Kloepper and T. F. McCall.
2. The Spencer De-Ashed Coal Process - presented to the Coal Technology Group of the American Chemical Society in Pittsburgh, Pa. on May 27, 1964 by Willard C. Bull.
3. The Spencer De-Ashed Coal Process - presented at Annual Off-the-Record Meeting of the AIME in Pittsburgh, Pa. on November 6, 1964 by Willard C. Bull.
4. Coal Liquefaction - The Spencer De-Ashed Coal Process - presented at the 6th Annual Pacific Northwest Regional Meeting of the AIChE on October 8, 1965 by Willard C. Bull at Seattle, Wa.
5. The Spencer De-Ashed Coal Process - presented to the Southeast Kansas Section of The American Chemical Society, on November 11, 1965 by Willard C. Bull.
6. Coal Research - presented to the Rocky Mountain Electric League, Denver, Colorado on April 18, 1966 by Willard C. Bull.
7. The Pittsburg & Midway Coal De-Ashing Process - presented at the Rocky Mountain Electrical League Meeting, Boulder, Col. on April 24, 1967 by Willard C. Bull.
8. Chemical Engineering Aspects of Coal Solution - presented at The Pennsylvania State University Seminar on Nature of Coal & Developments in the Technology of its Utilization, October 30 - November 3, 1967 by Willard C. Bull.
9. Separation of Fine Solids from Liquids at Elevated Temperature and Pressure - presented at AIME Meeting, New York, N.Y. on February 27, 1968 by B. K. Schmid.
10. Design of a Pilot Plant to Produce Ashless, Low-Sulfur Fuel from Coal - presented at 65th National Meeting of American Institute of Chemical Engineers on May 4-7, 1969 by V. L. Brant, Stearns-Roger Corp. and B. K. Schmid, P&M Coal Mining Co., at Cleveland, Ohio.
11. Processes for Coal Liquefaction and The Production of Low Sulfur Boiler Fuels - presented at Oklahoma State University, May 1971 by Willard C. Bull.
12. The Liquefaction of Lignite - presented at North Dakota Oil & Gas Association, Bismarck, N.D. on September 13, 1971 by Willard C. Bull.

13. Production of Ashless, Low-Sulfur Boiler Fuels from Coal - presented at Symposium on Pollution Control, Division of Fuel Chemistry, American Chemical Society in Washington, D.C. on September 12-17, 1971 by B. K. Schmid and W. C. Bull.
14. Ashless, Low-Sulfur Boiler Fuel from Coal - presented at the 10th Conference on Air Pollution Control, Purdue University, Lafayette, Indiana on October 13, 1971 by Willard C. Bull.
15. Use of a Refinery LP Model to Determine Value of Coal Derived Liquid - presented at ACS Meeting in Boston, April 1972 by B. K. Schmid, Marshall E. Frank and Stephen N. Rothman.
16. Experimental Evidence for Catalyst Activity of Coal Minerals - presented at ACS Meeting in Boston, April 1972 by C. H. Wright and D. E. Severson.
17. Economic Evaluation and Process Design of the Coal-Oil-Gas (COG) Refinery - presented at the Synthetic Fuels from Coal Conference, Oklahoma State University, May 1-2, 1972 by B. K. Schmid, Allan M. Benton and Marshall E. Frank.
18. Economic Evaluation and Process Design of a Coal-Oil-Gas (COG) Refinery - presented at Symposium on Conceptual Plants for the Production of Synthetic Fuels from Coal, 65th Annual Meeting, American Institute of Chemical Engineers, New York, N.Y. on November 26-30, 1972 by B. K. Schmid and M. E. Frank.
19. The Pittsburg & Midway Coal Mining Co.'s Solvent Refined Coal Process - presented at AIME Annual Meeting, Chicago, Illinois on February 25 - March 1, 1973 by C. D. Atkinson.
20. Environmental Aspects of the SRC Process - presented at EPA Symposium on May 14, 1974 at St. Louis, Missouri by C. R. Hinderliter.
21. The Solvent Refined Coal Process - presented at the Symposium on Coal Gasification and Liquifaction: Best Prospects for Commercialization, University of Pittsburgh, Pittsburgh, Pa. on August 6-8, 1974 by B. K. Schmid.
22. Evolution of Steady State Process Solvent in the Pittsburg & Midway Solvent Refined Coal Process - presented at Symposium on Coal Processing, American Institute of Chemical Engineers, Salt Lake City, Utah on August 20, 1974 by B. P. Anderson.
23. Research Needs for Project Independence - presented at FEA Hearings on Project Independence, Seattle, Wa. on September 5, 1974 by Willard C. Bull.
24. Current Status of Solvent Refined Coal Project - presented at the Panel Discussion on Clean Energy from Coal: Part III, Conversion to Liquids and Clean Solids, 67th Annual Meeting of American Institute of Chemical Engineers in Washington, D.C. on December 1-5, 1974 by B. K. Schmid.

25. Coal Desulfurization in the P&M Solvent Refining Process - presented at the Spring Meeting of Division of Fuel Chemistry, American Chemical Society, in Philadelphia, Pa. on April 6-11, 1975 by R. P. Anderson and C. H. Wright.
26. Studies with Kentucky No. 9 Coal in the Solvent Refined Coal Process - presented at the Catalytic Conversion of Coal Symposium, 41st Annual Chemical Engineering Meeting at Carnegie-Mellon University on April 22, 1975 by C. H. Wright and R. P. Anderson.
27. Recycling Solvent Techniques for the SRC Process - article published in the Chemical Engineering Progress, April 1975, Vol. 71, No.4. by R. P. Anderson.
28. Status of the SRC Project - article published in the Chemical Engineering Progress, April 1975, Vol. 71, No. 4 by B. K. Schmid.
29. Progress Report of Pilot Plant Work on the Solvent Refined Coal Process - presented at the Second Annual Conference on Coal Gasification and Liquefaction at the University of Pittsburgh, Pittsburgh, Pa. on August 5-7, 1975 by G. E. Chenoweth.
30. Solvent Refined Coal -- A Pilot Plant Report - presented at the 1975 Joint Power Generation Conference, Portland, Oregon on September 28-October 2, 1975 by G. R. Pastor.
31. Operation of the SRC Pilot Plant - presented at the 68th Annual Meeting of the AIChE, Los Angeles, CA. in November 1975 by G. R. Pastor.
32. Environmental Aspects of the SRC Process - presented at the 2nd Symposium on Environmental Aspects of Fuel Conversion Technology (EPA), Hollywood, FL. in December 1975 by R. E. Perrussel.
33. The SRC-II Process - presented at 3rd Annual Coal Gasification & Liquefaction Conference at the University of Pittsburgh on August 3-5, 1976 by B. K. Schmid and D. M. Jackson.
34. Environmental Controls of the Solvent Refined Coal Pilot Plant - presented at the 69th Annual Meeting of AIChE in Chicago, IL on November 28, 1976 by R. E. Perrussel and J. V. Herbison.
35. Recycle SRC Processing for Liquid & Solid Fuels - presented at the 4th Int'l Conference on Coal Gasification, Liquefaction and Conversion to Electricity, University of Pittsburgh on August 2, 1977 by B. K. Schmid and D. M. Jackson.
36. The SRC II Process - presented at the 1977 Annual Meeting of the American Chemical Society, Chicago, IL on August 28, 1977 by R. P. Anderson.

37. Coal Liquefaction and the Development of the SRC II Process - presented at the Washington State University, Department of Chemistry, Pullman, Washington, February 15, 1978, by D. K. Schmalzer.
38. SRC II Review of Development and Status - presented at the Fifth Annual Energy Technology Conference, Washington, D. C., February 28, 1978, by D. M. Jackson and B. K. Schmid.
39. An Overview of SRC Process Development - presented at DOE Project Review Meeting, Sandia Laboratories, Albuquerque, N.M., on June 15, 1978 by D. K. Schmalzer.
40. An Overview of SRC Process Development and Commercial Scale Development of the SRC II Process - August 1978 by J. C. Koenig and D. K. Schmalzer
41. Commercial Scale Development of SRC II Process - presented at the 5th Annual Conference on Coal Gasification, Liquefaction and Conversion to Electricity, University of Pittsburgh on August 1-3, 1978 by D. M. Jackson and B. K. Schmid.
42. Operation of the Ft. Lewis, Washington, Solvent Refined Coal (SRC) Pilot Plant in the SRC I and SRC II Processing Modes - presented at the 13th Inter-Society Energy Conversion Engineering Conference, August 20-25, 1978 by R. D. Moschitto.
43. The Solvent Refined Coal Pilot Plant Health Programs & Observations - presented at Electric Power Research Institute, Pacific Grove, California, on September 26, 1978 by D. K. Schmalzer.
44. SRC Pilot Plant Health Programs & Observations - presented at EPRI Workshop, Pacific Grove, CA, September 26, 1978, D. K. Schmalzer.
45. Environmental Assessment of SRC II - An Update - April 1979, C. R. Moxley & D. K. Schmalzer,

A P P E N D I X B

Tables and Figures

TABLE B-1

Summary of Process Conditions, Yields and Product Analyses

	DOE 301	DOE 302	DOE 303	DOE 304
<u>Conditions</u>				
Coal	← Ky 6/11 ^a →		← Ky 9/14 ^b →	
Solvent	← Wilsonville →			
Dissolver	← DOE 1 ^c →			
Nominal slurry residence time, min.	32.4	32.4	32.0	32.4
Coal feed rate, lb/hr/ft ³	50.0	48.0	48.4	49.6
Average dissolver temperature, °C	440	440	440	452
	824	824	824	846
Dissolver pressure, psig	1445	1445	1445	1445
H ₂ Feed				
Wt % based on slurry	1.72	1.72	1.70	1.72
MSCF/ton of coal	17.9	17.9	17.8	17.3
Additive	--	Pyrite	--	--
Additive level (% based on MF coal)	--	0.94	--	--
Slurry Formulation				
% Coal	36.2	36.2	36.3	37.7
% Recycle Solvent	63.8	63.4	63.7	62.3
% Additive	--	0.4	--	--
<u>Yields</u>				
H ₂	5.6	4.6	6.1	5.6
CO	0.1	0.1	0.2	0.3
CO ₂	0.5	0.5	1.1	1.1
H ₂ S	1.3	1.4	1.3	1.2
NH ₃	0.1	0.1	0.1	0.1
C ₁	2.0	1.7	2.0	2.9
C ₂	1.2	1.2	1.3	1.5
C ₃	0.7	0.7	0.7	0.9
C ₄	0.3	0.3	0.3	0.3
Total C ₁ -C ₄	4.2	3.9	4.3	5.6
Naphtha, C ₅ -193°C (380°F)	6.9	6.2	6.7	7.6
Wash Solvent, 193-249°C (380-480°F)	6.7	6.9	6.6	7.5
Recycle Solvent, >249°C (>480°F)	0.0	5.4	(1.3) ^d	(1.1)
Total Oil	13.6	18.5	12.0	14.0
SRC	61.7	58.7	60.7	58.3
Insoluble Organic Matter	5.1	4.9	6.1	5.0
Ash	10.0	9.9	10.0	10.0
Total	102.2	102.6	101.9	102.1
H ₂ Reacted, gas balance	2.2	2.3	1.9	2.1
Pyrite conversion byproducts ^e	--	0.3	--	--
<u>Product Analyses</u>				
Recycle Solvent Analyses				
% C	87.57	87.62	87.75	87.85
% H	8.46	8.16	8.14	7.97
% S	0.60	0.64	0.65	0.57
% N	0.79	0.84	0.82	0.93
% U (by difference)	2.58	2.74	2.64	2.60
Specific Gravity	1.028	1.042	1.040	1.041
SRC Analyses				
% C	87.46	87.21	86.89	87.43
% H	5.81	5.93	5.78	5.49
% S	0.89	0.86	0.93	0.78
% N	1.95	2.07	2.03	2.12
% Ash	0.31	0.24	0.22	0.20
% O (by difference)	3.58	3.69	4.15	3.98

a) Pyro Mine, Union County. b) Lot 7, P&M Colonial Mine, Hopkins County.

c) Volume = 1035 cc, 1.1" I.D. by 72" long. d) Losses shown in brackets.

e) Pyrite fed less ash from pyrite.

TABLE B-2
DOE 301-304

Simulated Distillation (by Gas Chromatography) of Recycle Solvents

	Dry Wil- sonville	301	301	301	301	301	301
Fraction Boiling	Solvent	P 6	P 17	P 27	P 33	P 43	P 51
Below, %	Temp °C	Temp °C	Temp °C	Temp °C	Temp °C	Temp °C	Temp °C
IBP	147.8	93.57	46.25	79.94	39.95	66.30	59.42
5	213.1	212.4	212.3	212.4	205.4	212.3	219.5
10	234.5	235.5	243.1	243.0	235.1	235.5	243.2
15	239.4	251.0	251.2	251.2	251.0	251.2	251.4
20	251.7	259.5	259.8	259.8	259.5	268.0	268.4
25	258.5	268.1	268.5	275.8	268.4	275.9	283.1
30	265.5	275.8	283.1	283.2	283.1	283.3	290.8
35	273.0	283.2	290.7	290.8	290.7	291.0	298.8
40	279.2	290.7	298.8	298.9	298.8	299.1	307.2
45	284.4	298.7	307.1	307.2	307.1	307.4	315.7
50	292.3	307.1	315.6	315.7	315.7	315.9	323.7
55	300.6	315.5	323.7	323.8	323.8	331.5	331.7
60	308.1	323.6	331.7	331.8	331.8	339.4	339.7
65	318.4	331.6	339.8	347.3	347.3	347.4	347.6
70	329.8	339.7	355.0	355.2	355.2	355.3	362.7
75	337.8	355.0	363.0	363.1	370.5	370.4	370.6
80	348.0	363.0	378.2	378.4	378.5	378.4	378.5
85	363.5	378.4	393.7	393.9	394.0	393.8	393.9
90	389.0	394.1	409.8	410.0	417.6	409.8	409.8
95	420.4	425.5	433.7	441.5	441.7	433.6	433.5
FBP	487.5	473.5	511.6	503.8	503.8	503.9	496.1

	302	302	302	302	302	303	303
Fraction Boiling	P 14	P 18	P 25	P 27	P 32&35	P 24	P 30
Below, %	Temp °C	Temp °C	Temp °C	Temp °C	Temp °C	Temp °C	Temp °C
IBP	79.78	59.63	39.87	59.49	46.37	97.13	112.3
5	212.2	219.7	212.3	205.4	198.7	221.7	222.3
10	235.5	243.5	243.3	235.3	235.2	245.0	244.1
15	251.3	259.8	259.8	251.3	251.2	260.7	256.6
20	268.2	276.0	276.1	268.4	268.2	269.6	270.6
25	276.2	283.4	283.5	283.2	283.1	283.9	281.8
30	283.5	298.7	298.8	291.0	290.8	291.7	291.2
35	298.7	307.1	307.2	306.9	298.9	299.9	301.7
40	307.1	315.5	315.7	315.4	307.3	308.1	310.0
45	315.5	323.6	323.8	323.5	315.8	316.2	317.4
50	323.5	331.6	331.7	331.5	331.4	324.1	326.2
55	331.5	339.5	339.8	339.5	339.4	332.0	333.9
60	339.4	347.5	347.6	347.4	347.4	339.9	343.0
65	347.3	355.3	355.4	355.2	355.2	347.8	351.9
70	355.1	363.1	370.5	363.0	363.1	362.5	361.3
75	363.0	378.2	378.3	370.8	378.3	370.5	371.0
80	370.8	386.2	386.2	386.0	386.3	378.6	381.7
85	386.2	401.7	401.8	394.0	401.9	393.7	393.5
90	401.9	417.5	417.6	409.8	425.5	402.3	408.7
95	425.9	433.8	441.5	433.7	449.9	425.7	429.9
FBP	496.4	488.6	496.1	519.3	526.9	474.0	482.2

TABLE B-2

(Continued)

	303	303	303	304	304	304	304
Fraction Boiling	P 35	P 36	P 38	P 15	P 20	P 29	P 36
Below, %	Temp °C	Temp °C	Temp °C	Temp °C	Temp °C	Temp °C	Temp °C
IBP	69.14	104.1	56.35	155.5	147.6	170.1	75.97
5	221.9	229.4	221.7	229.4	214.1	221.6	229.3
10	245.4	252.9	245.3	245.3	237.1	237.6	251.9
15	261.5	261.5	261.4	253.3	252.2	253.1	261.2
20	277.1	277.2	277.1	269.3	260.7	261.6	276.4
25	284.6	291.9	284.6	277.0	269.6	277.1	284.2
30	299.9	300.0	300.0	284.4	283.5	284.5	292.3
35	308.2	308.3	308.3	292.2	291.2	292.3	307.1
40	316.3	316.4	316.4	300.3	299.4	308.0	315.2
45	324.2	324.3	324.3	308.3	307.7	316.2	316.4
50	332.1	332.1	332.1	316.4	315.8	316.6	324.4
55	339.9	339.9	339.9	324.3	323.8	331.9	332.0
60	347.7	347.8	347.8	332.2	331.8	332.3	339.9
65	355.5	355.5	355.6	340.2	339.9	347.6	347.8
70	363.3	363.3	363.3	355.0	354.4	355.4	355.7
75	371.1	371.1	378.5	363.0	362.7	363.2	369.9
80	386.2	386.2	386.3	371.2	377.5	371.1	378.0
85	394.3	394.3	394.3	386.3	386.3	386.3	386.4
90	410.0	410.0	410.1	402.1	402.3	401.9	401.7
95	426.1	426.0	433.8	425.8	432.7	418.1	418.2
FBP	474.3	474.2	482.1	481.5	488.5	466.3	466.3

	304	304
Fraction Boiling	P 37	P 38
Below, %	Temp °C	Temp °C
IBP	62.80	155.4
5	221.5	221.1
10	237.6	237.5
15	253.2	252.9
20	269.7	269.2
25	284.3	277.2
30	292.1	284.6
35	300.2	299.5
40	308.4	307.8
45	316.5	315.8
50	324.4	316.8
55	332.2	331.5
60	340.1	332.2
65	347.9	347.1
70	363.0	354.9
75	370.8	362.8
80	378.7	370.8
85	394.0	385.8
90	402.4	394.4
95	426.1	418.0
FBP	474.4	473.6

FIGURE B-1
SIMULATED DISTILLATION CURVES FOR RECYCLE SOLVENTS

1079
 1129
 1179
 1229
 1279
 1329
 1379
 1429
 1479
 1529
 1579
 1629
 1679
 1729
 1779
 1829
 1879
 1929
 1979
 2029
 2079
 2129
 2179
 2229
 2279
 2329
 2379
 2429
 2479
 2529
 2579
 2629
 2679
 2729
 2779
 2829
 2879
 2929
 2979
 3029
 3079
 3129
 3179
 3229
 3279
 3329
 3379
 3429
 3479
 3529
 3579
 3629
 3679
 3729
 3779
 3829

FIGURE B-2
 Simulated Distillation
 Saturates Extracted from Wilsonville Solvent

% OFF	TEMP
.5	157.3
5	246.6
10	265.5
15	278.3
20	285.9
25	293.6
30	301.5
35	309.7
40	317.8
45	325.6
50	333.5
55	341.3
60	349.3
65	357.3
70	365.5
75	380.4
80	395.9
85	412
90	428.7
95	450.7
99.5	506.9

FIGURE B-3
Simulated Distillation
Wilsonville Solvent

FIGURE B-4
Simulated Distillation
DOE 301
Product 51 - Recycle Solvent

FIGURE B-5
Simulated Distillation
DOE 302
Product 27 - Recycle Solvent

Spectra-Physics

FIGURE B-6
Simulated Distillation
DOE 303
Product 38 - Recycle Solvent

Spectra-Physics

FIGURE B-7
 Simulated Distillation
 DOE 304
 Product 29 - Recycle Solvent

Spectra-Physics

I.D.

O.D.

10X

Figure B-8

Cross section of tubing showing cracks initiating at the I.D.

I.D.

O.D.

250X

Figure B-9

Cross section of tubing showing transgranular cracking.