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EXPLORATORY RESEARCH ON SOLVENT REFINED COAL LIQUEFACTION

Annual Technical Progress Report for the Period January 1—December 31, 1980

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April 1981

Work Performed Under Contract No. AC22-79ET14800

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

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EXPLORATORY RESEARCH
ON
SOLVENT REFINED COAL LIQUEFACTION

ANNUAL TECHNICAL PROGRESS REPORT
FOR THE PERIOD
JANUARY 1, 1980 THROUGH DECEMBER 31, 1980

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

April 1981

PREPARED FOR THE
U. S. DEPARTMENT OF ENERGY
DIVISION OF COAL CONVERSION AND UTILIZATION
UNDER CONTRACT DE-AC22-79ET14800

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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 during 1980. Six coals and a coal blend, representing the Interior, Rocky Mountain, Eastern and Northern Great Plains Provinces, were processed in the SRC I and SRC II modes to study the relationship between coal properties and liquefaction behavior. Disposable catalysts and specific compounds were added to Loveridge, Kaiparowits and Blacksville No. 2 coals during SRC II mode liquefaction. Kentucky 9/14, Indiana V and Loveridge coals were processed at short residence times (4-9 minutes) in the SRC I mode to evaluate the effects of temperature, pressure, residence time and disposable catalyst addition. Finally, coal from the Powhatan No. 1 Mine was evaluated as a feedstock for major coal liquefaction facilities.

I. SUMMARY

The activities of the Merriam Laboratory during 1980 were in four major areas:

1. Study of the relationship between coal properties and liquefaction behavior.
2. Addition of catalytic materials including both disposable catalysts and higher priced specific compounds.
3. Investigation of short residence time coal liquefaction.
4. Evaluation of coals as potential feedstocks for major liquefaction facilities.

A. Coal Properties and Liquefaction Behavior

Six coals were selected for this study:

Company and Mine	State	Seam
Amax Belle Ayr	WY	Smith-Roland
Mono Power (Kaiparowits Plateau)	UT	Red
Energy Fuels Energy Mine No. 1	CO	Wadge
Amax Ayrshire	IN	Indiana VI
Consol Georgetown No. 24	OH	Ohio No. 8 (Pittsburgh)
Consol Ireland	WV	Pittsburgh

as well as a blend of Ireland and Blacksville No. 2 coals. The Ireland Mine coal was processed after blending to ash levels of 10, 12, 14 and 16 wt %. The results with the first three coals were given in the first quarter 1980 report* and with the remainder in the second quarter 1980 report**.

Processing of the three western coals, Belle Ayr, Kaiparowits and Wadge seam, all of which are low in iron (0.2-0.4 wt %, dry coal basis), does not appear to be feasible at normal conditions without

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* "Exploratory Research on Solvent Refined Coal Liquefaction," Technical Progress Report for the Period January 1, 1980 through March 31, 1980; FE/14800-17.

** "Exploratory Research on Solvent Refined Coal Liquefaction," Technical Progress Report for the Period April 1, 1980 through June 30, 1980; FE/14800-21.

added catalyst. For all of the coals, total oil or SRC + IOM yields could be correlated with the composition of the coal and iron level in the feed slurry. The reactivity generally increased as the organic oxygen and sulfur content of the coal increased. Yields also improved as the iron concentration in the feed slurry increased.

B. Addition of Catalytic Materials

Work in this area was divided into study of disposable catalysts (iron compounds) and use of specific additives (molybdenum and promoted molybdenum-alumina catalysts).

1. Disposable Catalyst Studies

Three series of runs were made during the year investigating the use of disposable catalysts (other than in the study of coal properties discussed above). In the first study, detailed in the second quarter 1980 report, coal from the Loveridge Mine was processed at 455°C, 1800 psig and 1.0 hour residence time to compare the catalytic activity of pyrite (FeS_2) with ferric oxide (Fe_2O_3).

Total oil yield with pyrite was about 3% higher than with ferric oxide which was, in turn, about 1% higher than for a control run without additive. There were correspondingly lower SRC and IOM yields. Yields and hydrogen consumptions were essentially the same for runs catalyzed with pyrite or an equal iron blend of FeS_2 and Fe_2O_3.

The hydrogen level in the heavy distillate was a little higher with pyrite and the FeS_2/Fe_2O_3 blend than with Fe_2O_3 alone (7.6-7.7% vs 7.4%), although sulfur and nitrogen levels were similar for all three runs.

In a second study, described in the third quarter 1980 report*, Loveridge Mine coal was processed at 450°C, 2250 psig and 1.0 hour residence time in the SRC II mode. Yields and hydrogen consumptions were essentially the same for runs with added pyrite (FeS_2) or ferric oxide (Fe_2O_3). The yields obtained with Loveridge coal and 5 wt % added pyrite were similar to those obtained with the other Pittsburgh seam coals (Georgetown and an Ireland/Blacksville blend) studied under these conditions without added catalyst.

When Loveridge coal was processed in the SRC I mode at 450°C, 1900 psig and 26 minutes residence time, there was a significant increase in total oil yield (3.7% based on MF coal) with a corresponding decrease in SRC yield when pyrite was added to the feed slurry. There was a smaller effect with a FeS_2/Fe_2O_3 combination and little or no effect when ferric oxide alone was added.

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* "Exploratory Research on Solvent Refined Coal Liquefaction," Technical Progress Report for the Period July 1, 1980 through September 30, 1980; February 1981, FE/14800-25.

2. Specific Additives Studies

Two series of runs were made with molybdenum based catalysts. In the first study, discussed in the third quarter 1980 report*, two runs were conducted in the SRC II mode at 450°C, 2250 psig and 1.0 hour residence time with Loveridge coal containing 0.7 and 0.4 wt % molybdenum by aqueous impregnation.

Slightly higher oil yields and slightly lower hydrocarbon gas, SRC and IOM yields were obtained with 0.7 wt % molybdenum than with 5 wt % added pyrite or 3.3 wt % added ferric oxide. The lot of pyrite used in this comparison appears to be relatively low in activity, however, compared to some other pyrite samples. Higher oil yields and lower SRC and IOM yields were achieved with 0.7 wt % molybdenum on coal than with 0.4 wt %, although the differences were slight. The sulfur levels in the heavy distillate and distillation residue from the Mo catalyzed runs were much lower than in the runs catalyzed by iron compounds. Other analyses were roughly the same for the two catalyst types.

In work reported this quarter, two runs were conducted at 450°C, 2250 psig and 1 hour residence time with Loveridge coal to investigate the addition of molybdenum-alumina catalyts. Addition of 0.34 wt % Mo dispersed on alumina and promoted by nickel to Loveridge coal gave a 3.6 wt % higher oil yield and a 3.7 wt % lower SRC yield than the same amount of molybdenum added by aqueous impregnation of the coal. Molybdenum-alumina catalysts promoted by either nickel or cobalt gave essentially the same results and the yields generally improved as the molybdenum level was increased from 0.04 to 0.34 wt %, based on coal. The product sulfur and nitrogen levels were, in general, reduced significantly by the molybdenum-alumina catalysts which were originally developed as HDS (hydrodesulfurization) catalysts.

C. Short Residence Time Coal Liquefaction

Twenty short residence time SRC I runs were conducted with Kentucky 9/14 and Indiana V coals to extend earlier short residence time studies to higher pressures. Nominal temperatures of 460 to 487°C, pressures of 2000 to 3000 psig and residence times of 4 to 9 minutes were investigated. In addition, two runs were made with Loveridge coal for a preliminary investigation of disposable catalyst addition in the short residence time mode. Results are reported this quarter.

The unit was operated without difficulty during all of the runs, filterable products were obtained and no coke build-up in the

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* "Exploratory Research on Solvent Refined Coal Liquefaction," Technical Progress Report for the Period July 1, 1980 through September 30, 1980; February 1981, FE/14800-25.

preheater or dissolver was observed. Excess recycle solvent was generally obtained at 480°C, 3000 psig and 6 to 8 minutes residence time.

A total of 16 experiments with Indiana V coal were conducted during this study, including a 2^3 plus midpoint factorial to evaluate the effects of temperature, pressure and residence time. Increasing temperature in the range of 460 to 480°C generally increased hydrocarbon gas and total oil yields while SRC yield was decreased. Increasing pressure in the range of 2000 to 3000 psig increased recycle solvent and total oil production as well as hydrogen consumption. The effect of residence time in the range of 4 to 8 minutes was confounded by the small dissolver cross-section and high gas flow rates. It appears that the actual slurry residence time was somewhat below the nominal value due to "bridging" of the gas phase in the dissolver. Oil yields would probably be larger and SRC and IOM yields smaller if a larger diameter dissolver were used. Increasing nominal residence time resulted in increased hydrocarbon gas yield and hydrogen consumption and decreased oil and IOM yields.

At 480°C, 3000 psig and 8 minutes residence time, yields were generally improved by solvent recycle while residence time in the preheater between 5 and 8 minutes had little effect.

The run conditions had little effect on recycle solvent analyses, but the sulfur level in the SRC was reduced significantly by increasing dissolver temperature and/or residence time. The sulfur level in the SRC was below 0.9 wt % for all runs.

At 460°C, 2000 psig and 9 minutes residence time with Loveridge coal, a small improvement in yields was brought about by pyrite addition. Yields without added pyrite were similar to results with Indiana V coal, except for a somewhat higher yield of non-volatile products, SRC and IOM, with the Loveridge coal.

D. Evaluation of Coal Feedstocks

One run was conducted in the SRC II mode to evaluate Powhatan No. 1 coal as a feedstock for major liquefaction facilities. These results are also reported this quarter.

At 457°C, 1800 psig and 1.0 hour residence time in the SRC II mode, Powhatan No. 1 coal yielded 14.3 wt % hydrocarbon gas (based on MF coal), 41.4 wt % total oil, 24.8 wt % SRC and 5.7 wt % IOM. Hydrogen consumption was 4.4 wt %. These results were very similar to those obtained earlier with Powhatan No. 5 coal. Ammonium chloride deposits formed in the product separation system during this run and a rupture disc failed due to corrosion.

II. INTRODUCTION

A. Reports of Previous Work

Previous work at this laboratory has been described in interim, final, monthly, quarterly and annual reports to the Office of Coal Research, the Energy Research and Development Administration, and the Department of Energy. A summary of these documents appears in the second quarter 1979 report* along with a description of the current contract objectives and a history of the Merriam Laboratory in solvent refining of coal.

B. Currently Reported Work

During the fourth quarter of 1980, activities at the Merriam Laboratory were in three main areas: short residence time studies, addition of catalytic materials and evaluation of coal feedstocks.

1. Short Residence Time Studies

Four preliminary runs with Kentucky 9/14 coal and 16 runs with Indiana V coal were conducted with the primary objective of extending earlier short residence studies to higher pressures. The effects of temperature, pressure and dissolver residence time were investigated in a 2^3 plus midpoint factorial. The results of solvent recycle and varying preheater residence time were also explored. Coal from the Loveridge Mine was processed with and without added pyrite for a preliminary investigation of disposable catalysts in the short residence time SRC I mode.

2. Addition of Catalytic Materials

Five runs were conducted in the SRC II mode to complete a study on reaction paths and catalysts. One run was conducted with coal from the Kaiparowits Plateau in Utah and two runs with Pittsburgh seam coal from the Blacksville No. 2 Mine to compare the response of different types of coal to disposable catalyst (pyrite) addition. Discussion of those runs will be deferred until a later report. Two runs were also made with Pittsburgh seam coal from the Loveridge Mine to evaluate addition of molybdenum-alumina hydrodesulfurization catalysts (NiMo and CoMo on alumina) in the SRC II process.

3. Evaluation of Coal Feedstocks

Coal from the Powhatan No. 1 Mine was processed in the SRC II mode at 457°C, 1800 psig and 1.0 hour residence time to evaluate it as a potential feedstock for major liquefaction facilities.

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* "Exploratory Research on Solvent Refined Coal Liquefaction," Technical Progress Report for the Period April 1, 1979 through June 30, 1979; July 1980, FE/14800-10.

III. SHORT RESIDENCE TIME COAL LIQUEFACTION

A. Experimental

1. Run Conditions

The run conditions for all 22 short residence time runs reported this quarter are summarized in Table I.

a. Kentucky 9/14 Coal Runs (DOE 381-384)

A preliminary series of four runs was conducted with Kentucky 9/14 coal to determine if the ranges of variables proposed for the Indiana V coal factorial were appropriate. Of particular interest was the temperature that would give the maximum oil yields at 3000 psig and 8 minutes residence time. The Indiana V coal was in short supply and the Kentucky 9/14 coal was expected to give similar results.

Operability was checked at the most and least severe conditions contemplated for the factorial in runs DOE 381 and 383, respectively. The tendency to coke was tested by running at the highest temperature, lowest pressure and longest residence time anticipated, in run DOE 382.

When the total oil yield and recycle solvent yield (in preliminary calculations) in run DOE 381 at 487°C were found to be below that in earlier work at 475°C and 2000 psig, a nominal temperature of 475°C was investigated in run DOE 384. Since the oil yield was higher at 475°C than at 487°C, it was assumed the optimum temperature was between these two values. The factorial with Indiana coal was therefore set up with temperatures of 460°C and 480°C instead of 463°C and 487°C, as originally planned.

b. Indiana V Coal Runs (DOE 385-400)

(i) Factorial (Runs DOE 385-392)

These runs constitute a 2^3 factorial in temperature, pressure and residence time:

Factors	Levels -	 +
A. Temperature, °C	460	480
B. Pressure, psig	2000	3000
C. Residence Time, min.	4	8

The runs were made in a random sequence by use of a random number table. All runs had the same feed slurry formulation (30% coal, 70% recycle solvent) and the solvent was used single pass. The hydrogen feed rate was 2 wt %, based on slurry, for all runs. Each experiment was run until approximately 15 products were collected (~169 reactor volumes).

(ii) Midrange Runs (DOE 393, 394, 395 and 399)

The conditions for these runs were based on the 2^3 factorial above. Run DOE 393 was conducted at the midrange temperature, 470°C, at 3000 psig and 8 minutes residence time. Run DOE 394 was conducted at the midrange pressure, 2500 psig, at 480°C and 8 minutes residence time. Run DOE 395 was conducted at the midrange residence time, 6 minutes, at 480°C and 3000 psig. Finally, run DOE 399 was conducted at the midpoint, 470°C, 2500 psig and 6 minutes residence time.

(iii) Solvent Recycle (Run DOE 396)

Run DOE 396 was made with solvent recycle at 480°C, 3000 psig and 8 minutes residence time, whereas all the other runs in this series (DOE 385-400) were made with single pass solvent.

(iv) Replicate Runs (DOE 397 and 398)

Run DOE 397 was a repeat of run DOE 389 at the least severe conditions in the factorial and run DOE 398 was a repeat of run DOE 386 at the most severe conditions.

(v) Reduced Preheater Residence Time (Run DOE 400)

Run DOE 400 was made at the same conditions as runs DOE 386 and 398 except the preheater volume was 156 cm^3 instead of 240 cm^3. The purpose of this experiment was to determine if the residence time in the preheater (with a constant outlet temperature) affected the results.

c. Loveridge Coal Runs (DOE 407 and 408)

Both runs were made at a set of conditions used in the factorial study with Indiana V coal: 460°C (flat profile), 2000 psig and 8 minutes nominal residence time with 30 wt % Loveridge coal in the slurry and 2 wt % hydrogen, based on slurry. The lower temperature and pressure from the factorial were selected to maximize any effect of the catalyst.

Run DOE 407 was made with 5 wt % FeS_2 (as "Pyrite III") added to the coal and run DOE 408 was conducted without additive.

2. Dissolvers

Runs DOE 381-400 were conducted with a 9/16" ID dissolver and runs DOE 407 and 408 with an 11/16" ID dissolver. In both cases, a 1/4" thermowell was located in the center of the dissolver.

3. Coals

a. Kentucky 9/14 (Runs DOE 381-384)

The coal used in the preliminary experiments was from The Pittsburg & Midway Coal Mining Co.'s Colonial Mine in Hopkins County. This lot, LS8652 received from Harmarville, is similar to lot 7 from the Fort Lewis Pilot Plant used in earlier work. The moisture, ash, iron and sulfur analyses are shown in Table III-b.

b. Indiana V (Runs DOE 385-400)

The Indiana V coal was from the Old Ben Coal Company's Old Ben No. 1 Mine in Gibson County, sample 39393 from the Wilsonville Pilot Plant. The Commercial Testing and Engineering Co.'s analyses are shown in Table III-a.

c. Loveridge (Runs DOE 407 and 408)

The coal used in runs DOE 407 and 408 was from the Consol Loveridge Mine in West Virginia (Pittsburgh seam). Commercial Testing and Engineering Company analyses appear in Table III-a.

4. Solvent

The recycle solvent used single pass in runs DOE 381-395 and 397-400 was from a 5-drum shipment from the Fort Lewis Pilot Plant. The material, as received, contained 8.20% SRC, 0.31% IOM and 0.50% ash and was therefore subjected to a standard laboratory vacuum distillation with an endpoint of 270°C at 2 mm Hg. Analyses of the five drums after distillation, shown in Table IV, indicated that no further blending was necessary. In run DOE 396, the solvent was recycled. In runs DOE 407 and 408, solvent reclaimed from the series above, used once single pass, was employed.

5. Additive

The pyrite used in run DOE 407 ("Pyrite III") was obtained from the coal cleaning operation of the U. S. Steel Robena Mine, Green County, Pennsylvania. It was finely ground by the Jet Pulverizer Company, Palmyra, New Jersey. Analysis of the material, as shown in Table V, indicates 83 wt % purity as FeS_2. A particle size distribution (Coulter Counter method) was shown in the second quarter 1980 report*. The average particle size by "Fisher Subsieve Sizer" is 0.95 µm and the surface area is 2.61 m^2/g.

6. Operation

The unit was operated with little difficulty during these runs, filterable products were obtained and there was no appreciable buildup of coke in the dissolver or preheater. Temperature control was more difficult than usual in the preheater and bottom zone of the dissolver, due to the high slurry flow rates necessary to achieve the short residence times. The bottom zone of the dissolver ran cooler than desired, so the resulting average temperatures were usually somewhat lower than the nominal value for the run.

B. Results

The yields and recycle solvent and SRC analyses for all runs reported this quarter are shown in Table II. The unfiltered coal solution viscosity data are shown in Table VI and the solubilities of the SRC products in hexane and toluene are listed in Table VIII.

1. Yields and Hydrogen Consumptions

a. Preliminary runs with Kentucky 9/14 coal (DOE 381-384)

Increasing the pressure from 2000 to 3000 psig at 487°C and 8 minutes residence time had little effect on yields (run DOE 381 vs DOE 382), although the comparison is based on a limited amount of data. The effect of pressure was studied more thoroughly with Indiana V coal, as discussed below.

A slight improvement in recycle solvent yield (2.6% loss vs 6.7% loss) was brought about by decreasing the temperature from 487°C to 475°C at 3000 psig and 8 minutes residence time although there was little effect on total oil yield.

b. 2^3 Factorial in Temperature, Pressure and Residence Time, Indiana V Coal

The effects of all three variables (results of the 2^3 factorial) are summarized in Table X.

Increasing temperature from 460 to 480°C (variable A) increased hydrocarbon gas yield an average of 2.2 wt % (based on MF coal), total oil yield by 1.3 wt % and hydrogen consumption by 0.4 wt %. SRC yield was decreased by 3.2 wt %. There were also small decreases in

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* "Exploratory Research on Solvent Refined Coal Liquefaction," Technical Progress Report for the Period April 1, 1980 through June 30, 1980; FE/14800-21.

recycle solvent and IOM yields although the differences were close to or within experimental error.

Increasing pressure from 2000 to 3000 psig (variable B) increased recycle solvent yield an average of 6.7 wt %, total oil yield by 4.3 wt % and hydrogen consumption by 0.6 wt %. There were small decreases in hydrocarbon gas, SRC and IOM yields.

Increasing residence time from 4 to 8 minutes (variable C) apparently increased hydrocarbon gas yield and hydrogen consumption while oil and IOM yields were decreased. The effect of residence time is confounded, however, by the small dissolver diameter* and high gas flow rates used during this series of runs. The gas bubbles may have bridged the space between the thermowell and the wall of the reactor, creating a piston effect (slug flow). The true liquid residence time is therefore not known, but is likely to be less than the nominal value. This effect appears to be more pronounced for the nominal 4 minute residence time runs (see below). The total factorial is broken down into two 2^2 factorials in temperature and pressure in the next section because of the scatter in the data at 4 minutes residence time.

There was little 2 factor interaction between temperature and pressure (AB) in their effect on yields. There was also little 2 factor interaction between temperature and residence time (AC) except for the effect on total oil yield. The pressure/residence time interaction (BC) resulted in a generally larger pressure effect at longer residence times and vice versa. There was little 3 factor interaction between temperature, pressure and residence time (ABC) in their effect on yields.

c. 2^2 Factorials in Temperature and Pressure, Indiana V Coal

(i) Eight Minute Residence Time Runs

The effects of temperature and pressure at 8 minutes residence time are summarized in Table XI and illustrated in Figure 1. The beneficial effects of increasing the temperature and pressure are more evident than in the 2^3 factorial. There were average increases in total oil yields of 3.4 and 5.8 wt % and corresponding decreases in SRC yields of 4.2 and 4.0 wt % with increases in temperature or pressure, respectively. The two factor interaction resulted in less change in total oil, SRC and IOM yields with increased temperature at the higher pressure, and vice versa.

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* The dissolver had an ID of 9/16" and a 1/4" thermowell.

(ii) Four Minute Residence Time Runs

The effects of temperature and pressure at 4 minutes residence time are summarized in Table XII and illustrated in Figure 2. The results are not as straight forward as with the 8 minute residence time runs, possibly due to reactor hydrodynamics, as mentioned above. Increasing temperature (variable A) increased hydrocarbon gas yield and hydrogen consumption while recycle solvent and SRC yields were decreased. Increasing pressure (variable B), on the other hand, decreased hydrocarbon gas and IOM yields while recycle solvent, total oil and SRC yields and hydrogen consumption were increased. The two factor interaction was rather large, and for all yields except SRC, the effect of pressure was greater at the higher temperature and the effect of temperature was greater at the lower pressure.

d. Intermediate Values of the Variables, Indiana V Coal

The yields at 8 minutes residence time and either 2500 psig and 480°C (Run DOE 394), or 470°C and 3000 psig (Run DOE 393), are also shown in Figure 1.

The results (except for IOM yield) at 2500 psig and 480°C are better than would be expected, based on results at 2000 and 3000 psig and 480°C. The reason for the apparent optimum at 2500 psig is not known, but the results are within normal experimental error of those from the runs at 480°C, 3000 psig and 8 minutes residence time.

The results at 470°C and 3000 psig are nearly identical to those at 480°C and 3000 psig.

The yields (except for recycle solvent and hydrocarbon gas) and hydrogen consumption at 6 minutes residence time, 480°C and 3000 psig (run DOE 395) were nearly identical to the average results at 8 minutes, 480°C and 3000 psig:

> 13.3-13.7 wt % total oil, 62.4 wt % SRC, 4.6-4.8 wt % IOM and 2.5-2.6 wt % hydrogen consumption

Hydrocarbon gas production increased from 2.5 to 3.5 to 5.1 wt % as residence time was increased from 4 to 6 to 8 minutes, however, and there was a shift to lighter oil production (naphtha and wash solvent) from recycle solvent as residence time was increased from 6 to 8 minutes at 480°C and 3000 psig.

The results of the midpoint run (DOE 399) at 470°C, 2500 psig and 6 minutes residence time were as expected, with yields (except for recycle solvent) and hydrogen consumption at about the middle of the ranges observed for the runs in the factorial.

e. Solvent Recycle, Indiana V Coal

The yields and hydrogen consumption for the run with recycled solvent (DOE 396) are compared to the average of the two runs at the same conditions without solvent recycle:

Conditions*	Average of DOE 386 and 398	DOE 396
Solvent	Single Pass	Recycled
Yields, wt % MF Coal		
C_1-C_4	5.1	4.1
Recycle Solvent (loss)	(1.3)	0.7
Total Oil	13.3	15.7
SRC	62.4	59.9
IOM	4.8	4.7
Hydrogen Consumption, wt % MF Coal	2.5	2.7

* All at 480°C, 3000 psig and 8 minutes residence time

The recycle solvent yield was higher by 2.0 wt %, the total oil yield higher by 2.4 wt % and the SRC yield lower by 2.5 wt % when the solvent was recycled. There was a positive balance on recycle solvent when it was recycled, even with the apparently poor dissolver hydrodynamics.

f. Replicate Runs, Indiana V Coal

The two sets of replicate experiments are compared below:

Conditions	DOE 389	DOE 397	DOE 386	DOE 398
Temperature, °C	← 460 →		← 480 →	
Pressure, psig	← 2000 →		← 3000 →	
Residence Time, min.	← 4 →		← 8 →	
Yields, wt % MF Coal				
C_1-C_4	1.4	1.3	5.5	4.7
Recycle Solvent (loss)	(5.5)	(2.6)	(2.1)	(0.4)
Total Oil	10.6	11.8	13.2	13.4
SRC	66.6	65.8	63.1	61.7
IOM	6.2	5.9	4.7	4.9
Hydrogen Consumption, wt % MF Coal	1.0	1.1	2.9	2.1

The agreement between results for the replicate runs was relatively good, except for recycle solvent yield, considering the high flow rates, small diameter dissolver and short duration of the runs. The average of the differences was 0.5 wt % for hydrocarbon gas, 2.3 wt % for recycle solvent, 0.7 wt % for total oil, 1.1 wt % for SRC, 0.3 wt % for IOM and 0.5 wt % for hydrogen consumption.

g. Preheater Residence Time, Indiana V Coal

The yields and hydrogen consumptions with two different size preheaters are compared below:

Conditions*	Average DOE 386/398	DOE 400
Preheater Nominal Residence Time, min.	8.3	5.3
Yields, wt % MF Coal		
C_1-C_4	5.1	4.7
Recycle Solvent	(1.3)	4.0
Total Oil	13.3	14.9
SRC	62.4	61.5
IOM	4.8	4.7
Hydrogen Consumption, wt % MF Coal	2.5	1.9

* All at 480°C, 3000 psig and 8 min. dissolver residence time.

The differences are close to or within experimental error, except for the higher recycle solvent yield with the smaller preheater. The recycle solvent yield with the smaller preheater was the largest obtained in all 16 current short residence time runs with Indiana V coal.

h. Catalyst Addition with Loveridge Coal (Runs DOE 407 and 408)

The yields and hydrogen consumptions for the Loveridge coal runs with and without pyrite addition and an earlier run with Indiana V coal at the same conditions are compared below:

Conditions*	DOE 392	DOE 407	DOE 408
Coal	Indiana V	← Loveridge →	
Pyrite Addition level, wt % MF coal	--	5.1	--
Yields, wt % MF coal			
C_1-C_4	2.8	2.9	2.9
Recycle Solvent (loss)	(9.7)	(3.8)	(8.2)
Total Oil	4.1	7.2	4.3
SRC	71.2	72.7	74.5
IOM	5.1	7.9	8.0
Hydrogen Consumption wt % MF coal	1.8	1.6	1.4

* All at 460°C, 2000 psig, 8-9 minutes residence time, 30% coal in the slurry, 2% H_2 on slurry.

(i) Effect of Added Pyrite

There was a small improvement in yields brought about by pyrite addition. Recycle solvent loss was decreased by 4.4 wt %, from 8.2 to 3.8 wt % (based on MF coal), total oil yield was increased by 2.9 wt %, from 4.3 to 7.2 wt %, and SRC yield was decreased by 1.8 wt %, from 74.5 to 72.7 wt %. Hydrocarbon gas and IOM yields and hydrogen consumption were essentially unchanged.

(ii) Comparison to Indiana V Coal*

The Loveridge coal without added pyrite (run DOE 408) and the Indiana V coal (run DOE 392) produced essentially the same hydrocarbon gas, recycle solvent and total oil yields and about the same hydrogen consumption under these conditions. The yield of non-volatile products, SRC and IOM, was higher with the Loveridge coal. The yields with Indiana V coal might have been better if run DOE 392 had been made with an 11/16" ID dissolver (as run DOE 408 was) instead of a 9/16" dissolver. This is due to the possibility of bridging by the gas phase as discussed above.

2. Product Quality

a. Preliminary Runs with Kentucky 9/14 Coal (DOE 381-384)

Due to the limited number of samples and scatter in the data, no significant variation in the hydrogen analyses** for either recycle solvent or SRC are evident for the Kentucky 9/14 coal runs. The sulfur levels were all about the same except for run DOE 383 at the least severe conditions (463°C, 2000 psig and 4 minutes residence time), where the sulfur level was significantly higher in both the recycle solvent and SRC (0.52 vs 0.45-0.46% and 1.29 vs 0.86-0.88%, respectively). The SRC product from this run also had a significantly higher fusion point (181°C vs 105-132°C).

The solubilities of the SRC products from all four runs were about the same in hexane (18.5-25.4%). The solubilities in toluene were also about the same except for a low value for the SRC product from run DOE 381 (43.8% vs 64-73%). The reason for this low solubility is not known.

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* The yields are compared on an MF basis since ash varied only from 9.1% with Indiana V to 7.7% with Loveridge coal.

** The scatter in the hydrogen analyses was due to a faulty absorption reagent in the carbon train apparatus.

b. Effect of Temperature, Pressure and Residence Time, Indiana V Coal

The run conditions had little effect on recycle solvent analyses. In runs DOE 385-392, (the 2^3 factorial) recycle solvent hydrogen varied only from 7.58 to 7.86 wt %, sulfur from 0.40 to 0.46 wt % and nitrogen from 0.77 to 0.82 wt %.

There was considerably more variation in SRC analyses, as shown by the results of the 2^3 factorial in Table X. Although the nitrogen level was essentially the same in all SRC products (2.03-2.21 wt %), hydrogen level in the SRC was reduced an average of 0.20% and sulfur level was reduced 0.13% by increasing the temperature from 460 to 480°C. Increasing the pressure from 2000 to 3000 psig had little effect on SRC analyses. Increasing nominal residence time from 4 to 8 minutes had little effect on hydrogen level, but decreased sulfur level an average of 0.17 wt %, decreased fusion point by 23°C and increased the solubility in hexane by 7.5 wt %.

There was little two factor interaction between temperature and pressure (AB) except for the effect on SRC hydrogen level. Hydrogen level was reduced much more by increasing the temperature at the lower pressure than at the higher pressure. The temperature/residence time (AC) interaction resulted in a larger temperature effect at the longer residence time. The interaction between temperature and residence time (BC) had essentially no effect on the SRC analyses.

The three factor interaction between temperature, pressure and residence time (ABC) led to a relatively large effect on fusion point which probably indicates some inconsistencies in the data.

The solubility in toluene followed the same trends as that in hexane, although the relative changes were smaller. The solubility in toluene ranged from 60 to 72 wt % for this series of runs with an average of 67 wt % or 33% preasphaltenes (toluene insoluble).

c. Effect of Solvent Recycle, Indiana V Coal

The recycle solvent and SRC analyses for the solvent recycle run (DOE 396) and average analyses for the replicate runs at the same conditions with single pass solvent (DOE 386 and 398) are compared below:

Conditions*	Average DOE 386/398	DOE 396
Solvent	Single Pass	Recycled
Recycle Solvent		
% H	7.57	7.68
% S	0.38	0.36
% N	0.80	0.83
SRC		
% H	5.59	5.66
% S	0.54	0.47
% N	2.19	2.19
Fusion Point, °C	155	169
Solubility in Hexane, wt %	18.3	13.1

* All at 480°C, 3000 psig and 8 minutes residence time

The hydrogen levels in the recycle solvent and SRC were slightly higher in the solvent recycle run than in the single pass solvent experiments although the differences were close to experimental error. The hydrogen level in the equilibrated recycle solvent from the solvent recycle run was essentially the same as in the solvent from the Fort Lewis Pilot Plant used in startup.

The difference in other analyses are close to or within experimental error.

d. Replicate Runs, Indiana V Coal

The product analyses from the two sets of replicate runs are compared below:

Conditions	DOE 389	DOE 397	DOE 386	DOE 398
Temperature, °C	← 460 →		← 480 →	
Pressure, psig	← 2000 →		← 3000 →	
Residence Time, min.	← 4 →		← 8 →	
Recycle Solvent Analyses				
% H	7.83	7.48	7.66	7.48
% S	0.45	0.42	0.41	0.35
% N	0.78	0.79	0.82	0.78
SRC Analyses				
% H	5.75	5.66	5.62	5.56
% S	0.84	0.68	0.53	0.54
% N	2.03	2.07	2.21	2.16
Fusion Point, °C	170	220	165	144
Hexane Solubility, wt %	13.6	3.9	16.1	20.4

The recycle solvent analyses were in good agreement except for hydrogen level. There was scatter in the hydrogen analyses for these runs which could account for the discrepancy.

The SRC analyses were also generally in good agreement except for fusion point, hexane solubility and sulfur level in the DOE 389/397 set. The SRC product from run DOE 397 appears to be unusual in many respects with the highest fusion point and one of the lowest solubilities in hexane (and toluene) of all SRC products from this series of experiments.

e. Preheater Residence Time, Indiana V Coal

The differences in recycle solvent and SRC analyses between run DOE 400 (156 cm^3 preheater) and the average for runs DOE 386 and 398 (240 cm^3 preheater) at the same conditions are close to or within experimental error.

f. Catalyst Addition with Loveridge Coal (Runs DOE 407 and 408)

The product analyses for runs DOE 407 and 408 with Loveridge coal and run DOE 392 with Indiana V coal are compared below:

	DOE 392	DOE 407	DOE 408
Conditions*			
Coal	Indiana V	← Loveridge →	
Pyrite Addition Level, wt % MF Coal	--	5.1	--
Recycle Solvent Analysis			
% H	7.78	7.56	7.66
% S	0.40	0.43	0.43
% N	0.80	0.93	0.79
SRC Analyses			
% H	5.96	5.69	5.75
% S	0.70	1.15	1.24
% N	2.12	1.87	1.82
Fusion Point, °C	167	132	141
Solubility, wt % in			
hexane	12.5	16.7	13.8
toluene	68.6	74.7	72.6

* All at 460°C, 2000 psig, 8-9 minutes residence time, 30% coal in the slurry, 2% H_2 on slurry.

(i) Effect of Added Pyrite

The recycle solvent and SRC elemental analyses were essentially the same for the Loveridge runs with and without added pyrite (DOE 407 and 408, respectively) except for recycle solvent nitrogen. This difference is based on a single analysis for each product and could be an analytical error. The fusion point was slightly lower and the solubilities slightly higher for the SRC from the catalyzed run (DOE 407) although the differences are close to experimental error.

(ii) Comparison to Indiana V Coal

The recycle solvent analyses were essentially the same for Loveridge and Indiana coals (runs DOE 408 and 392, respectively). The SRC from Loveridge coal was higher in sulfur (1.24 vs 0.70 wt %) and lower in nitrogen (1.82 vs 2.12 wt %) and hydrogen (5.75 vs 5.96 wt %) than that from Indiana coal. The difference in product sulfurs may be due to the lower organic sulfur in the Indiana feed coal (1.3 vs 1.8 wt %). The fusion point was a little higher (167 vs 141°C) for the SRC from the Indiana coal although the solubilities were a little lower. The differences are again close to experimental error.

3. Viscosity Measurements

a. Preliminary Runs with Kentucky 9/14 Coal (DOE 381-384)

The unfiltered coal solution (UFCS) viscosities for the four Kentucky 9/14 coal runs are compared in Figure 3.

The UFCS viscosities were all about the same except for that from run DOE 383 at the least severe conditions (463°C, 2000 psig and 4 minutes residence time), which was significantly higher. This is in conjunction with the higher sulfur level and fusion point for the SRC product from this run.

b. Effect of Temperature, Pressure and Residence Time, Indiana V Coal

The results of the 2^3 factorial are summarized in Table X. Increasing the temperature from 460 to 480°C, the pressure from 2000 to 3000 psig and the residence time from 4 to 8 minutes decreased the UFCS viscosity by an average of 4.1, 10.5 and 15.7 cP, respectively.

The temperature/pressure (AB) and temperature/residence time (AC) interactions had a significant effect because of the unusual result for the DOE 392/387 comparison (change in temperature at 2000 psig and 8 minutes residence time). The viscosity increased with temperature for this comparison whereas it decreased for the other three temperature change comparisons. The three factor, temperature/pressure/residence time, interaction also was significant in the effect of the variable conditions on viscosity. This was also probably due to the unusual response to temperature in the DOE 392/387 comparison.

The effects of temperature and pressure at 4 and 8 minutes residence time are shown graphically in Figures 4 and 5, respectively.

c. Effect of Solvent Recycle, Indiana V Coal

There was essentially no difference in UFCS viscosity between the solvent recycle run (DOE 396) and runs DOE 386 and 398 at the same conditions without solvent recycle, as shown in Figure 6.

d. Replicate Runs, Indiana V Coal

The UFCS viscosities for the replicate experiments (runs DOE 386/398 and 389/397) were close together for each set. The results for the first set are also shown in Figure 6.

e. Preheater Residence Time, Indiana V Coal

The residence time in the preheater (5 or 8 minutes) did not have a significant effect on UFCS viscosity (compare runs DOE 400 at 5 minutes preheater residence time and either run DOE 386 or 398 at 8 minutes preheater residence time in Figure 6).

f. Catalyst Addition with Loveridge Coal
(Runs DOE 407 and 408)

The unfiltered coal solution (UFCS) viscosities for runs DOE 407 and 408 are compared in Figure 7.

The UFCS viscosities in runs DOE 407 and 408, with and without added pyrite, respectively, were essentially the same. The UFCS viscosity for the Indiana V run was about 50% higher than that for the Loveridge run without added pyrite. The levels of solids (4.9-6.3 wt %) and SRC (22.1-25.1 wt %) were about the same in the UFCS from these two runs, so the SRC apparently had a lower molecular weight with the Loveridge coal.

IV. ADDITION OF CATALYTIC MATERIALS

A. Experimental

1. Run Conditions

All of the runs in this series (DOE 404R-405R) were conducted at the standard SRC II conditions established during our recent study on the relationship between coal properties and liquefaction behavior:

Average Dissolver Temperature:	450°C (flat profile)
Pressure:	2250 psig
Residence Time	1.0 hour
Coal Concentration in Slurry	30 wt %
Hydrogen Feed Rate:	4 wt % based on slurry

Run DOE 404R was made with Loveridge coal and NiMo on alumina catalyst added at a rate of 1.0, 0.5 and 0.1 wt %, based on slurry (0.34, 0.17 and 0.04 wt % Mo based on coal) in parts A, B and C of the experiment, respectively.

Run DOE 405R was made with Loveridge coal and CoMo on alumina catalyst added at a rate of 0.5 and 0.1 wt %, based on slurry (0.18 and 0.04 wt % Mo based on coal) in parts A and B of the experiment, respectively.

2. Coal

The coal used in runs DOE 404R and 405R was from the Loveridge Mine in West Virginia (Pittsburgh seam). Commercial Testing and Engineering Company analyses appear in Table III-a.

3. Additives

a. NiMo on Alumina

The catalyst used in run DOE 404R was supplied by the American Cyanamid Company and is designated HDS-1443. It was supplied as nominal 1/32" cylindrical extrudates and was ground by hand to pass 150 mesh before use. Measurements conducted before and after grinding show no significant change in surface area. The catalyst, which is of the NiMo type, was developed for expanded bed, heavy oil hydroprocessing. Analyses of the material are shown in Table V.

b. CoMo on Alumina

This catalyst, used in run DOE 405R, was also supplied by Cyanamid and designated HDS-1442. It was supplied as nominal 1/16" cylindrical extrudates and was ground by hand to pass 150 mesh before use, also without loss of surface area. This CoMo type catalyst was developed for the H-coal process. Analyses are shown in Table V.

4. Operability

Deposition of ammonium chloride in the product separation system apparently occurred throughout these runs and caused the shutdown of run DOE 404RC. This is the first time that ammonium chloride deposition has been observed with Loveridge coal at the Merriam Laboratory. Ammonium chloride formation in the current runs may be due to the increased product denitrogenation brought about by the molybdenum-alumina catalysts and resulting higher concentration of ammonia in the product streams, particularly in runs DOE 404RA and 404RB.

Operability was noticeably worse in runs DOE 404RC and 405RB where only 0.1 wt % catalyst, based on slurry, was used than in runs DOE 404RA, 404RB and 405RA where 0.5 or 1.0 wt % catalyst was employed.

5. Elemental Balances

The elemental balances for runs DOE 404R through 405RB are shown in Tables XIII through XVII. These studies were carried out to elucidate the nature of the products and verify overall material balances. The yields in Tables XIII through XVII vary somewhat from those found elsewhere in the report because they were based on preliminary calculations. The results are summarized below:

Out/In	DOE 404RA	DOE 404RB	DOE 404RC	DOE 405RA	DOE 405RB
Carbon	1.00	1.01	0.99	0.99	1.00
Nitrogen	1.03	1.04	1.06	1.05	1.04
Sulfur	0.79	0.97	1.01	0.95	0.93

The balances for carbon were excellent. The low values obtained for sulfur in some cases may be due to loss of dissolved H_2S.

6. Unfiltered Coal Solution Distillations

The pressure during vacuum distillation of unfiltered coal solution generally increased near the endpoint (270°C) when added catalysts were employed. This was most pronounced for runs DOE 404RA and 404RB and indicates that the added catalyst contributed to decomposition during distillation.

B. Results

1. Yields and Hydrogen Consumptions

The yields and hydrogen consumptions for these runs are shown in Table II.

The results of processing Loveridge coal in the SRC II mode with various types of added catalysts are shown below:

	DOE 371R***	DOE 378R***	DOE 404RA	DOE 404RB	DOE 404RC	DOE 405RA	DOE 405RB
Conditions*							
Additive	FeS_2	Mo	← NiMo → on Alumina			← CoMo → on Alumina	
Addition Level, wt % MF Coal**	4.92	0.36	0.34	0.17	0.04	0.18	0.04
Yields, wt % MF Coal							
C_1-C_4	13.4	12.4	12.3	11.5	12.0	11.7	11.5
Total Oil	39.5	40.6	44.2	42.7	39.3	43.9	37.6
SRC	31.7	31.8	28.1	29.0	32.5	28.2	35.4
IOM	5.5	5.0	5.1	5.4	5.2	5.2	4.8
Hydrogen Consumption, wt % MF Coal	4.7	4.9	5.3	5.0	4.8	5.4	5.4

* All at 450°C, 2250 psig and 1.0 hour residence time with 30% Loveridge coal in the slurry.

** As pure FeS_2 or Mo.

*** From "Exploratory Research on Solvent Refined Coal Liquefaction," Technical Progress Report for the Period July 1, 1980 through September 30, 1980; February 1981, FE/14800-25.

The molybdenum catalyst dispersed on alumina and promoted by Ni (run DOE 404RA) gave a 3.6 wt % higher oil yield and 3.7 wt % lower SRC yield than the same amount of molybdenum added by aqueous impregnation of the coal (run DOE 378R). IOM yields and hydrogen consumptions were about the same. As noted in a previous report[†], the run catalyzed with 0.36 wt % molybdenum based on coal (DOE 378R) in general produced slightly better yields than the run with 4.9 wt % added pyrite (DOE 371R), although the differences were close to experimental error.

At an addition level of 0.17-0.18 wt % Mo, based on coal, the molybdenum-alumina catalyst promoted by cobalt (run DOE 405RA) gave slightly better yields than molybdenum-alumina promoted by nickel (DOE 404RB), although at the 0.04 wt % addition level the opposite was generally true. In both comparisons, the results are within experimental error, so it is concluded that there is not a significant difference between the NiMo and CoMo catalysts at these conditions.

The yields generally improved as the level of molybdenum-alumina catalyst was increased. In run DOE 404R, the oil yield increased in direct proportion to the added catalyst level, although the SRC yield was essentially the same at the two highest additive levels.

2. Product Quality

The naphtha (<193°C), middle distillate (193-288°C), heavy distillate[††] (>288°C) and distillation residue[††] analyses

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[†] "Exploratory Research on Solvent Refined Coal Liquefaction," Technical Progress Report for the Period July 1, 1980 through September 30, 1980; February 1981, FE/14800-25.

[††] The endpoint for the distillation is 270°C at 2 mm Hg which results in a final atmospheric boiling point (by simulated distillation) of about 270°C (878°F).

for runs DOE 404R-405R are shown in Table II. This represents a new analysis scheme at Merriam for SRC II runs since previously only the heavy distillates and distillation residues were submitted routinely for elemental analysis[†]. The solubilities of the distillation residues in hexane, toluene and pyridine are shown in Table IX and the unfiltered coal solution and feed slurry viscosities are listed in Tables VI and VII, respectively.

The analyses of heavy distillates and distillation residues from the SRC II processing of Loveridge coal with various types of added catalysts are shown below:

	DOE 371R***	DOE 378R***	DOE 404RA	DOE 404RB	DOE 404RC	DOE 405RA	DOE 405RB
Conditions*							
Additive	FeS_2	Mo	← NiMo on alumina →			← CoMo on alumina →	
Addition Level, wt % MF Coal**	4.92	0.36	0.34	0.17	0.04	0.18	0.04
Heavy Distillate Analyses							
% S	0.41	0.22	0.00	0.07	0.13	0.13	0.25
% N	1.16	1.22	0.97	1.03	1.08	1.09	1.20
Distillation Residue Analyses							
% S	4.54	2.19	1.88	1.87	1.69	2.14	2.01
% N	1.38	1.42	1.16	1.26	1.42	1.28	1.47
Fusion Point °C	132	104	113	97	106	111	108
Solubility Ratio							
Hexane/Pyridine	0.55	0.54	0.51	0.51	0.49	0.57	0.49
Toluene/Pyridine	0.93	0.95	0.92	0.94	0.93	0.94	0.91

* All at 450°C, 2250 psig and 1.0 hour residence time with 30% Loveridge coal in the slurry and 4 wt % hydrogen, based on slurry.

** As pure FeS_2 or Mo.

*** From the "Exploratory Research on Solvent Refined Coal Liquefaction," Technical Progress Report for the Period July 1, 1980 through September 30, 1980; February 1981, FE/14800-25.

The unfiltered coal solution viscosities for these runs are compared in Figure 8 and the feed slurry viscosities in Figure 10.

The heavy distillate from the run with the molybdenum-alumina catalyst (DOE 404RA) had significantly lower sulfur (<0.005 versus 0.2-0.4 wt %) and nitrogen (0.97 vs 1.16-1.22 wt %) levels than the runs catalyzed with the same amount of molybdenum by aqueous impregnation (DOE 378R) or by pyrite (DOE 371R). These are the lowest heavy distillate[††] sulfur and nitrogen levels ever achieved at Merriam with a Pittsburgh seam coal.

[†] Analyses of these four products for selected runs appears in the third quarter 1980 report. In addition, proportional blends of products from Belle Ayr, Kaiparowits, Wadge seam and Ayrshire coals were distilled into ten fractions and analyzed, as reported in the first and second quarter 1980 reports.

[††] For heavy distillate with an initial boiling point of 288°C.

There was also less nitrogen in the distillation residue (1.16 vs 1.38-1.42 wt %) with the molybdenum-alumina catalyst and less sulfur; 1.9 wt % in run DOE 404RA versus 2.2 wt % in DOE 378R and 4.5 wt % in run DOE 371R. (The value is higher in the latter run due to the added pyrite.) The fusion points and relative solubilities of the distillation residues were nearly the same with all three catalysts, indicating that the average molecular weight and functionality of the residues were not widely different.

The UFCS viscosities for runs catalyzed with 0.34-0.36 wt % molybdenum (based on coal) by aqueous impregnation (DOE 378R) or as molybdenum-alumina (DOE 404RA) were not significantly different. The UFCS from the run catalyzed by 4.9 wt % pyrite (DOE 371R) had a viscosity about half that of UFCS from the Mo catalyzed runs. The reason for this is not known. Due to differences in formulation, the feed slurry viscosity was highest with the molybdenum-alumina catalyst (DOE 404RA) and lowest with pyrite (DOE 371R), with the Mo by aqueous impregnation in between.

The heavy distillates and distillation residues from the run catalyzed by NiMo on alumina (DOE 404R) appeared to be of slightly higher quality (lower S and N) than those from the run catalyzed by CoMo on alumina (DOE 405R) at the same addition levels, although the differences were close to experimental error. The fusion points and relative solubilities were all essentially the same.

The molybdenum-alumina catalysts promoted by nickel or cobalt gave essentially the same UFCS viscosities (Figure 8) at an addition level of 0.17 wt % Mo, based on coal. There was an unusually high viscosity with the cobalt promoted catalyst at the 0.04 wt % addition level (DOE 405RB) for an unknown reason. The feed slurry viscosities (Figure 10) were lower for the nickel promoted catalyst than for the cobalt promoted catalyst at the same addition levels (DOE 404RB vs 405RA and DOE 404RC vs 405RB).

The heavy distillate sulfur and nitrogen levels increased as the level of added molybdenum-alumina catalyst decreased. The nitrogen level in the distillation residue also increased with decreased catalysis although the sulfur level decreased. This decrease in sulfur may be due to a combination of the variable MoS_2 level in the residue and the concentrating effect in the residue because of higher conversion with increased catalysis. The catalyst level had little effect on fusion points and relative solubilities.

The catalyst level had no apparent effect on UFCS viscosity in run DOE 404R with the NiMo on alumina. With the CoMo on alumina catalyst there was an order of magnitude increase in UFCS viscosity at the 0.04% Mo on coal addition level compared to the 0.18% addition level. The viscosity at the lower addition level (DOE 405RB) appears to be out of line, however. The feed slurry viscosities do not appear to correlate with catalyst level. They, in fact, decreased as catalyst level was decreased even though operability generally got worse.

V. EVALUATION OF COAL FEEDSTOCKS

A. Experimental

1. Run Conditions

The run conditions for this experiment are shown in Table I.

Run DOE 406R was conducted at 457°C average dissolver temperature (nominal 450°C inlet and 460°C outlet), 1800 psig and 1.0 hour residence time with 30 wt % Powhatan No. 1 coal in the slurry and 4 wt % hydrogen, based on slurry. The target levels for total solids and middle distillate in the feed slurry were 45 and 6 to 10 wt %, respectively.

During part A of the experiment, the temperature was 59°C in the slurry mix pot and 66°C in the slurry feed pot. In part B of the run, the mix pot temperature was increased to 67°C and the feed pot temperature to 97°C to determine the effect of these temperatures on loss of volatile materials from the feed slurry.

2. Coal

The coal used in run DOE 406R was from the North American Coal Company's Powhatan No. 1 Mine in Ohio (Pittsburgh seam). The elemental analyses are shown in Table III-a, the petrographic analysis in Table III-c and the float and sink analysis in Table III-d.

3. Operation

About seven days after beginning the run, plugging was observed around the intermediate temperature, high pressure (ITHP) separator. A plug at the bottom of the separator prevented most of the liquid phase from being fed to the distillation column. A restriction in the overhead line from the separator caused a pressure differential between the dissolver and gas letdown valve which fluctuated between 20 and 130 psi. Water was injected into the line between the high temperature, high pressure (HTHP) separator and ITHP separator for 37 minutes (59 grams fed) shortly after switching to part B of the run. The pressure differential disappeared about 15 minutes after water injection was begun and normal amounts of product came off the distillation column soon thereafter.

The run ended when the rupture disc on the transfer line between the dissolver and the HTHP separator burst. This disc was sent to GR&DC, Harmarville, where it was found that the failure apparently occurred due to corrosion. A copy of their report is included as Appendix B.

A deposit was found in the vapor line from the ITHP separator after the run which was confirmed to be ammonium chloride.

4. Material Balances

a. Ash Balance

There was no apparent effect on ash balance brought about by raising the feed tank temperature from 66 to 97°C (experimental ash output of 102.1 vs 102.8% of theoretical*). The ash balances were satisfactory for both parts of the run.

b. Elemental Balance

The elemental balance for run DOE 406RB is shown in Table XVIII and summarized below:

	OUT/IN
Carbon	1.01
Nitrogen	0.95
Sulfur	0.93

There was the usual excellent balance on carbon and apparent loss of dissolved H_2S and NH_3 from the oil products.

B. Results

The yields and product analyses for all runs reported this month are shown in Table II. The solubilities of the distillation residue in hexane, toluene and pyridine are shown in Table IX.

1. Yields and Hydrogen Consumptions

The SRC II yield structures for Powhatan No. 1 and Powhatan No. 5 coals are compared below:

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* It is possible that a small increase in volatile loss occurred, but any difference was obscured by other errors in the ash balance.

Conditions*	DOE 276R†	DOE 406RB
Coal	Powhatan No. 5	Powhatan No. 1
Yields, wt % MF Coal		
C_1-C_4	14.2	14.3
Total Oil	39.9	41.4
SRC	26.0	24.8
IOM	4.6	5.7
Ash	9.5	9.2
Hydrogen Consumption, wt % MF Coal	5.1	4.4

* Both at 457°C average dissolver temperature (450-451°C inlet, 458-460°C outlet), 1800 psig, 1.0 hour residence time, 30 wt % coal and 42.1-46.5 wt % solids in the slurry, 4% hydrogen or slurry.

Part B of run DOE 406R is used in the comparison because operation was more stable than in part A.

The total oil yield was slightly higher with Powhatan No. 1 coal and SRC yield slightly lower. The Powhatan No. 5 coal had a slightly lower IOM yield and a slightly larger hydrogen consumption, however. All these differences are close to or within experimental error. Hydrocarbon gas yield was essentially the same in both runs.

2. Product Quality

The product analyses for Powhatan No. 1 and Powhatan No. 5 coals are compared below:††

Conditions*	DOE 276R	DOE 406RB
Coal	Powhatan No. 5	Powhatan No. 1
Heavy Distillate Analyses		
% H	7.28	7.68
% S	0.40	0.50
% N	1.25	1.16
Distillation Residue Analyses		
% H	3.72	3.69
% S	2.93	3.83
% N	1.30	1.35
Fusion Point, °C	121	102

* Both at 457°C average dissolver temperature (450-451°C inlet, 458-460°C outlet), 1800 psig, 1.0 hour residence time, 30 wt % coal and 42.1-46.5 wt % solids in the slurry, 4% hydrogen on slurry.

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† From the "Exploratory Research on Solvent Refined Coal Liquefaction" Technical Progress Report for the Period January 1, 1978 through December 31, 1978; FE/496-170.

†† Distillation residue solubility data are not available for run DOE 276R.

The hydrogen level was higher in the heavy distillate from the Powhatan No. 1 coal, although it was about the same in both distillation residues. The sulfur level was higher in both products from run DOE 406RB, possibly due to the higher total sulfur level in the Powhatan No. 1 feed coal (4.7 vs 3.5 wt %)[†] The nitrogen levels were about the same in both products from both coals, but the residue fusion point was a little lower with Powhatan No. 1.

3. Viscosity Measurements

The unfiltered coal solution (UFCS) and feed slurry viscosities for run DOE 406R are listed in Tables VI and VII, respectively.

The UFCS viscosity from run DOE 406R is compared to that from an earlier run[††] with Powhatan No. 5 coal in Figure 9. The UFCS viscosities were fairly close together for runs DOE 344RD and 406RA with similar feed pot temperatures. The viscosity was about twice as high for run DOE 406RB with a feed pot temperature of 97°C, however. The reason for this large difference is not known. The UFCS from run DOE 406RB had only slightly higher levels of solids (25.4 vs 24.0 wt %) and SRC (42.2 vs 40.9 wt %) than run DOE 406RA.

The feed slurry viscosities for these runs are compared in Figure 11. The feed slurry viscosity from run DOE 406RA was about 50% higher than that from run DOE 344RD due to differences in slurry formulation (including 3.5 wt % higher solids, 2.4 wt % higher SRC). The viscosity was much higher in part B of run DOE 406R, with a feed pot temperature of 97°C, than in part A. This may be due to the more viscous UFCS used in the formulation or an acceleration in the increase in viscosity that normally occurs when feed slurry resides in the feed pot, due to the elevated temperature.

VI. SIMULATED DISTILLATION ANALYSIS OF FEED SLURRIES

A. Introduction

Information can be obtained on feed slurries if the soluble portion is analyzed by simulated distillation gas chromatography. Carbon disulfide is a useful solvent because it gives minimal response in the flame ionization detector.

The resulting solutions contain distillate oils plus soluble materials which boil above the normal range recovered in vacuum distillation (endpoint of 270°C at 2 mm Hg). Thus, when they are analyzed by simulated distillation, the spectrum has a tail which does not return to the baseline by the normal cut off of 450 to 470°C. See, for example, a spectrum of the feed slurry from run DOE 405RA, shown in Figure 12.

[†] An analysis of sulfur forms is not available for the Powhatan No. 5 coal.

[††] Viscosities were not measured at the time run DOE 276R, discussed earlier, was made. Run DOE 344RD was made at the same conditions as DOE 406R, but only one half dissolver was used.

B. Comparison of Feed Slurry and Product

When the simulated distillation of the feed slurry sample is compared to that of the proportional blend of product oils in Figure 12, the reduction in average molecular weight during liquefaction can be clearly seen. The proportional blend of oils in this case represents about 41 wt % of the feed coal. Hydrocarbon gases (11.5 wt % of the feed coal) are also produced which are not represented in the proportional blend. If an area corresponding to this yield is sketched on the proportional blend spectrum at the location that would be occupied by C_1-C_4 hydrocarbons, a visual impression of the product is presented which can be compared to that of the feed slurry.

Tie lines have also been drawn on the percent off versus temperature table to compare the volatile material in the feed to that in the products. This is done by locating similar temperatures in both. This is another way to indicate the substantial shift in boiling range between input and output streams.

The comparatively large yield of methane through butane becomes an important consideration in discussing the chemical reactions that take place during coal liquefaction. One of the frequent reactions will be removal of small fragments from larger molecules which are quenched to form C_1 to C_4 hydrocarbons. Moreover, the molar proportion of methane is large compared to ethane which is, in turn, large compared to propane. N-butane is about three times as abundant as i-butane. This would suggest that these fragments are produced by opening of aromatic rings and heteroatom removal reactions which also open rings.

C. Effect of Catalysis

Figure 13 shows similar results for run DOE 405RB which was made at the same conditions as 405RA except for 0.1 wt % added CoMo catalyst in the feed slurry instead of 0.5 wt %. Using less catalyst resulted in an increase in the average molecular weight of the feed slurry. The proportional blend of products for this run is also shown in Figure 13 with the relative amount of C_1 to C_4 gasses (11.5 wt % of the feed coal) sketched in. The oil yield for this run was slightly less than in the run shown in Figure 12, but a direct comparison between runs is difficult because different signal attenuations were used during the simulated distillation. The qualitative comparison between product and feed is the same, however.

D. Comparison to Powhatan No. 1 Product

The procedure was repeated during run DOE 406RB with the results shown in Figure 14. The proportional blends of product from runs DOE 405R and 406R seem to be composed of the same compounds (or groups of compounds with essentially the same boiling point), in slightly different proportions, even though the runs were made with different coals, catalysts and conditions.

VII. CONCENTRATION OF IRON COMPOUNDS IN UNFILTERED COAL SOLUTION BY GRAVITY SETTLING

A preliminary experiment was conducted to determine if the iron compounds in unfiltered coal solution (UFCS) could be concentrated by gravity settling. The iron compounds apparently settled less than other solids in the UFCS so that their concentration in the ash was increased in the overflow stream from the settler. The details of the study are given in Appendix B.

VII. MAINTENANCE AND MODIFICATIONS

A. Maintenance

1. Production Curtailment

Time Period	On Stream Factor, %
Jan-June 1980	86.3
July-Dec 1980*	80.5
Jan-Dec 1980*	83.4

*Excludes scheduled Christmas shutdown.

The maximum on-stream factor is about 88% since the unit is shut down voluntarily about twice a month.

2. Dissolver Air Furnace Overhaul

The maintenance required to keep the dissolver air furnace operating has increased in recent months. This maintenance involves primarily the fan bearings and motors, although wiring was replaced on several occasions. The furnace fans were designed to pull a slip stream of cool air through the hollow fan shafts to keep the fan bearings from overheating. This was not possible because the exit flues on the furnace had been blocked either at the time of manufacture or when the furnace was received. An even more serious problem was the fact that the electrical wiring was not designed to withstand the temperature in the wireways. In addition, several of the heating elements had deteriorated to the point of becoming a shock and spark hazard. The air furnace was therefore overhauled between runs DOE 397 and 398.

The heating elements for zones 3, 4 and 5 were replaced along with the insulation in the dissolver area. The wiring and plug-in sockets were replaced with components resistant to high temperature and the inside surface of the furnace was coated with a thin layer of fire clay to protect the insulation from abrasion.

3. Pressure Products Industries (PPI) Hydrogen Compressor

This equipment continued giving problems during the reporting period, requiring overhaul during or after runs DOE 387, 390, 393, 397 and 402. The '0" rings and diaphragms were generally replaced as part of this maintenance. During the last overhaul, it was found that the "0" ring on the oil side of the diaphragm had unraveled into sheets resembling pieces of black Teflon tape which had plugged up the check valves in the hydraulic oil lines.

4. Temperature Controllers

The number of relay failures in the Love temperature controllers have been dramatically reduced. This is due, in part, to an inspection program that was instituted to replace defective relays before they could malfunction. In addition, silicon controlled rectifier (SCR) controllers and a controller with a resistance capacitance (RC) circuit (to cut down on the inductive load) were tested in areas which have had high failure rates. The improvement in service brought about by these changes will be monitored over the next few months.

5. Christmas Shutdown (12-23-80 to 1-5-81)

The unit was given a thorough overhaul during this shutdown. This included inspection of the condensors on the distillation column and ITHP separator and the ITHP separator itself for evidence of corrosion. Only a minor amount of corrosion was found in the separator and none in the condensors.

B. Modification

A new analytical laboratory has been outfitted and is now in service. This larger, multipurpose facility will allow for more efficient laboratory operations.

TABLE I

Conditions Summary

	DOE 381	DOE 382	DOE 383	DOE 384	DOE 385	DOE 386	DOE 387	DOE 388	DOE 389	DOE 390	DOE 391	DOE 392
Coal	Kentucky 9/14[a]				Indiana V[b]							
Operating Mode	Short Residence Time, SRC I (Single Pass Solvent)											
Dissolver	"Short Residence Time"											
Slurry Formulation[c]												
% Coal	30											
% Recycle Solvent[d]	70											
Slurry Feed Rate												
g/hr	2047	2037	4140	2034	3996	1977	1979	4022	4130	4042	2035	2025
$lb/hr/ft^3$	545	542	1101	541	1063	526	526	1070	1099	1075	541	539
Dissolver Nominal Slurry Residence Time, min.[e]	7.9	8.0	3.9	8.0	4.1	8.2	8.2	4.0	3.9	4.0	8.0	8.0
Preheater Nominal Slurry Residence Time, min.[f]	8.1	8.1	4.0	8.1	4.1	8.4	8.4	4.1	4.0	4.1	8.1	8.2
Coal Feed Rate[c]												
g/hr	606	604	1226	603	1177	582	583	1184	1216	1190	599	596
$lb/hr/ft^3$	161	161	326	160	313	155	155	315	323	317	159	159
Hydrogen Feed Rate[g]												
Mole/hr	20.2	20.3	40.5	20.2	40.5	20.2	20.3	40.5	40.5	40.5	20.2	20.3
Wt % based on slurry	1.97	1.99	1.96	1.99	2.03	2.04	2.05	2.01	1.96	2.00	1.99	2.00
MSCF[h]/ton of coal	25.3	25.5	25.0	25.4	26.1	26.3	26.4	25.9	25.2	25.8	25.6	25.8
Reactor Pressure, psig	3000	2000	2000	3000	2000	3000	2000	3000	2000	3000	3000	2000
Nominal Dissolver Temperature, °C	487	487	463	475	480	480	480	460	460	480	460	460
Average Dissolver Temperature, °C	483	483	460	472	474	477	476	456	457	472	458	458
Dissolver Temperature, °C[i]												
Zone 6 (top)	487±1	487±1	464±1	475±1	479±1	480±2	480±1	460±1	460±1	480±1	459±1	460±0
5	487±1	487±1	463±1	475±1	480±1	480±0	479±1	461±1	460±0	480±1	460±1	460±1
4	487±1	486±1	464±1	475±1	480±1	480±1	479±2	460±0	460±0	480±1	460±1	460±0
3	487±1	486±2	464±1	475±1	481±1	480±1	479±2	460±1	461±1	480±2	461±1	460±1
2	487±1	485±2	463±1	475±1	476±1	480±2	479±3	460±0	461±1	473±3	460±1	459±1
1 (bottom)	465±2	466±4	444±1	459±1	449±1	463±3	462±3	437±1	440±1	440±1	449±2	449±1
Preheater Temperature, °C[i]												
Top	400±1	400±2	406±1	399±3	396±1	395±3	399±3	401±1	396±3	391±5	401±3	397±3
Middle	407±1	405±3	385±3	397±6	383±2	400±1	400±1	382±1	382±5	379±2	398±2	397±1
Bottom	368±4	364±5	333±3	353±5	346±3	384±4	386±4	338±1	338±5	336±5	371±2	370±1

a) P&M Colonial Mine, lot LS 8652, from Harmarville, see Table IIIb for analyses. b) Old Ben Mine No. 1, sample 39393, see Table IIIa for analyses. c) Dry coal basis. d) Lot 1927 recycle solvent from Fort Lewis, redistilled to remove SRC, see Table IV for analyses; used single pass. 3) Volume of dissolver = 235 cm^3 (0.00828 ft^3). f) Volume of preheater 240 cm^3 (0.00846 ft^3). g) Hydrogen feed rates do not include that added as DP cell purge. h) One thousand cubic feet at one atmosphere and 60°F. i) Average and standard deviation.

TABLE I (Continued)

Conditions	DOE 393	DOE 394	DOE 395	DOE 396	DOE 397	DOE 398	DOE 399	DOE 400
Coal	Indiana V^{a}							
Operating Mode	Short Residence Time SRC I							
Solvent Source	Single Pass			Recycled	Single Pass			
Dissolver	"Short Residence Time"							
Slurry Formulation, wt %^{b}								
Coal	30							
Recycle Solvent	70							
Slurry Feed Rate								
g/hr	2007	2033	2628	2006	4041	2052	2716	2044
lb/hr/ft^{3c}	534	541	699	534	1075	546	723	544
Dissolver Nominal Slurry Residence Time, min.^{c}	8.1	8.0	6.2	8.1	4.0	7.9	6.0	7.9
Preheater Nominal Slurry Residence Time, min.^{d}	8.3	8.1	6.3	8.3	4.1	8.1	6.1	5.3
Coal Feed Rate^{b}								
g/hr	590	597	772	589	1187	603	798	600
lb/hr/ft^{3}	157	159	205	157	316	160	212	160
Hydrogen Feed Rate^{e}								
Mole/hr	20.2	20.3	27.0	20.2	40.5	20.4	27.0	20.4
Wt % based on slurry	2.01	2.00	2.05	2.01	2.00	2.00	2.00	2.00
MSCF^{f}/ton of coal	25.9	25.8	26.5	26.0	25.9	25.6	25.6	25.8
Reactor Pressure, psig	3000	2500	3000	3000	2000	3000	2500	3000
Nominal Dissolver Temperature, °C	470	480	480	480	460	480	470	480
°F	878	896	896	896	860	896	878	896
Average Dissolver Temperature, °C	466	476	476	476	458	476	465	476
Temperature Profile, °C^{g}								
Zone 6 (top)	470±1	480±1	480±1	480±0	459±1	480±1	469±1	479±1
5	470±1	480±1	480±1	480±0	460±0	480^{h}±1	470^{h}±1	480±1
4	469±1	480±0	480±1	480±0	460±1	480±1	470±1	481±2
3	469±1	480±1	480±1	480±0	460±1	482±1	471±1	481±2
2	469±1	480±1	480±3	480±0	460±3	479±1	468±1	478±1
1 (bottom)	447±1	457±1	456±3	457±1	446±1	455±4	450±2	454±2
Preheater Temperature, °C^{g}								
Top	397±4	397±1	399±1	399±1	399±1	403±3	396±1	401±5
Middle	401±2	401±1	392±1	401±1	385±2	398±1	392±1	393±4
Bottom	374±1	371±1	356±2	370±3	345±5	371±2	368±1	349±3

a) Old Ben Mine No. 1, sample 39393, see Table IIIa for analyses. b) Dry coal basis. c) Volume of dissolver = 235 cm^{3} (0.00828 ft^{3}). d) Volume of preheater = 156 cm^{3} (.00553 ft^{3}) for run DOE 400 and 240 cm^{3} (0.00846 ft^{3}) for all other runs. e) Hydrogen feed rates do not include that added as DP cell purge. f) One thousand cubic feet at one atmosphere and 60°F. g) Average and standard deviation. h) Skin temperature.

TABLE I (Continued)

	DOE 404RA	DOE 404RB	DOE 404RC	DOE 405RA	DOE 405RB
Coal	Loveridge[a]				
Operating Mode	SRC II				
Dissolver	DOE 3			DOE 1	
Slurry Formulation, wt % [b]					
Coal	30.0	30.0	30.0	30.0	30.0
Recycled Coal Solution (UFCS)	65.0	65.5	65.9	65.5	65.9
Recycled Solvent	4.0	4.0	4.0	4.0	4.0
Additive	1.0	0.5	0.1	0.5	0.1
Slurry Composition, wt % [b]					
Coal	30.0	30.0	30.0	30.0	30.0
Middle Distillate (193-288°C, 380-550°F)	6.2	6.3	6.2	6.3	5.3
Heavy Distillate (>288°C, 550°F)	19.4	20.1	20.0	19.5	17.6
SRC	27.8	28.6	31.1	28.7	34.5
Ash (from UFCS)	10.5	9.1	7.6	9.7	7.8
IOM (from UFCS)	5.1	5.4	5.0	5.3	4.7
Additive	1.0	0.5	0.1	0.5	0.1
Total Solids	46.6	45.0	42.7	45.5	42.5
Slurry Feed Rate					
g/hr	1181	1196	1195	1145	1139
lb/hr/ft^3 [c]	69.2	70.1	70.1	69.0	68.6
Dissolver Nominal Slurry Residence time, hr[c]	1.04	1.02	1.02	1.04	1.04
Preheater Nominal Slurry Residence Time, min.[d]	18.1	17.9	17.9	18.7	18.8
Coal Feed Rate [b]					
g/hr	352	358	357	342	341
lb/hr/ft^3 [c]	20.6	21.0	20.9	20.6	20.5
Hydrogen Feed Rate					
Moles/hr	24.6	24.6	24.6	23.8	23.8
Wt % based on slurry	4.17	4.11	4.12	4.16	4.18
MSCF[e]/ton of coal	53.0	52.1	52.2	52.7	52.9
Additive	NiMo on Alumina			CoMo on Alumina	
Addition Rate, wt % based on coal[b]					
as received material	3.33	1.65	0.34	1.67	0.33
as pure FeS_2 or Mo	0.344	0.170	0.035	0.179	0.035
Reactor Pressure, psig	2250				
Dissolver Temperature, °C					
Average	450				
Dissolver Zone [f]					
6 (top)	451±2	451±1	451±1	451±0	451±0
5	450±1	449±1	449±1	450±1	450±1
4	451±1	450±0	450±1	450±0	450±0
3	450±1	450±1	450±0	450±1	450±1
2	450±1	450±1	450±1	451±1	451±1
1 (bottom)	450±2	449±2	448±2	448±1	448±1
Preheater Temperature, °C [f]					
Top	397±2	398±1	397±1	395±2	395±1
Middle	404±2	405±1	404±1	403±2	403±1
Bottom	379±12	383±10	377±7	358±9	356±13

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a) Pittsburgh seam coal from the Loveridge Mine, see Table IIIa for analyses.
b) Dry coal basis.
c) Reactor volume = 1035 cm^3 (.03655 ft^3) for run DOE 405R and 1064 cm^3 (.03757 ft^3) for run DOE 404R; assumed slurry density = 1.15 g/cm^3.
d) Preheater volume = 310 cm^3.
e) One thousand cubic feet at 60°F and 1 atmosphere.
f) Average and standard deviation.

TABLE I (Continued)

	DOE 406RB	DOE 407	DOE 408
Coal	Powhatan No. 1[a]	← Loveridge[b] →	
Operating Mode	SRC II	← Short Residence Time SRC I →	
Dissolver	DOE 3	← SRT[c] →	
Slurry Formulation, wt %[d]			
Coal	30.0	30.0	30.0
Recycled Solvent	5.0	68.2	70.0
Unfiltered Coal Solution (UFCS)	65.0	--	--
Pyrite	--	1.8	--
Slurry Blend Composition, wt %[d]			
Coal	30.0		
Middle Distillate	4.6		
Heavy Distillate	21.5		
SRC	27.4		
Ash (from UFCS)	10.2		
IOM (from UFCS)	6.3		
Total Solids	46.5		
Slurry Feed Rate			
g/hr	1195	1672	1655
lb/hr/ft^3 [e]	70.1	496	491
Dissolver Nominal Slurry Residence Time[e], hr. (min.)	1.02	(8.7)	(8.8)
Preheater Nominal Slurry Residence Time[f], min.	17.9	9.9	10.0
Coal Feed Rate[d]			
g/hr	357	500	494
lb/hr/ft^3	20.9	148	147
Hydrogen Feed Rate[g]			
Moles/hr	24.5	17.4	17.4
Wt % based on slurry	4.10	2.08	2.10
MSCF[h]/ton of coal	52.0	26.3	26.7
Pyrite Addition Rate, wt % based on coal[d]			
as received material	--	6.17	--
as pure FeS_2	--	5.12	--
Reactor Pressure, psig	1800	2000	2000
Dissolver Temperature, °C			
Average	457	460	460
Zone[i]			
6 (top)	458±2	--[j]	--
5	461±1	--	--
4	460±0	--	--
3	457±1	460±0	460±1
2	454±1	461±0	461±1
1 (bottom)	451±2	458±0	459±3
Preheater Temperature, °C[i]			
Top	394±1	400±1	402±3
Middle	398±1	406±1	404±1
Bottom	364±16	361±11	377±9

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a) Pittsburgh seam coal from the Powhatan No. 1 Mine, see Table IIIa, c, d for analyses.
b) Pittsburgh seam coal from the Loveridge Mine, see Table IIIa for analyses.
c) A tubular reactor with an 11/16" ID, 37" long and a 1/4" thermowell.
d) Dry coal basis.
e) Based on reactor volume = 1064 cm^3 (0.03757 ft^3) for run DOE 406R and 210 cm^3 (0.00742 ft^3) for runs DOE 407 and 408. Assumed slurry density = 1.15 gm/cm^3.
f) Preheater volume = 310 cm^3 for run DOE 406R and 240 cm^3 for runs DOE 407 and 408.
g) Hydrogen feed rates do not include that added as DP cell purge.
h) One thousand standard cubic feet at one atmosphere and 60°F.
i) Average and standard deviation.
j) Dissolver occupied only zones 1-3 in runs DOE 407 and 408.

TABLE II

Results Summary

	DOE 381	DOE 382	DOE 383	DOE 384	DOE 385	DOE 386	DOE 387	DOE 388	DOE 389	DOE 390	DOE 391	DOE 392
Yields, wt % based on MF coal[a]												
H_2O	6.2	6.4	4.9	6.1	6.9	4.6	6.6	5.0	5.2	4.1	6.4	6.8
CO	0.3	0.3	0.1	0.2	0.2	--	0.4	0.2	0.2	0.3	0.3	0.3
CO_2	1.3	1.4	1.0	1.0	1.3	1.2	1.1	0.6	0.7	0.8	0.9	1.0
H_2S	1.2	1.4	1.1	1.3	1.1	1.1	1.0	0.8	1.0	1.0	1.1	1.3
NH_3	0.2	0.2	0.1	0.1	0.1	0.1	0.2	0.1	0.1	0.1	0.1	0.1
C_1	2.9	3.1	0.7	2.0	1.2	2.6	2.5	0.5	0.6	1.2	1.2	1.2
C_2	2.2	2.4	0.6	1.5	1.0	1.6	1.6	0.4	0.4	0.7	0.7	0.8
C_3	1.6	1.7	0.4	1.1	0.7	1.1	1.0	0.2	0.2	0.4	0.5	0.5
C_4	1.1	1.1	0.3	0.8	0.4	0.2	0.5	0.1	0.2	0.2	0.3	0.3
Total C_1-C_4	7.8	8.3	2.1	5.4	3.3	5.5	5.6	1.2	1.4	2.5	2.7	2.8
C_{5+} (gas)	0.6	0.4	0.3	0.8	0.3	2.2	1.7	1.0	1.1	1.0	0.9	0.9
Naphtha, $<$193°C	6.3	6.6	2.0	3.9	2.7	3.6	6.3	1.6	2.0	2.8	2.1	2.5
Wash Solvent, 193-249°C	12.4	14.3	11.1	10.0	13.6	9.5	13.0	11.4	13.0	12.0	10.1	10.4
Recycle Solvent, >249°C (loss)	(6.7)	(8.4)	(16.5)	(2.6)	(9.2)	(2.1)	(12.7)	(3.0)	(5.5)	(3.0)	(2.4)	(9.7)
Total Oil, C_5- Recycle Solvent (loss)	12.6	12.9	(3.1)	12.1	7.4	13.2	8.3	11.0	10.6	12.8	10.7	4.1
SRC	59.0	57.8	68.6	62.0	65.2	63.1	65.9	68.6	66.6	65.7	66.1	71.2
Insoluble Organic Matter	5.4	5.3	17.0	5.3	6.6	4.7	4.4	5.5	6.2	5.5	4.9	5.1
Ash	9.1	8.5	9.4	9.3	9.3	9.4	8.8	8.7	9.0	9.1	9.1	9.1
Total	103.1	102.5	101.2	102.8	101.4	102.9	102.3	101.7	101.0	101.9	102.3	101.8
H_2 Consumed (Gas Balance)	3.1	2.5	1.2	2.8	1.4	2.9	2.3	1.7	1.0	1.9	2.3	1.8
Recycle Solvent Analyses												
% C	88.42	88.78	88.73	88.91	88.52	88.48	88.58	88.44	88.83	88.84	88.48	88.36
% H	7.44	7.56	7.58	7.90	7.58	7.66	7.66	7.81	7.83	7.55	7.86	7.78
% S	0.45	0.46	0.52	0.46	0.46	0.41	0.40	0.45	0.45	0.43	0.40	0.40
% N	0.84	0.85	0.81	0.82	0.80	0.82	0.81	0.79	0.78	0.81	0.77	0.80
% O (by difference)	2.85	2.35	2.36	1.91	2.64	2.63	2.55	2.51	2.11	2.37	2.48	2.66
Specific Gravity	1.0553	1.0552	1.0562	1.0477	1.0540	1.0551	1.0507	1.0539	1.0546	1.0531	1.0464	1.0526
SRC Analyses												
% C	86.88	87.29	85.28	86.26	86.06	86.14	86.92	84.51	84.88	85.62	85.40	85.63
% H	5.65	5.44	5.78	5.76	5.58	5.62	5.44	5.70	5.75	5.68	5.72	5.96
% S	0.86	0.86	1.29	0.88	0.74	0.53	0.56	0.84	0.84	0.72	0.68	0.70
% N	2.15	2.19	2.01	2.13	2.12	2.21	2.17	2.05	2.03	2.13	2.14	2.12
% O (by difference)	4.34	4.00	5.44	4.60	5.33	5.20	4.74	6.36	6.14	5.52	5.75	5.03
% Ash	0.12	0.23	0.20	0.37	0.17	0.30	0.17	0.54	0.36	0.33	0.31	0.56
Fusion Point, °C	105	120	181	132	197	165	147	196	170	181	173	167

a) Based on distillation of filtrate.

TABLE II (Continued)

	DOE 393	DOE 394	DOE 395	DOE 396	DOE 397	DOE 398	DOE 399	DOE 400
Yields, wt % based on MF Coal[a]								
H_2O	6.7	6.8	6.7	6.2	4.7	5.4	5.1	4.3
CO	0.3	0.4	0.4	0.4	0.2	0.4	0.3	0.4
CO_2	1.0	1.0	0.9	1.1	0.8	1.1	1.0	1.1
H_2S	1.1	1.2	1.2	1.0	1.0	0.9	0.9	0.9
NH_3	0.2	0.1	0.2	0.2	0.2	0.2	0.1	0.1
C_1	1.5	2.1	1.6	1.9	0.6	2.2	1.3	2.2
C_2	0.9	1.3	1.0	1.2	0.4	1.4	0.8	1.4
C_3	0.5	0.8	0.6	0.7	0.2	0.8	0.4	0.8
C_4	0.3	0.3	0.3	0.3	0.1	0.3	0.2	0.3
Total C_1-C_4	3.2	4.5	3.5	4.1	1.3	4.7	2.7	4.7
C_{5+} (gas)	1.8	1.4	1.3	1.6	1.2	1.2	1.2	1.5
Naphtha, <193°C (380°F)	3.1	3.5	3.0	4.7	2.4	4.1	2.4	3.1
Wash Solvent, 193-249°C (380-480°F)	8.7	10.5	7.7	8.7	10.8	8.5	6.4	6.3
Recycle Solvent, >249°C (480°F) (Loss)	(0.8)	(0.7)	1.7	0.7	(2.6)	(0.4)	(0.5)	4.0
Total Oil, C_5-Recycle Solvent	12.8	14.7	13.7	15.7	11.8	13.4	9.5	14.9
SRC	62.6	60.0	62.4	59.9	65.8	68.7	67.6	61.5
Insoluble Organic Matter	4.9	4.6	4.6	4.7	5.9	4.9	5.0	4.7
Ash	9.5	9.1	9.0	9.4	9.4	9.4	9.3	9.3
Total	102.3	102.4	102.6	102.7	101.1	102.1	101.5	101.9
Hydrogen Consumed (Gas Balance)	2.3	2.4	2.6	2.7	1.1	2.1	1.5	1.9
Recycle Solvent Analyses								
% C	88.50	88.39	88.40	88.33	88.66	88.40	88.46	88.45
% H	7.65	7.52	7.58	7.68	7.48	7.48	7.62	7.94
% S	0.44	0.37	0.39	0.36	0.42	0.35	0.38	0.35
% N	0.80	0.78	0.79	0.83	0.79	0.78	0.79	0.80
% O (by difference)	2.61	2.94	2.84	2.80	2.65	2.99	2.75	2.46
Specific Gravity	1.0497	1.0466	1.0477	1.0560	1.0554	1.0487	1.0463	1.0478
SRC Analyses								
% C	86.17	86.35	85.40	86.18	84.68	85.33	85.63	86.72
% H	5.70	5.70	5.78	5.66	5.66	5.56	5.77	5.64
% S	0.58	0.48	0.57	0.47	0.68	0.54	0.62	0.49
% N	2.14	2.15	2.13	2.19	2.07	2.16	2.11	2.17
% O (by difference)	5.18	4.98	5.69	5.30	6.52	5.09	5.70	4.78
% Ash	0.23	0.34	0.43	0.20	0.39	0.32	0.17	0.20
Fusion Point, °C	190	178	174	169	220	144	154	162

a) Based on distillation of filtrate.

TABLE II (Continued)

	DOE 404RA	DOE 404RB	DOE 404RC	DOE 405RA	DOE 405RB
Yields, wt % based on MF Coal					
H_2O	5.2	5.5	5.0	5.4	5.3
CO	0.3	0.4	0.4	0.3	0.4
CO_2	0.6	0.6	0.8	0.5	0.7
H_2S	1.5	1.6	1.5	1.7	1.5
NH_3	0.8	0.7	0.5	0.6	0.5
C_1	4.9	4.7	5.0	4.7	4.9
C_2	3.0	2.8	2.9	2.8	2.8
C_3	2.7	2.5	2.6	2.5	2.4
C_4	1.7	1.5	1.5	1.7	1.4
Total C_1-C_4	12.3	11.5	12.0	11.7	11.5
C_{5+} (gas)	2.3	2.6	2.0	2.6	1.0
Naphtha, <193°C (380°F)	11.8	10.8	8.7	10.3	8.6
Middle Distillate, 193-288°C (380-550°F)	15.2	15.1	15.8	16.6	15.1
Heavy Distillate, >288°C (550°F)	14.9	14.2	12.8	14.4	12.9
Total Oil (C_5-Heavy Distillate)	44.2	42.7	39.3	43.9	37.6
SRC	28.1	29.0	32.5	28.2	35.4
Insoluble Organic Matter	5.1	5.4	5.2	5.2	4.8
Ash	7.4	7.7	7.6	8.0	7.7
Total	105.5	105.1	104.8	105.5	105.4
H_2 Consumed (Gas Balance)	5.3	5.0	4.8	5.4	5.4
Additive Conversion Byproducts	0.2	0.1	0.0	0.1	0.0
Naphtha Analyses[a]					
% C	86.36	86.25	86.13	86.47	86.23
% H	13.13	13.20	13.30	12.90	12.93
% S	0.02	0.05	0.06	0.05	0.10
% N	0.19	0.17	0.17	0.19	0.21
% O (by difference)	0.30	0.33	0.34	0.39	0.53
Middle Distillate Analyses[a]					
% C	86.52	86.30	85.72	86.45	85.85
% H	9.78	9.70	9.38	9.64	9.45
% S	0.00[b]	0.01	0.00[b]	0.00[b]	0.05
% N	0.96	1.01	1.01	1.06	1.02
% O (by difference)	2.74	2.98	3.89	2.85	3.63
Heavy Distillate Analyses[a]					
% C	89.82	89.84	89.24	89.78	89.41
% H	7.93	8.02	8.28	8.18	8.14
% S	0.00[b]	0.07	0.13	0.13	0.25
% N	0.97	1.03	1.08	1.09	1.20
% O (by difference)	1.28	1.04	1.27	0.82	1.00
Distillation Residue Analyses[a]					
% C	67.27	69.59	72.42	69.90	72.80
% H	4.19	4.20	4.46	4.17	4.43
% S	1.88	1.87	1.69	2.14	2.01
% N	1.16	1.26	1.42	1.28	1.47
% Ash	25.78	23.30	19.37	22.39	19.21
Fusion Point, °C	113	97	106	111	108

a) Based on only one analysis per sample.
b) The detection limit of the sulfur analysis is about 0.005%.

TABLE II (Continued)

	DOE 406RB	DOE 407	DOE 408
Yields, Wt % based on MF Coal[a]			
H_2O	4.4	2.7	2.2
CO	0.4	0.2	0.1
CO_2	0.7	0.7	0.7
H_2S[b]	3.1	1.8	0.9
NH_3	0.4	0.1	0.1
C_1	6.1	1.4	1.4
C_2	3.5	0.7	0.7
C_3	3.0	0.5	0.5
C_4	1.7	0.3	0.3
Total C_1-C_4	14.3	2.9	2.9
C_{5+} (gas)	2.9	0.7	0.9
Naphtha, $<$193°C (380°F)	11.0	3.4	3.1
Middle Distillate, 193-288°C (380-550°F)[c]	15.2	6.9	8.5
Heavy Distillate, $>$288°C (550°F)[d] (loss)	12.3	(3.8)	(8.2)
Total Oil (C_5-Heavy Distillate[c])	41.4	7.2	4.3
SRC	24.8	72.7	74.5
Insoluble Organic Matter	5.7	7.9	8.0
Ash	9.2	7.5	7.7
Total	104.4	103.7	101.4
Hydrogen Consumed (Gas Balance)	4.4	1.6	1.4
Pyrite Conversion Byproducts	--	2.1	--
Naphtha Analysis			
% C	86.19		
% H	12.75		
% S	0.23		
% N	0.28		
% O (by difference)	0.55		
Middle Distillate Analysis			
% C	85.49		
% H	9.02		
% S	0.13		
% N	1.03		
% O (by difference)	4.33		
Heavy Distillate Analysis[d]			
% C	89.07	88.46	38.72
% H	7.68	7.56	7.66
% S	0.50	0.43	0.43
% N	1.16	0.93	0.79
% O (by difference)	1.59	2.62	2.40
Distillation Residue Analysis[e]			
% C	67.73	86.25	86.50
% H	3.69	5.69	5.75
% S	3.83	1.15	1.24
% N	1.35	1.87	1.82
% Ash	23.67	0.65	0.37
% O (by difference)	--	4.39	4.32
Fusion Point, °C	102	132	141

a) Based on distillation of filtrate for runs DOE 407 and 408.
b) Includes H_2S derived from added pyrite.
c) Wash Solvent, 193-249°C (380-480°F) for runs DOE 407 and 408.
d) Recycle Solvent, $>$249°C (480°F) for runs DOE 407 and 408.
e) SRC for runs DOE 407 and 408.

TABLE III

Analyses of Feed Coals

a. Elemental Analyses[a]

Coal Identification			
Company	Old Ben	Consol	North American
Mine	Old Ben No. 1	Loveridge	Powhatan No. 1
Seam	Indiana V	Pittsburgh	Pittsburgh
Proximate Analysis			
% Ash	8.70	7.44	9.08
% Volatile	38.26	39.13	41.97
% Fixed Carbon	53.04	53.43	48.95
	100.00	100.00	100.00
% Moisture	4.51	0.92	3.85
Heating Value, Btu/lb	12,718	13,958	13,444
Sulfur Forms			
% Pyrite	1.03	0.90	2.38
% Sulfate	0.46	0.00	0.10
% Organic	1.33	1.80	2.36
% Total	2.82	2.70	4.84
Free Swelling Index	1	8	6
Ultimate Analyses, Wt %			
Carbon	71.63	77.25	73.07
Hydrogen	4.77	5.34	4.95
Nitrogen	1.47	1.13	1.30
Chlorine	0.03	0.10	0.10
Sulfur	2.82	2.70	9.08
Ash	8.70	7.44	4.84
Oxygen (difference)	10.58	6.04	6.66
	100.00	100.00	100.00
Mineral Analysis of Ash, Wt %, ignited basis			
Silica, SiO_2	44.35	42.71	34.07
Alumina, Al_2O_3	21.09	21.98	19.54
Titania, TiO_2	1.07	1.10	0.74
Ferric Oxide, Fe_2O_3	24.68	17.67	39.63
Lime, CaO	2.39	5.70	1.65
Magnesia, MgO	0.90	1.25	0.53
Potassium Oxide, K_2O	2.30	1.19	1.05
Sodium Oxide, Na_2O	0.22	1.12	0.56
Sulfur Trioxide, SO_3	1.90	6.17	2.13
Phos. Pentoxide, P_2O_5	0.34	0.17	0.00
Strontium Peroxide, SrO_2	--	--	0.03
Barium Oxide, BaO	--	--	0.02
Manganese Oxide, MnO	--	--	0.05
Undetermined	0.60	0.94	--
	100.00	100.00	100.00
% Iron in coal	1.50	0.92	2.52
Fusion Temperature of Ash, °F (Reducing)			
Initial Deformation	--	2080	2150
Softening (H=W)[b]	--	2190	2220
Softening (H=1/2W)	--	2220	2300
Fluid	--	2430	2400
% Equilibrium Moisture	--	--	2.01

a) All analyses performed by Commercial Testing & Engineering Company. All analyses except moisture are on a dry basis.

b) H = cone height; W = cone width.

TABLE III (Continued)

b. Kentucky 9/14 Elemental Analyses[a]

	Wt %
Moisture	
ASTM D-3173 (oven drying)	3.18
ASTM D-95 (toluene distillation)	4.08
Ash	9.09
Iron in ash	17.58
Iron in coal	1.60
Sulfur	3.28

a) Analyses by Merriam Laboratory, dry basis (D-95) except for moisture.

TABLE III (Continued)

c. Powhatan No. 1 Coal Petrographic Analysis[a]

MACERAL ANALYSIS
(VOLUME PERCENT)
(MINERAL-MATTER CONTAINING BASIS)

MACERAL		MACERAL GROUP	
VITRINITE	73.2	VITRINITE	74.2
PSEUDOVITRINITE	1.0		
EXINITE	3.7	EXINITE (LIPTINITE)	4.2
RESINITE	0.5		
SEMI-FUSINITE*	5.7		
SEMI-MACRINITE*	0.3		
FUSINITE	4.6	INERTINITE	15.3
MACRINITE	0.2		
MICRINITE	4.5		
MINERAL MATTER**	6.3		6.3
TOTAL	100%		100%

TOTAL REACTIVES- 80.4
TOTAL INERTS- 19.6

++++++++++++

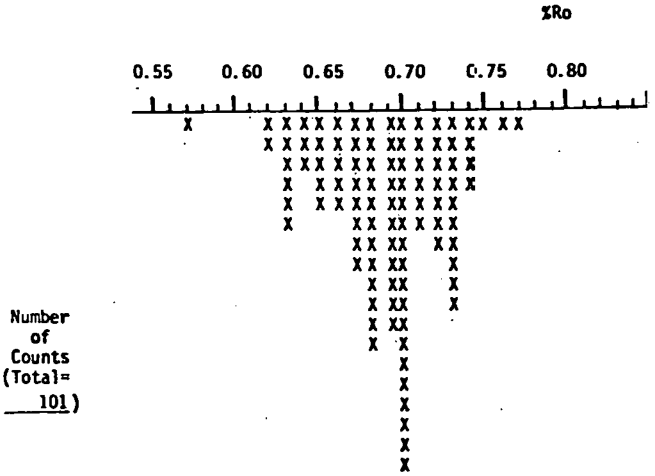
* CONSIDERED 1/3 REACTIVE, 2/3 INERT FOR PURPOSES OF COKE STABILITY PREDICTIONS.

** CALCULATED FROM 9.08 % DRYASH, 4.84 % DRY SULFUR

REFLECTANCE ANALYSIS

Mean-Maximum Vitrinite Ro- 0.69

Distribution of Vitrinite Reflectance Readings:



V-Type Table for Vitrinites (=100%)

V-5	V-6	V-7	V-
1.0	51.5	47.5	

V-Type Table for Vitrinites (= 74.2%)
(Adjusted to = Maceral % of Reactive Vitrinites)

V-5	V-6	V-7	V-
0.8	38.2	35.2	

a) Analysis performed by Commercial Testing and Engineering Company.

TABLE III (Continued)

d. Powhatan No. 1 Coal Float & Sink Analysis[a]

FLOAT & SINK ANALYSIS

1	2	3	4	5	6	7	8	9	10	11
SPECIFIC GRAVITY			DRY BASIS		CUM. RECOVERY (FLOAT)			CUM. REJECT (SINK)		
Sink	Float	% Wt.	% Ash	% Sul.	% Wt.	% Ash	% Sul.	% Wt.	% Ash	% Sul.
14 Mesh x 0 = 100% of Sample										
	1.30	23.6	3.40	2.80	23.6	3.40	2.80	100.0	9.15	4.83
1.30	1.40	53.4	6.43	3.54	77.0	4.46	3.31	76.4	10.92	5.46
1.40	1.60	17.3	14.89	6.74	94.3	7.22	3.94	23.0	21.35	9.91
1.60	1.90	3.2	29.98	12.82	97.5	7.97	4.23	5.7	40.97	19.53
1.90	-	2.5	55.03	28.11	100.0	9.15	4.83	2.5	55.03	28.11

FLOAT & SINK ANALYSIS

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
SPECIFIC GRAVITY			DRY BASIS % SULFUR				CUMULATIVE RECOVERY (FLOAT) % SULFUR					CUMULATIVE REJECT (SINK) % SULFUR				
Sink	Float	% Wt.	Total	Pyritic	Sulf.	Org.	% Wt.	Total	Pyritic	Sulf.	Org.	% Wt.	Total	Pyritic	Sulf.	Org.
14 Mesh x 0 = 100% of Sample																
	1.30	23.6	2.80	0.26	0.02	2.52	23.6	2.80	0.26	0.02	2.52	100.0	4.83	2.32	0.06	2.45
1.30	1.40	53.4	3.54	0.92	0.05	2.57	77.0	3.31	0.72	0.04	2.55	76.4	5.46	2.95	0.07	2.43
1.40	1.60	17.3	6.74	4.44	0.09	2.21	94.3	3.94	1.40	0.05	2.49	23.0	9.91	7.67	0.13	2.11
1.60	1.90	3.2	12.82	10.75	0.16	1.91	97.5	4.23	1.71	0.05	2.47	5.7	19.53	17.48	0.25	1.80
1.90		2.5	28.11	26.10	0.36	1.65	100.0	4.83	2.32	0.06	2.45	2.5	28.11	26.10	0.36	1.65

a) Analysis performed by Commerical Testing and Engineering Company.

TABLE IV

Process Solvent Analyses*
Runs DOE 381-395 and 397-400

a. Elemental Analyses

Drum No.	% C	% H	% S	Basic % N	Total % N	% O	Specific Gravity
1927-1	88.58	7.42	0.38	0.50	0.80	2.82	1.0476
1927-2	88.58	7.85	0.40	0.52	0.78	2.39	1.0466
1927-3	88.60	7.79	0.41	0.53	0.79	2.41	1.0484
1927-4	88.75	7.56	0.41	0.51	0.79	2.49	1.0471
1927-5	88.70	7.86	0.43	0.53	0.79	2.22	1.0472
Average	88.64	7.70	0.41	0.52	0.79	2.46	1.0474
Std. Dev.	±0.08	±0.20	±0.02	±0.01	±0.01	±0.22	±0.0007

b. Simulated Distillation (by Gas Chromatography)

	Drum 1927-1	Drum 1927-2	Drum 1927-3	Drum 1927-4	Drum 1927-5
Fraction Boiling Below, %	Temp °C	Temp °C	Temp °C	Temp °C	Temp °C
IBP	81.57	104.7	97.52	104.5	89.78
5	231.0	231.0	231.0	230.6	231.0
10	238.9	238.9	239.0	238.8	238.9
15	254.3	254.3	254.3	253.9	254.3
20	262.9	262.9	263.0	262.6	262.9
25	271.5	271.5	271.6	271.3	271.5
30	279.2	279.2	279.3	279.2	279.3
35	286.8	286.8	286.8	286.7	286.8
40	294.3	294.3	294.4	294.2	294.3
45	301.9	301.3	309.8	309.3	301.9
50	310.2	310.2	310.2	310.1	310.2
55	318.2	318.2	318.3	318.0	318.2
60	318.5	318.5	318.5	318.5	318.5
65	326.5	326.4	334.0	333.5	334.0
70	334.4	334.4	342.0	341.6	342.0
75	349.9	349.9	350.0	349.8	350.0
80	357.7	357.7	357.7	357.6	357.7
85	365.5	365.5	372.8	372.4	372.8
90	380.9	380.8	380.9	387.8	388.2
95	404.6	404.4	404.6	404.6	404.6
FBP	461.4	461.0	461.2	460.8	461.2
Oil, IBP-193°C	3.86	3.65	3.72	3.66	3.79
Oil, 193-249°C	9.42	9.63	9.55	9.72	9.49
Oil, >249°C	86.72	86.72	86.73	86.62	86.72
Total	100.00	100.00	100.00	100.00	100.00

* Lot 1927 from Ft. Lewis.

TABLE V

Additives Analysis

Composition, wt %[a]	Robena Pyrite III	NiMo on alumina (HDS 1442)	CoMo on alumina (HDS 1443)
Fe	33.8	--	--
S	41.4	--	--
C	5.4	--	--
H	0.40	--	--
CoO	--	3.0	--
NiO	--	--	3.8
MoO_3	--	16.1	15.5
Na_2O	--	0.01	0.02
SO_4	--	0.3	0.6
Moisture[b]	0.22	--	--
Ash[c]	65.8	94.0	94.0
Mole Ratio S/Fe	1.86	--	--
Purity, as FeS_2	83	--	--
Physical Properties[d]			
Average Particle Size, μm		Through 150 mesh	
by Fisher Subsieve Sizer	0.95		
by Coulter Counter	3.5		
Surface Area, m^2/g	2.61[e]	287[f]	312[f]

a) Dry basis. b) As volatiles at 110°C under He atmosphere. c) Wet basis. d) As fed. e) Nitrogen adsorption B.E.T. measurement by Sandia Laboratories. f) B.E.T. measurement by Coors Spectro-Chemical Laboratory.

TABLE VI

Unfiltered Coal Solution Viscosities[a]
Runs DOE 381-408

Run No. DOE	Temperature °F	Shear Rate sec^{-1}	Viscosity cP
381 P 18	210	39.64	10.7
	210	79.28	9.95
	230	39.64	8.30
	230	79.28	7.70
	270	39.64	5.40
	270	79.28	4.95
382 P 16	210	15.86	17.0
	210	39.64	13.2
	210	79.28	12.2
	230	39.64	9.80
	230	79.28	9.10
	270	39.64	6.60
	270	79.28	5.70
383 P 14	210	7.93	48.5
	210	15.86	44.8
	210	39.64	40.7
	210	79.28	37.4
	230	15.86	32.5
	230	39.64	29.4
	230	79.28	26.7
	270	15.86	19.5
	270	39.64	16.4
	270	79.28	14.6
384 P 14	210	15.86	14.2
	210	39.64	11.8
	210	79.28	11.1
	230	39.64	9.30
	230	79.28	8.45
	270	39.64	6.20
	270	79.28	5.45
385 P 15	210	7.93	54.5
	210	15.86	47.8
	210	39.64	44.6
	210	79.28	39.8
	230	7.93	36.0
	230	15.86	33.2
	230	39.64	30.9
	230	79.28	27.8
	270	15.86	19.0
	270	39.64	16.1
	270	79.28	14.6
386 P 15	210	39.64	12.1
	210	79.28	11.6
	230	39.64	9.70
	230	79.28	8.60
	270	39.64	6.40
	270	79.28	5.60
387 P 14	210	15.86	44.5
	210	39.64	40.5
	210	79.28	37.8
	230	15.86	32.0
	230	39.64	28.7
	230	79.28	26.3
	270	39.64	16.1
	270	79.28	14.2
388 P 15	210	7.93	63.5
	210	15.86	54.0
	210	39.64	49.9
	210	79.28	47.4
	230	7.93	43.0
	230	15.86	37.0
	230	39.64	33.6
	230	79.28	31.8
	270	15.86	21.5
	270	39.64	18.1
	270	79.28	16.2
389 P 15	210	7.93	91.0
	210	15.86	80.5
	210	39.64	72.8
	210	79.28	63.8
	230	7.93	55.0
	230	15.86	48.5
	230	39.64	44.3
	230	79.28	37.6
	270	15.86	23.8
	270	39.64	19.8
	270	79.28	17.6
390 P 15	210	7.93	42.0
	210	15.86	34.0
	210	39.64	29.7
	210	79.28	27.6
	230	15.86	24.2
	230	39.64	21.9
	230	79.28	20.2
	270	15.86	16.5
	270	39.64	13.4
	270	79.28	11.4
391 P 15	210	15.86	21.0
	210	39.64	18.8
	210	79.28	17.5
	230	39.64	13.3
	230	79.28	12.4
	270	39.64	8.00
	270	79.28	7.35
392 P 15	210	15.86	25.2
	210	39.64	22.9
	210	79.28	21.6
	230	39.64	16.4
	230	79.28	15.2
	270	39.64	9.20
	270	79.28	8.45

TABLE VI (Continued)

Run No. DOE	Temperature °F	Shear Rate sec^{-1}	Viscosity cP
393 P 15	210	39.6	14.9
	210	79.3	14.3
	230	39.6	10.6
	230	79.3	10.2
	270	39.6	6.70
	270	79.3	6.15
394 P 14	210	39.6	11.3
	210	79.3	11.0
	230	39.6	8.40
	230	79.3	8.15
	270	39.6	5.10
	270	79.3	4.85
395 P 15	210	39.6	13.8
	210	79.3	13.1
	230	39.6	9.50
	230	79.3	9.15
	270	39.6	6.20
	270	79.3	5.75
396 P 47	210	39.6	12.4
	210	79.3	12.1
	230	39.6	9.40
	230	79.3	9.15
	270	39.6	6.10
	270	79.3	5.65
397 P 15	210	15.9	71.0
	210	39.6	65.5
	210	79.3	59.4
	230	7.93	48.0
	230	15.9	43.0
	230	39.6	37.5
	230	79.3	33.4
	270	15.9	22.0
	270	39.6	18.1
	270	79.3	16.4
398 P 12	210	39.6	12.5
	210	79.3	11.9
	230	39.6	9.50
	230	79.3	8.85
	270	39.6	6.00
	270	79.3	5.60
399 P 12	210	15.9	20.2
	210	39.6	17.7
	210	79.3	17.0
	230	39.6	12.8
	230	79.3	12.0
	270	39.6	7.75
	270	79.3	7.10
400 P 12	210	15.9	14.0
	210	39.6	12.5
	210	79.3	11.9
	230	39.6	9.20
	230	79.3	8.90
	270	39.6	6.10
	270	79.3	5.63

Run No. DOE	Temperature °F	Shear Rate sec^{-1}	Viscosity cP
404RA P 58	210	0.396	1.78×10^3
	210	0.793	1.20×10^3
	210	1.98	868
	210	3.96	672
	230	0.396	1.57×10^3
	230	0.793	945
	230	1.98	584
	230	3.96	441
	230	7.93	349
	270	0.793	810
	270	1.98	414
	270	3.96	275
	270	7.93	186
	270	15.9	142
404RB P 100	210	0.395	1.12×10^3
	210	0.793	860
	210	1.98	644
	210	3.96	546
	210	7.93	479
	230	0.793	645
	230	1.98	420
	230	3.96	342
	230	7.93	292
	270	1.98	270
	270	3.96	187
	270	7.93	148
	270	15.9	120
	270	39.6	93.0

TABLE VI (Continued)

Run No. DOE	Temperature °F	Shear Rate sec^{-1}	Viscosity cP
404RC P 143	210	0.793	1.10×10^3
	210	1.98	882
	210	3.96	774
	230	0.793	710
	230	1.98	558
	230	3.96	438
	230	7.93	371
	270	1.98	280
	270	3.96	207
	270	7.93	167
	270	15.9	136
405RA P 90	210	0.793	960
	210	1.98	744
	210	3.96	634
	230	0.793	690
	230	1.98	494
	230	3.96	401
	230	7.93	333
	270	1.98	286
	270	3.96	214
	270	7.93	162
	270	15.9	130
405RB P 144	210	0.396	6.07×10^4
	210	0.793	5.46×10^4
	230	0.396	3.57×10^4
	230	0.793	2.85×10^4
	230	1.98	2.33×10^4
	270	0.396	2.01×10^4
	270	0.793	1.45×10^4
	270	1.98	9.90×10^3
	270	3.96	7.42×10^3
	270	7.93	6.07×10^3

Run No. DOE	Temperature °F	Shear Rate sec^{-1}	Viscosity cP
406RA P 94	210	0.793	790
	210	1.98	628
	210	3.96	573
	230	1.98	382
	230	3.96	333
	230	7.93	292
	270	1.98	264
	270	3.96	201
	270	7.93	162
	270	15.9	127
	270	39.6	99.4
406RB P 131	210	0.396	2.25×10^3
	210	0.793	2.00×10^3
	210	1.98	1.75×10^3
	230	0.396	1.38×10^3
	230	0.793	1.14×10^3
	230	1.98	888
	230	3.96	772
	270	0.793	660
	270	1.98	416
	270	3.96	322
	270	7.93	272
	270	15.9	234
	310	1.98	292
	310	3.96	201
	310	7.93	156
	310	15.9	124
	310	39.6	95.5
407 P 14	210	15.9	16.9
	210	39.6	15.6
	210	79.3	15.2
	230	39.6	11.4
	230	79.3	11.0
	270	39.6	7.40
	270	79.3	6.70
408 P 14	210	15.9	18.2
	210	39.6	16.8
	210	79.3	16.1
	230	39.6	12.3
	230	79.3	11.7
	270	39.6	7.4
	270	79.3	7.1

a) Brookfield Model LV Viscometer
Spindle No. 18
Procedure: 45 min. initial warmup and equilibration;
20-30 minute equilibration after temperature change;
10 minute equilibration after shear rate change.

TABLE VII

Feed Slurry Viscosities[a]
Runs DOE 404R-406R

Run No. DOE	Temperature °F	Shear Rate sec^{-1}	Viscosity 10^4 cP
404RA P 58	210	0.083	11.20
	210	0.166	7.31
	230	0.083	9.52
	230	0.166	6.52
	270	0.083	11.0
	270	0.166	6.89
404RB P 101	210	0.083	4.96
	210	0.166	2.83
	210	0.415	1.53
	210	0.830	1.01
	210	1.66	0.738
	230	0.083	4.74
	230	0.166	2.47
	230	0.415	1.47
	230	0.830	1.09
	270	0.166	2.30
	270	0.415	1.41
	270	0.830	0.848
	270	1.66	0.561
404RC P 143	210	0.083	3.17
	210	0.166	2.45
	210	0.415	1.81
	210	0.830	1.49
	230	0.083	3.65
	230	0.166	2.29
	230	0.415	1.47
	230	0.830	0.986
	270	0.083	3.22
	270	0.166	2.26
	270	0.415	1.41
	270	0.830	1.04

Run No. DOE	Temperature °F	Shear Rate sec^{-1}	Viscosity 10^4 cP
405RA P 97	210	0.083	10.3
	210	0.166	5.46
	230	0.083	7.83
	230	0.166	4.58
	230	0.415	2.80
	270	0.083	9.60
	270	0.166	5.58
	270	0.415	2.92
405RB P 144	210	0.083	7.26
	210	0.166	5.76
	230	0.083	8.23
	230	0.166	5.57
	270	0.083	10.4
	270	0.166	5.77
406RA P 94	210	0.083	45.7
	210	0.166	27.1
	210	0.415	16.0
	210	0.830	11.2
	230	0.083	38.5
	230	0.166	24.7
	230	0.415	13.3
	230	0.830	8.83
	230	1.66	5.70
	270	0.083	49.3
	270	0.166	32.4
	270	0.415	16.1
	270	0.830	9.56
	270	1.66	6.02
406RB P 131	210	0.083	61.4
	210	0.166	41.4
	210	0.415	26.9
	230	0.083	66.9
	230	0.166	40.5
	230	0.415	23.2
	270	0.083	82.9
	270	0.166	42.1
	270	0.415	21.6
	270	0.830	14.9
	310	0.083	124
	310	0.166	71.4

a) Brookfield Model LV viscometer
Spindle 28
Procedure: 45 minutes initial warmup and equilibration;
20-30 minute equilibration after temperature change;
10 minute equilibration after shear rate change.

TABLE VIII

Solvent Extraction of SRC Product
Runs DOE 381-384, Kentucky 9/14 Coal;
DOE 385-400, Indiana V Coal; DOE 407 & 408, Loveridge Coal

Liquefaction Conditions	DOE 381 P 19	DOE 382 P 14	DOE 383 P 15	DOE 384 P 14	DOE 385 P 12	DOE 385 P 14	DOE 386 P 15	DOE 387 P 12	DOE 387 P 15	DOE 388 P 12	DOE 388 P 15	DOE 389 P 15	DOE 390 P 10	DOE 390 P 15	DOE 391 P 15	DOE 392 P 15
Coal	Kentucky 9/14				Indiana V											
Temperature, °C	483	483	460	472	474		477	476		456		457	472		458	458
Pressure, psig	3000	2000	2000	3000	2000		3000	2000		3000		2000	3000		3000	2000
Residence Time, min.	8	8	4	8	4		8	8		4		4	4		8	8
Solubility of the SRC																
Wt % in																
Hexane	20.2	23.6	20.7	18.0	6.0	3.8	15.0	12.2	21.9	3.7	1.7	13.6	6.0	8.0	12.2	11.4
	21.1	27.1	22.2	19.0	6.8	3.3	17.2	12.1	23.2	3.6	2.4	13.5	6.4	8.4	13.1	13.6
Toluene	43.7	61.6	68.3	75.1	66.6	61.5	71.0	66.2	72.4	62.5	60.0	67.0	68.4	64.0	72.1	68.2
	43.8	66.5	65.7	70.1	65.3	60.1	72.3	67.7	71.4	61.7	56.0	66.9	69.1	65.5	69.7	68.9
Average Solubility																
Hexane	20.7	25.4	21.5	18.5	5.0		16.1	17.4		2.9		13.6	7.2		12.7	12.5
Toluene	43.8	64.1	67.0	72.6	63.4		71.7	69.5		60.1		67.0	66.8		70.9	68.6

TABLE VIII (Continued)

	DOE 393 P 14	DOE 394 P 14	DOE 395 P 15	DOE 396 P 47	DOE 397 P 15	DOE 398 P 12	DOE 399 P 12	DOE 400 P 12	DOE 407 P8/12/13	DOE 408 P8/10/13/14
Liquefaction Conditions										
Coal	← Indiana V →								← Loveridge →	
Temperature, °C	470	480	480	480	460	480	470	480	← 460 →	
Pressure, psig	3000	2500	3000	3000	2000	3000	2500	3000	← 2000 →	
Dissolver Residence Time, min.	8	8	6	8	4	8	6	8	← 9 →	
Preheater Residence Time, min.	8	8	6	8	4	8	6	5	← 10 →	
Solvent Source	← Single Pass →			Recycled	← Single Pass →					
FeS_2 Addition Rate, wt % MF coal	--	--	--	--	--	--	--	--	5.1	--
Solubility of the SRC, wt % in										
Hexane	16.7	17.5	10.8	12.4	3.6	19.8	12.9	16.1	16.5	13.2
	--	18.4	12.9	13.7	4.1	21.0	13.8	17.4	16.8	14.4
Toluene	73.9	63.4	62.7	69.3	57.2	67.1	68.0	61.8	74.8	72.4
	72.4	65.8	64.8	71.1	56.3	65.6	66.6	65.6	74.5	72.8
Average Solubility										
Hexane	16.7	18.0	11.9	13.1	3.9	20.4	13.4	16.8	16.7	13.8
Toluene	73.2	64.6	63.8	70.2	56.8	66.4	67.3	63.7	74.7	72.6

TABLE IX

Solvent Extraction of Distillation Residues
Runs DOE 404R-406R

Liquefaction Conditions*	DOE 404RA P 48/ 52/58	DOE 404RB P 86/ 96/101	DOE 404RC P132/137/ 143/146	DOE 405RA P 80/ 85/97	DOE 405RB P 135/ 141/144	DOE 406RB P 117/123/ 131/133
Coal	← Loveridge →					Powhatan No. 1
Additive	← NiMo on alumina →			← CoMo on alumina →		--
Addition Rate, wt % based on slurry	1.0	0.5	0.1	0.5	0.1	--
Solubility of the Distillation Residue, wt % in						
Hexane	32.7 30.3	32.7 32.8	35.0 33.2	38.5 37.4	35.5 33.4	21.0 21.9
Toluene	57.3 57.1	60.6 60.8	65.1 65.4	62.4 62.5	64.0 64.1	53.1 53.2
Pyridine	62.1 61.9	64.5 64.6	70.0 69.8	66.2 66.1	71.5 69.6	61.5 61.5
Ratio of Average Solubilities						
Hexane/Pyridine	0.508	0.507	0.488	0.573	0.488	0.350
Toluene/Pyridine	0.923	0.940	0.934	0.943	0.908	0.865

* For all: 450°C, 2250 psig, 1.0 hour residence time.

TABLE X

Results of 2^3 Factorial (Runs DOE 385-392)

Constant Conditions

30% Indiana V Coal
2% Hydrogen on slurry
Single Pass Solvent

Variable Conditions

	Level -	Level +
A. Temperature °C	460	480
B. Pressure, psig	2000	3000
C. Residence Time, min.	4	8

Run No. DOE-	Yates Order	A Temperature	B Pressure	C Residence Time	Yields, wt % MF Coal C_1-C_4	Yields, wt % MF Coal Recycle Solvent	Yields, wt % MF Coal Total Oil	Yields, wt % MF Coal SRC	Yields, wt % MF Coal IOM	H_2 Consumed	SRC % H	SRC % S	SRC Fusion Point, °C	SRC Solubility in Hexane, wt %	UFCS Viscosity* cP
389	(1)	-	-	-	1.4	-5.5	10.6	66.6	6.2	1.0	5.75	0.84	170	13.6	44.3
385	a	+	-	-	3.3	-9.2	7.4	65.2	6.6	1.4	5.58	0.74	197	5.0	30.9
388	b	-	+	-	1.2	-3.0	11.0	68.6	5.5	1.7	5.70	0.84	196	2.9	33.6
390	ab	+	+	-	2.5	-3.0	12.8	65.7	5.5	1.9	5.68	0.72	181	7.2	21.9
392	c	-	-	+	2.8	-9.7	4.1	71.2	5.1	1.8	5.96	0.70	167	12.5	16.4
387	ac	+	-	+	5.6	-12.7	8.3	65.9	4.4	2.3	5.44	0.56	147	17.4	28.7
391	bc	-	+	+	2.7	-2.4	10.7	66.1	4.9	2.3	5.72	0.68	173	12.7	13.3
386	abc	+	+	+	5.5	-2.1	13.2	63.1	4.7	2.9	5.62	0.53	165	16.1	9.7
	Main Effects														
	A				2.2	-1.6	1.3	-3.2	-0.1	0.4	-0.20	-0.13	-4	1.0	-4.1
	B				-0.3	6.7	4.3	-1.4	-0.4	0.6	0.00	-0.02	9	-2.4	-10.5
	C				2.1	-1.6	-1.4	0.1	-1.2	0.8	0.01	-0.17	-23	7.5	-15.7
	Two Factor Interactions														
	AB				-0.2	1.8	0.8	0.2	0.0	0.0	0.14	-0.01	-8	2.9	-3.6
	AC				0.6	0.3	2.0	-1.0	-0.3	0.1	-0.11	-0.02	-10	3.2	8.5
	BC				0.2	2.3	1.4	-2.6	0.5	0.0	-0.03	-0.01	4	1.9	-0.6
	Three Factor Interaction														
	ABC				0.2	-0.1	-1.7	1.0	0.2	0.1	0.07	0.00	14	3.6	-4.4

* Viscosity measured at 230°F and a shear rate of 39.6 sec^{-1}.

TABLE XI

Results of 2^2 Factorial
(Runs DOE 386, 387, 391 and 392)

Constant Conditions

30% Indiana V Coal
2% Hydrogen on Slurry
Single Pass Solvent
8 minutes Residence Time

Variable Conditions

	Level -	Level +
A. Temperature, °C	460	480
B. Pressure, psig	2000	3000

Run No. DOE-	Yates Order	A Temperature	B Pressure	C_1-C_4	Recycle Solvent	Total Oil	SRC	IOM	H_2 Consumed	% S in SRC
392	(1)	-	-	2.8	-9.7	4.1	71.2	5.1	1.8	0.70
387	a	+	-	5.6	-12.7	8.3	65.9	4.4	2.3	0.56
391	b	-	+	2.7	-2.4	10.7	66.1	4.9	2.3	0.68
386	ab	+	+	5.5	-2.1	13.2	63.1	4.7	2.9	0.53

	C_1-C_4	Recycle Solvent	Total Oil	SRC	IOM	H_2 Consumed	% S in SRC
Main Effects							
A	2.8	-1.4	3.4	-4.2	-0.5	0.6	-0.15
B	-0.1	9.0	5.8	-4.0	0.1	0.6	-0.03
Two Factor Interaction							
AB	0	1.7	-0.9	1.2	0.3	0.1	-0.01

TABLE XII

Results of 2^2 Factorial
(Runs DOE 385, 388, 389 and 390)

Constant Conditions

30% Indiana V Coal
2% Hydrogen on Slurry
Single Pass Solvent
4 Minutes Residence Time

Variable Conditions

	Level -	Level +
A. Temperature	460	480
B. Pressure, psig	2000	3000

Run No. DOE-	Yates Order	A Temperature	B Pressure	C_1-C_4	Recycle Solvent	Total Oil	SRC	IOM	H_2 Consumed	% S in SRC
389	(1)	-	-	1.4	-5.5	10.6	66.6	6.2	1.0	0.84
385	a	+	-	3.3	-9.2	7.4	65.2	6.6	1.4	0.74
388	b	-	+	1.2	-3.0	11.0	68.6	5.5	1.7	0.84
390	ab	+	+	2.5	-3.0	12.8	65.7	5.5	1.9	0.72

Main Effects

	C_1-C_4	Recycle Solvent	Total Oil	SRC	IOM	H_2 Consumed	% S in SRC
A	1.6	-1.9	-0.7	-2.2	0.2	0.3	-0.11
B	-0.5	4.4	2.9	1.3	-0.9	0.6	-0.01

Two Factor Interaction

	C_1-C_4	Recycle Solvent	Total Oil	SRC	IOM	H_2 Consumed	% S in SRC
AB	-0.3	1.9	2.5	-0.8	-0.2	-0.1	-0.01

TABLE XIII

DOE 404RA Elemental Balance Study

Loveridge Coal with 1% NiMo catalyst on feed slurry

SUBSTANCE	YIELD	% C	% H	% N	% S	% O	G C	G H	G N	G S	G O
Methane	4.76	74.86	25.13				3.56	1.20			
Ethane	2.92	79.89	20.11				2.33	0.59			
Propane	2.65	81.71	18.29				2.17	0.48			
Butanes	1.51	82.62	17.34				1.25	0.26			
Water	5.51		11.19			88.81		0.62			4.89
CO_2	0.38	27.29				72.71	0.10				0.28
CO	0.25	42.89				57.11	0.11				0.14
Ammonia	0.76		17.75	82.26				0.13	0.63		
H_2S	1.32		5.91		94.09			0.08		1.24	
Naphtha	11.83	86.36	13.13	0.19	0.02	0.30	10.22	1.55	0.022	0.002	0.035
Middle Distillate	19.43	86.52	9.78	0.96	0.002	2.73	16.81	1.900	0.188	-----	0.530
Heavy Distillate	14.20	88.50	7.55	0.97	0.002	2.98	12.57	1.07	0.137	-----	0.423
Residue Ash 25.88%	40.58	67.27	4.19	1.16	1.88	?	27.30	1.70	0.471	0.763	?
TOTALS							76.60	9.58	1.45	2.01	6.30
Input with 100 grams of coal							77.78	5.28	1.14	2.72	5.31
Calc. dry basis (for 1.78% water & 7.55% ash as received) CT&E analysis							76.52*	5.20*	1.41*	2.55*	

ASH OUTPUT	10.50 gm
Cat. ASH	2.96 gm
Ash net	7.54 gm

Hydrogen Reacted from Elemental balance	4.30
Hydrogen Reacted from Gas balance	5.18%

* P & M Analytical Result-

TABLE XIV

DOE 404RB Elemental Balance Study

Loveridge Coal with 0.5% NiMo catalyst on feed slurry

SUBSTANCE	YIELD	% C	% H	% N	% S	% O	G C	G H	G N	G S	G O
Methane	5.06	74.86	25.13				3.79	1.27			
Ethane	3.03	79.89	20.11				2.42	0.61			
Propane	2.70	81.71	18.29				2.21	0.49			
Butanes	1.53	82.62	17.34				1.93	0.40			
Water	5.09		11.19			88.81		0.57			4.52
CO_2	0.51	27.29				72.71	0.14				0.37
CO	0.37	42.89				57.11	0.16				0.21
Ammonia	0.73		17.75	82.26				0.13	0.60		
H_2S	1.82		5.91		94.09			0.11		1.71	
Naphtha	11.11	86.25	13.20	0.17	0.05	0.33	9.58	1.47	0.019	0.006	0.037
Middle Distillate	19.77	85.30	9.70	1.01	0.011	2.98	17.06	1.92	0.197	0.002	0.589
Heavy Distillate	13.49	89.80	7.60	1.03	0.065	1.30	12.11	1.05	0.135	0.009	0.175
Residue (Ash 22.81% INCL. CAT.)	40.22	69.59	4.20	1.26	1.87	1.95 ?	27.99	1.69	0.506	0.752	0.784 ?
TOTALS							77.39	9.71	1.45	2.48	6.69
Input with 100 grams of coal Calc. from CT & E analysis							77.78	5.28	1.14	2.72	5.68
P&M Analytical Results							76.52	5.20	1.41	2.55	5.31

ASH OUTPUT	9.17 gm	Hydrogen reacted by elemental balance	4.43%
Cat. Ash	1.49 Gm		
Net Ash	7.68 gm	Hydrogen reacted by gas balance	5.50
Coal Ash	7.63 gm		

TABLE XV

DOE 404RC Elemental Balance Study

Loveridge Coal with 0.1% NiMo Catalyst on Feed Slurry

SUBSTANCE	YIELDS	% C	% H	% N	% S	% O	G C	G H	G N	G S	G O
Methane	4.91	74.86	25.13				3.68	1.23			
Ethane	2.87	79.89	20.11				2.29	0.58			
Propane	2.53	81.71	18.29				2.07	0.46			
Butanes	1.46	82.62	17.34				1.21	0.25			
Water	4.63		11.19			88.81		0.52			4.11
CO_2	0.60	27.29				72.71	0.16				0.44
CO	0.41	42.89				57.11	0.18				0.23
Ammonia	0.64		17.75	82.26				0.11	0.53		
H_2S	1.94		5.91		94.09			0.11		1.83	
Naphtha	7.61	86.57	13.56	0.17	0.06	?	6.59	1.03	0.0129	0.005	-- --
Middle Distillate	16.00	85.18	9.47	1.01	0.002	4.34	13.63	1.52	0.162	0.0003	0.694
Heavy Distillate	16.85	89.24	8.28	1.08	0.13	1.27	15.04	1.40	0.182	0.022	0.214
Residue (Ash 19.08% Incl. Cat.)	42.63	72.42	4.46	1.42	1.69	?	30.87	1.90	0.605	0.720	?
TOTALS							75.72	9.12	1.49	2.58	5.68
		Input with 100 grams of coal Calc. from CT & E analysis					77.78	5.28	1.14	2.72	5.31
				P&M Analysis			76.52	5.20	1.41	2.55	

Ash Output	8.13 gm	
Cat. Ash	0.31 gm	
Net Ash	7.82 Gm	
Coal Ash	7.86 Gm	

Hydrogen reacted by elemental balance 3.83%

Hydrogen reacted by gas balance 4.62%

TABLE XVI

DOE 405RA Elemental Balance Study

Loveridge Coal with CoMo Catalyst at 0.5% on Feed Slurry

SUBSTANCE	YIELD	% C	% H	% N	% S	% O	G C	G H	G N	G S	G O
Methane	4.64	74.86	25.13				3.47	1.17			
Ethane	2.82	79.89	20.11				2.25	0.57			
Propane	2.46	81.71	18.29				2.01	0.45			
Butanes	1.53	82.62	17.34				1.26	0.27			
Water	4.63		11.19			88.81		0.52			4.11
CO_2	0.51	27.29				72.71	0.14				0.37
CO	0.31	42.89				57.11	0.13				0.18
Ammonia	0.70		17.75	82.26				0.12	0.58		
H_2S	1.54		5.91		94.09			0.09		1.45	
Naphtha	10.31	86.46	13.31	0.19	0.05	----	8.91	1.37	0.020	0.005	-----
Middle Distillate	16.71	86.45	9.64	1.06	0.002	2.84	14.44	1.61	0.178	0.0003	0.475
Heavy Distillate	14.56	89.78	8.18	1.09	0.251	0.82	13.07	1.19	0.158	0.036	0.119
Residue (Ash 21.64% incl. cat. res.)	43.11	69.90	4.17	1.28	2.14	?	30.13	1.80	0.551	0.922	
TOTALS							75.81	9.16	1.487	2.41	5.25
Input with 100 grams of coal as calc. from CT & E analysis							77.78	5.28	1.14	2.72	
Input on basis of P&M analysis							76.52	5.20	1.41	2.55	

Ash output	9.32
Cat. Ash	1.56
Net Ash	7.76
ASH FED	7.77

Hydrogen reacted by elemental balance 3.88%

Hydrogen reacted by gas balance data 4.50

TABLE XVII

DOE 405RB Elemental Balance Study

Loveridge Coal with CoMo Catalyst at 0.1% on Feed Slurry

SUBSTANCE	YIELD	% C	% H	% N	% S	% O	G C	G H	G N	G S	G O
Methane	4.80	74.86	25.13				3.59	1.21			
Ethane	2.79	79.89	20.11				2.23	0.56			
Propane	2.39	81.71	18.29				1.95	0.44			
Butanes	1.42	82.62	17.34				1.17	0.25			
Water	5.08		11.19			88.81		0.57			4.51
CO_2	0.64	27.29				72.71	0.17				0.47
CO	0.36	42.89				57.11	0.15				0.21
Ammonia	0.61		17.75	82.26				0.11	0.50		
H_2S	1.61		5.91	94.09				0.10		1.51	
Naphtha	10.74	86.23	12.93	0.21	0.10	0.53	9.26	1.39	0.023	0.011	0.057
Middle Distillate	22.95	85.85	9.45	1.02	0.045	3.64	19.70	2.17	0.234	0.011	0.835
Heavy Distillate	10.61	89.41	8.14	1.20	0.251	1.00	9.49	0.86	0.127	0.027	0.106
Residue (Ash 19.21% Incl. Cat.)	40.07	72.80	4.43	1.47	2.005	?	29.17	1.78	0.589	0.803	
TOTALS							76.88	9.44	1.473	2.36	
Input with 100 grams of coal by CT & E analysis							77.78	5.28	1.14	2.72	
Input with 100 grams of coal by P & M analysis							76.52	5.20	1.41	2.55	

Ash output	7.70
Catalyst ash	0.31
Net	7.39
Coal Ash	7.39%

Hydrogen reacted by elemental balance	4.16%
Hydrogen reacted by gas consumption data	5.11%

TABLE XVIII

DOE 406RB Elemental Balance Study

Powhatan No. 1 Mine Coal

SUBSTANCE	YIELD	% C	% H	% N	% S	% O	G C	G H	G N	G S	G O
Methane	6.06	74.86	25.13				4.54	1.52			
Ethane	3.47	79.89	20.11				2.77	0.70			
Propane	3.05	81.71	18.29				2.49	0.56			
Butanes	1.71	82.62	17.34				1.41	0.30			
Water	4.46		11.19			88.81		0.50			3.96
CO_2	0.68	27.29				72.71	0.19				0.49
CO	0.41	42.89				57.11	0.18				0.23
Ammonia	00.44		17.75	82.25				0.08	0.36		
H_2S	3.06		5.91		94.09			0.18		2.88	
Naphtha	13.87	86.19	12.75	0.28	0.23	0.55	11.95	1.77	C.039	0.032	0.076
Middle Distillate	15.20	85.49	9.02	1.03	0.13	4.33	12.99	1.37	0.156	0.020	0.658
Heavy Distillate	12.31	89.07	7.67	1.16	0.50	1.60	10.70	0.92	0.139	0.060	0.192
Residue	39.68	67.73	3.69	1.35	3.83	?	26.87	1.46	0.536	1.520	?
Ash 23.67%											
							74.09	9.37	1.23	4.51	5.606
Input with 100 grams of coal by CT & E analysis							73.07	4.95	1.30	4.84	6.66
P&M Analysis									1.26		

Ash Balance

In distillation residue	9.39
By coal analysis	9.08
Difference	0.31
Ash unbalance is on CT & E analysis	3.4%

Average ash fed this run was 9.20 and error was then 0.19

Hydrogen reacted by elemental balance method	4.42%
Hydrogen reacted by gas balance method	4.40%

Figure 1

Effect of Pressure and Temperature on Yields and Desulfurization at
8 Minutes Residence Time
Runs DOE 385-400, Indiana V Coal

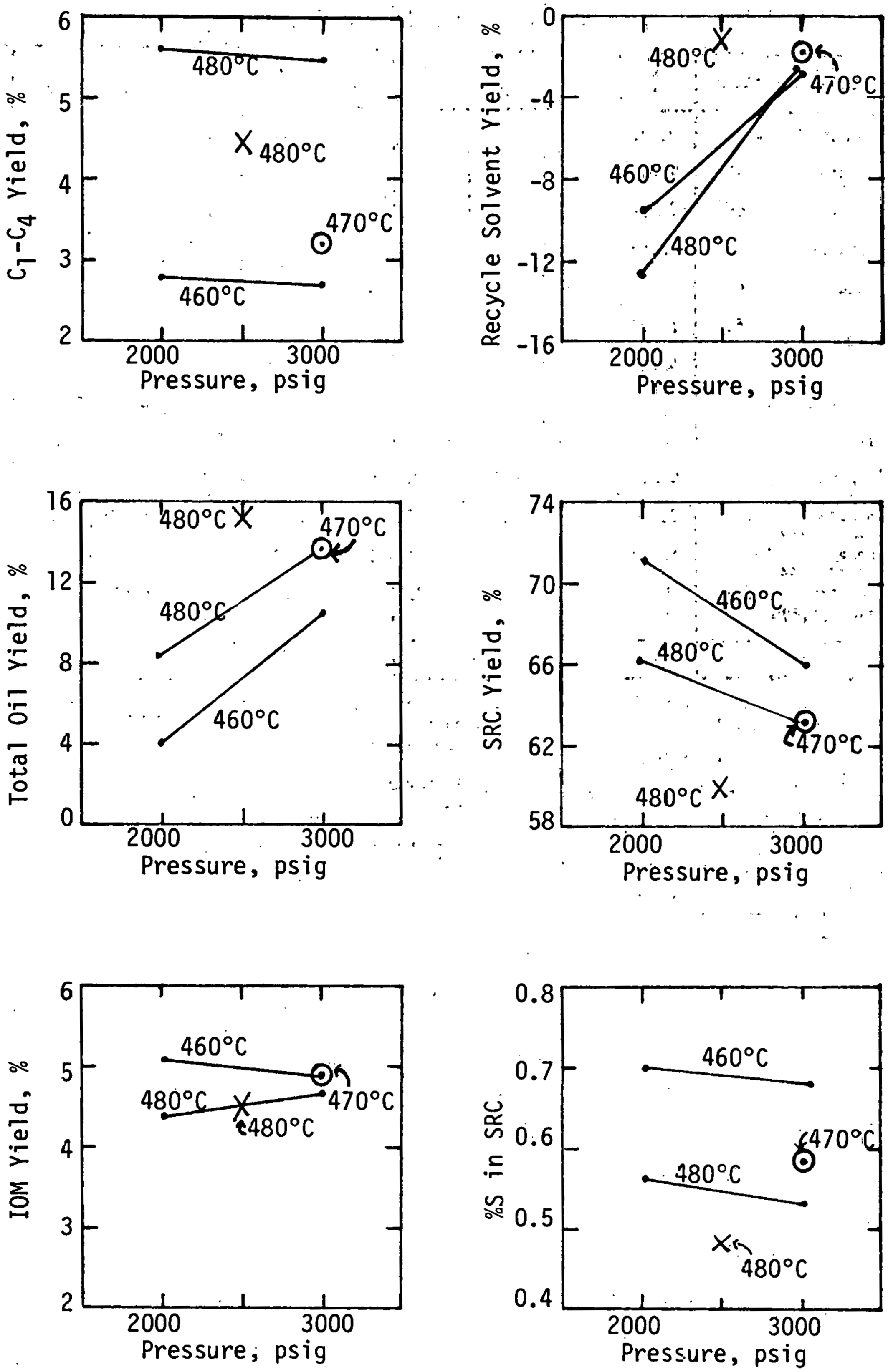


Figure 2

Effect of Pressure and Temperature on Yields and Desulfurization at
4 Minutes Residence Time
Runs DCE 385-400, Indiana V Coal

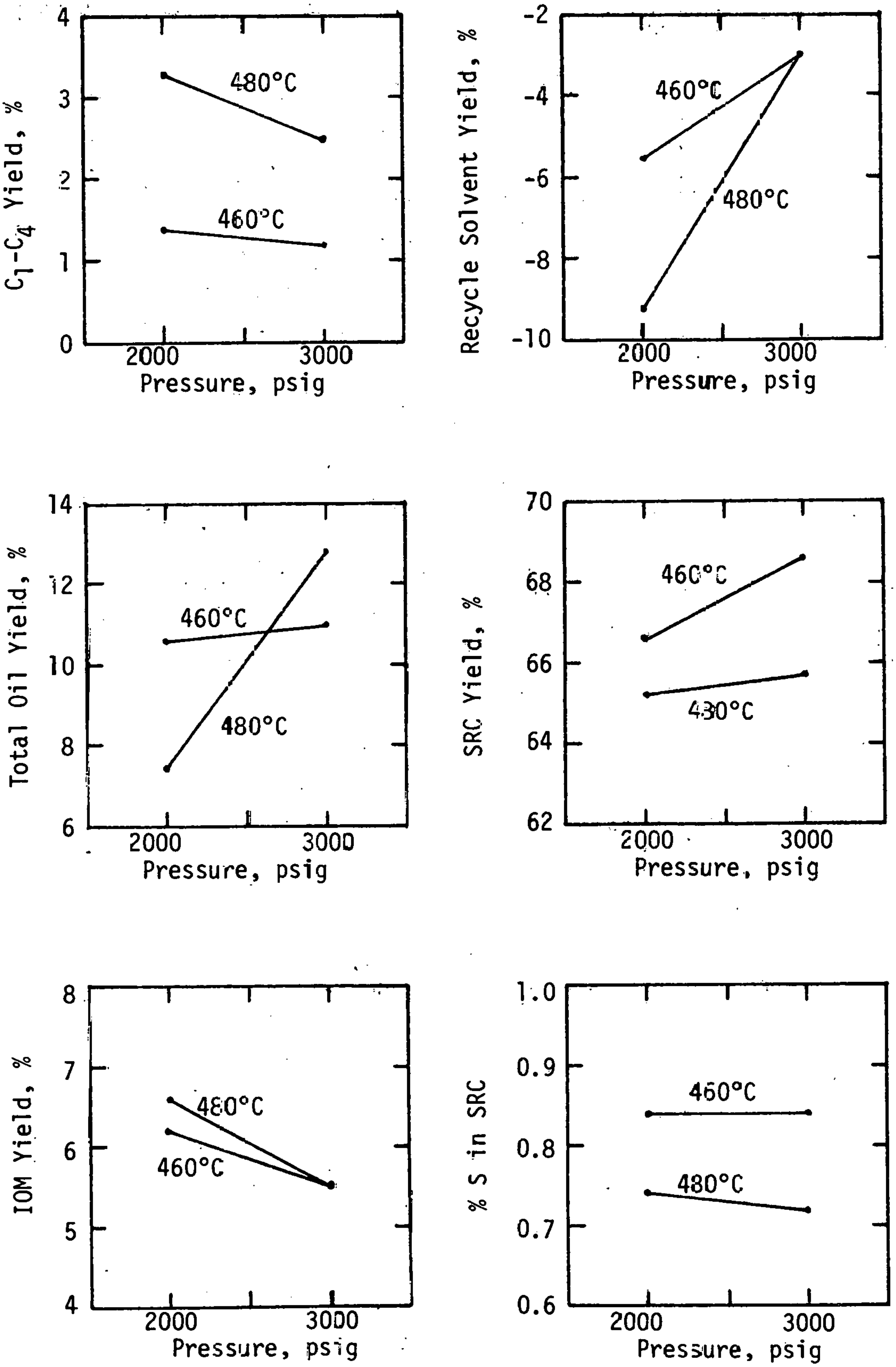


Figure 3
Unfiltered Coal Solution Viscosities
Runs DOE 381-384, Kentucky 9/14 Coal

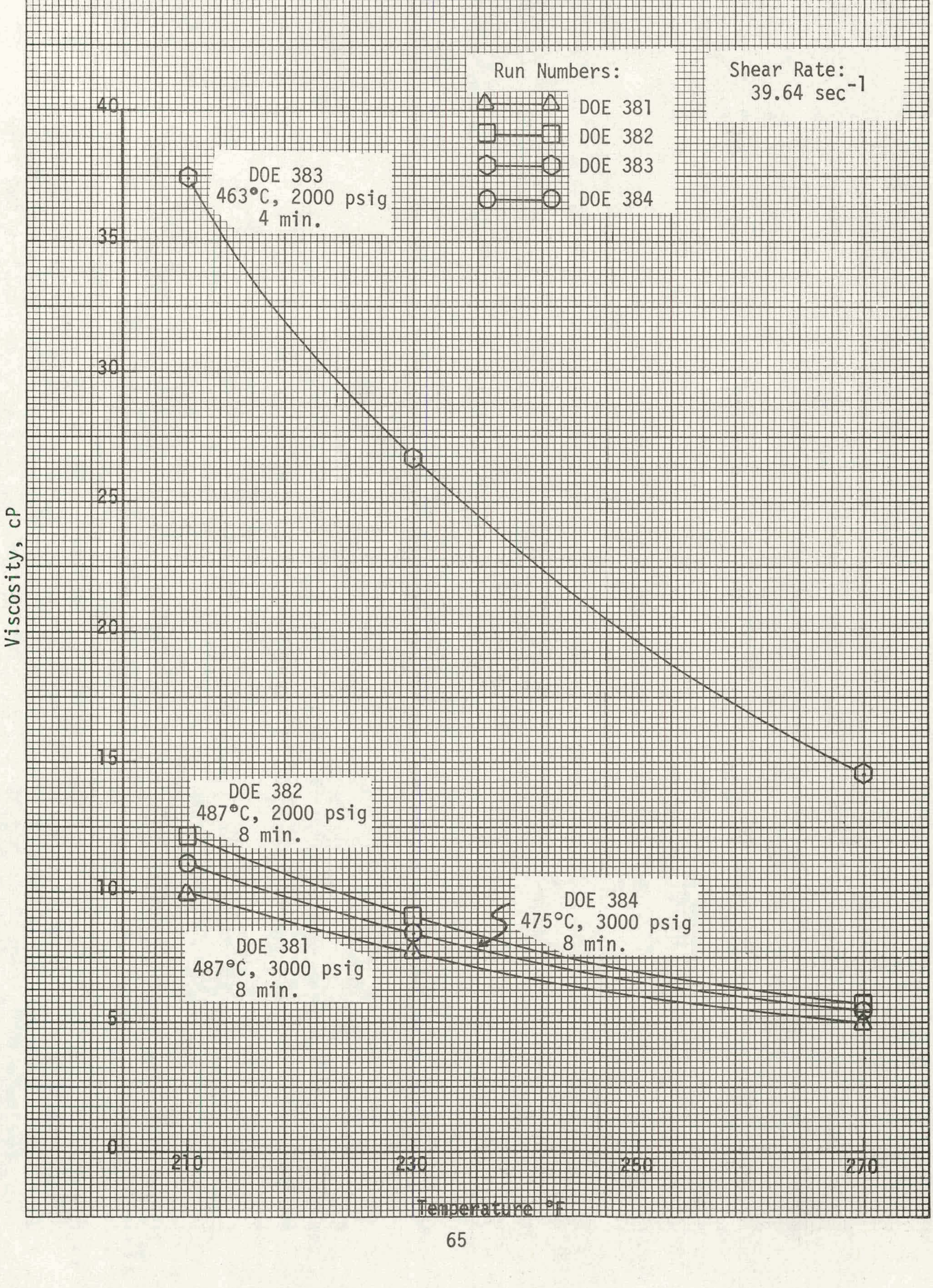
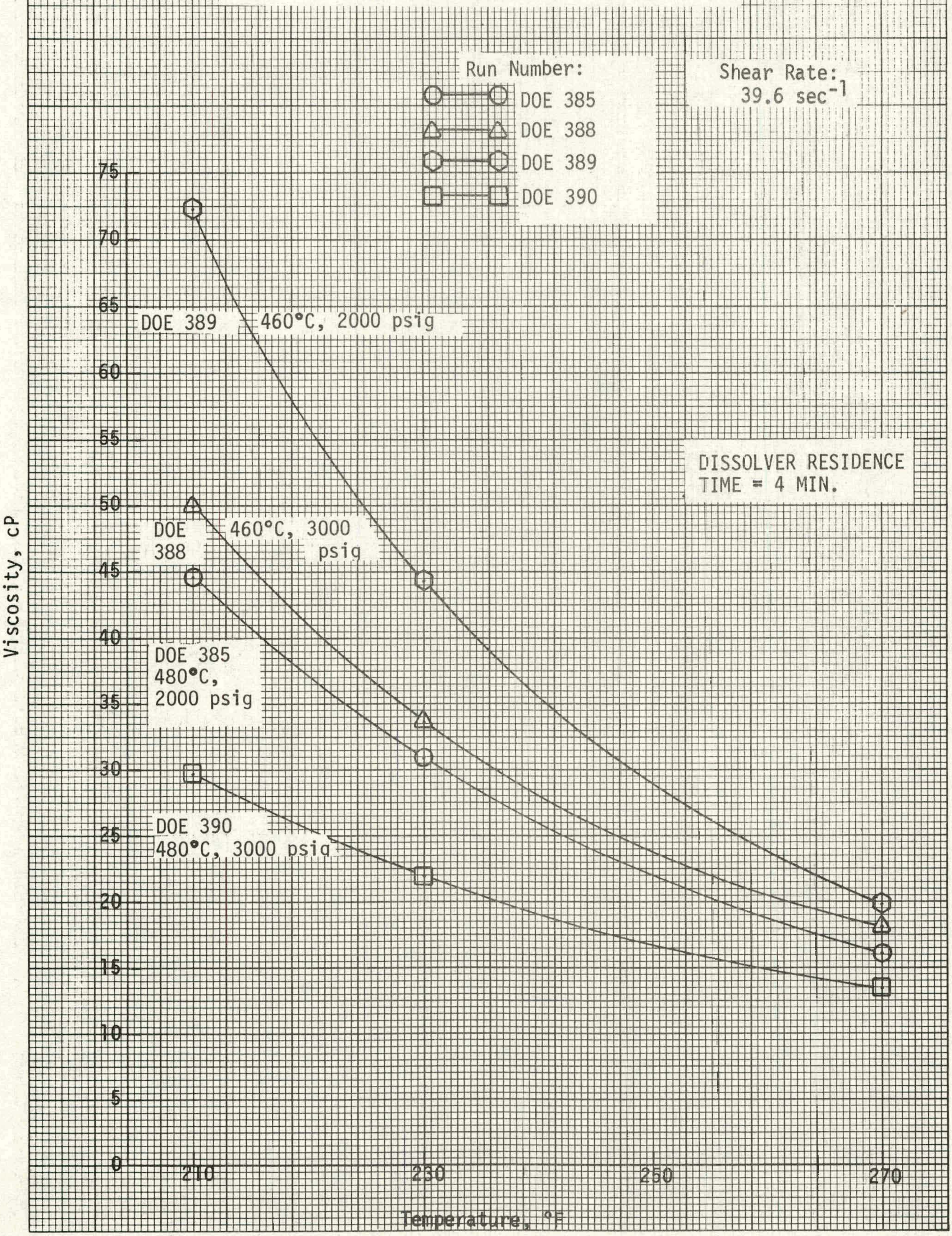


Figure 4

Unfiltered Coal Solution Viscosities
Runs DOE 385, 388, 389, 390
Indiana V Coal



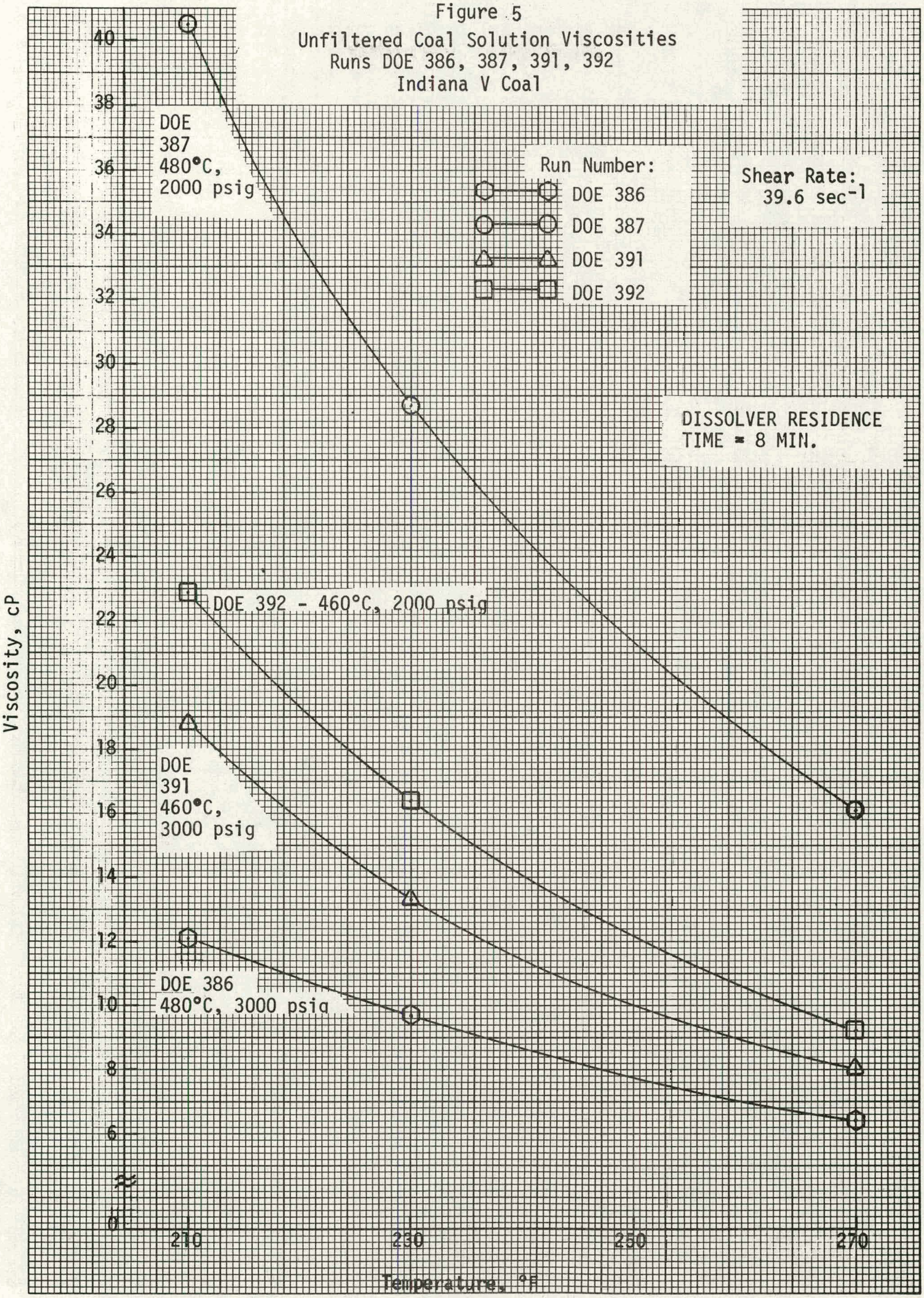
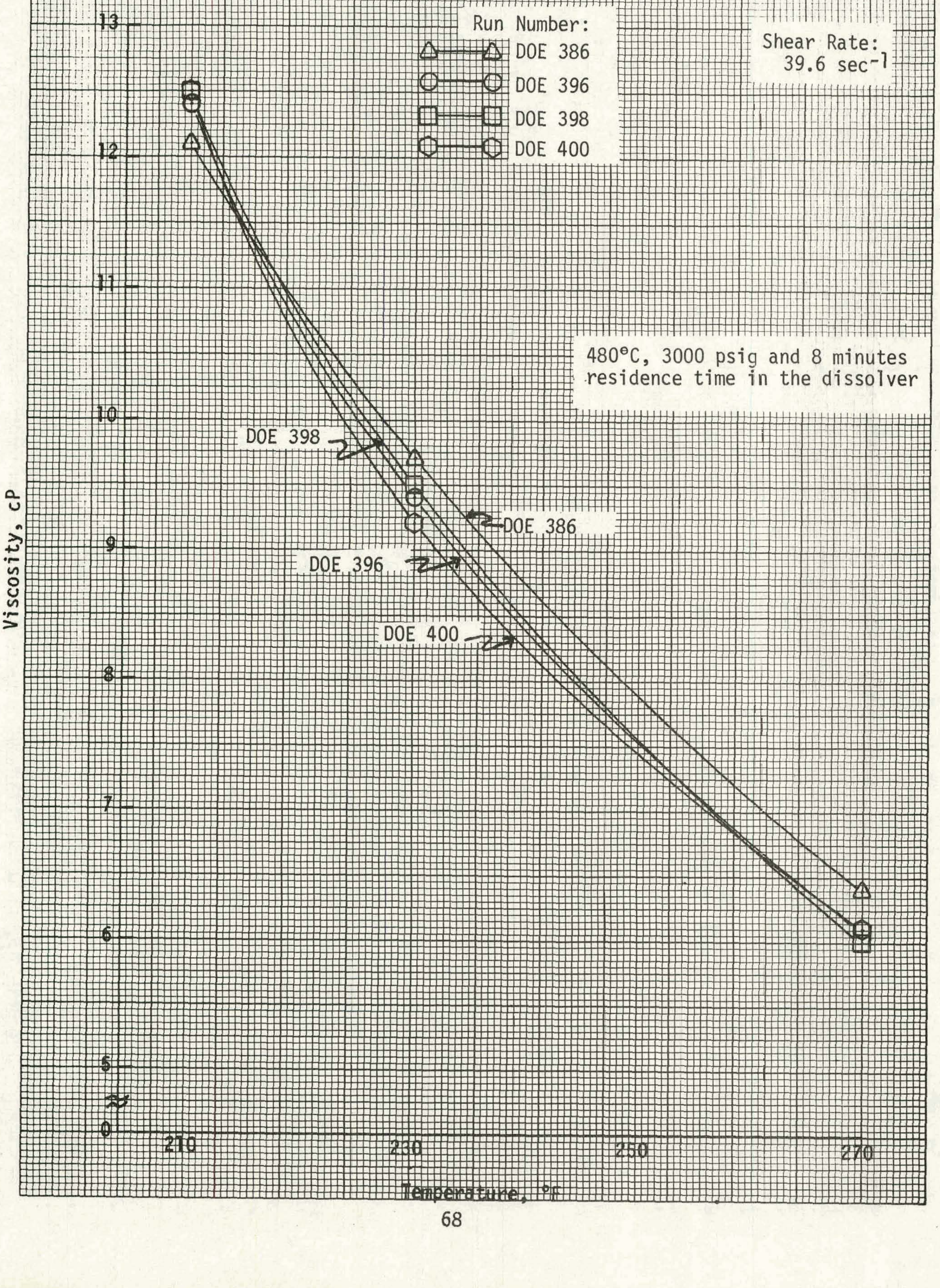


Figure 5
Unfiltered Coal Solution Viscosities
Runs DOE 386, 387, 391, 392
Indiana V Coal

Figure 6

Unfiltered Coal Solution Viscosities
Runs DOE 386, 396, 398, 400
Indiana V Coal



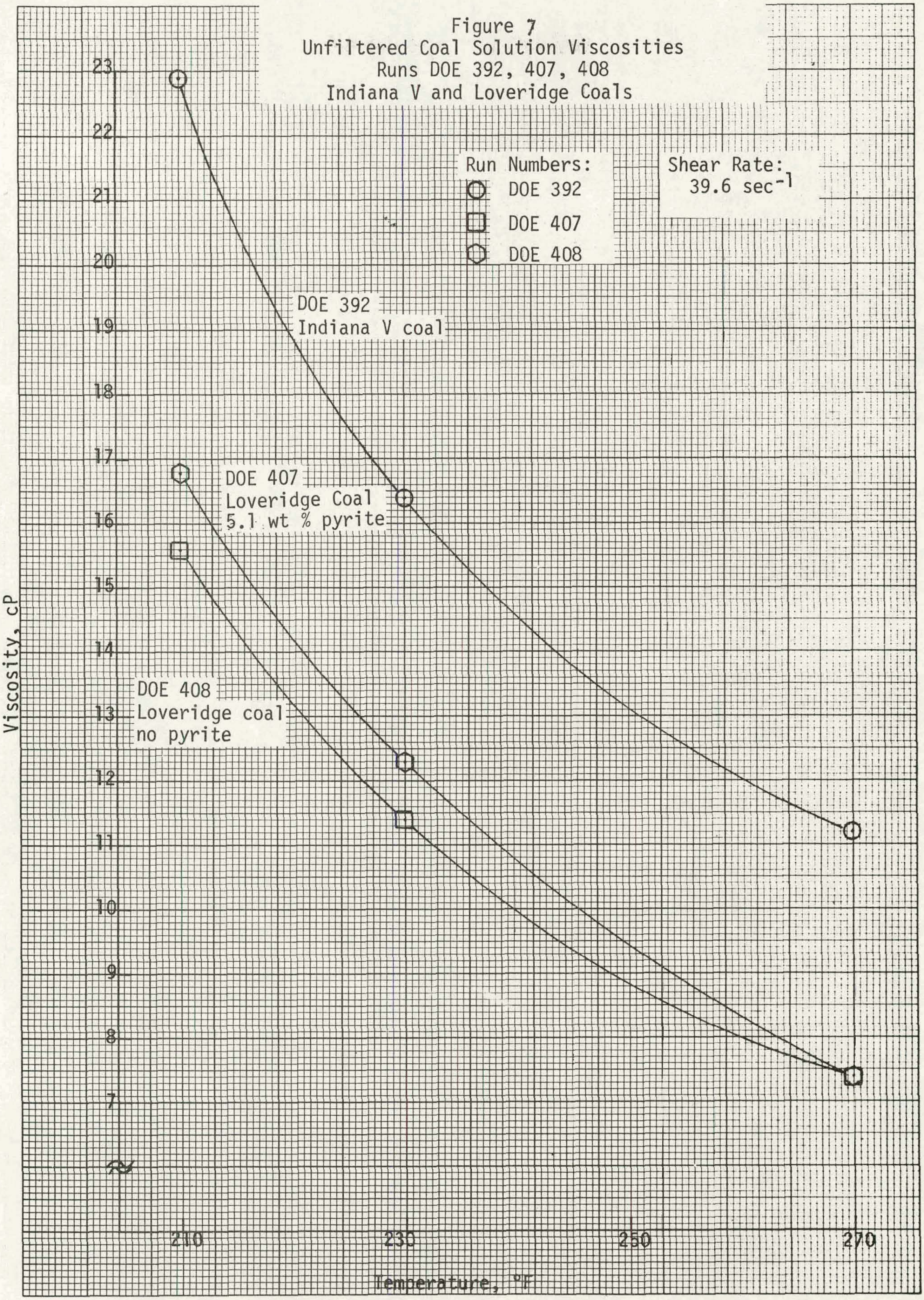
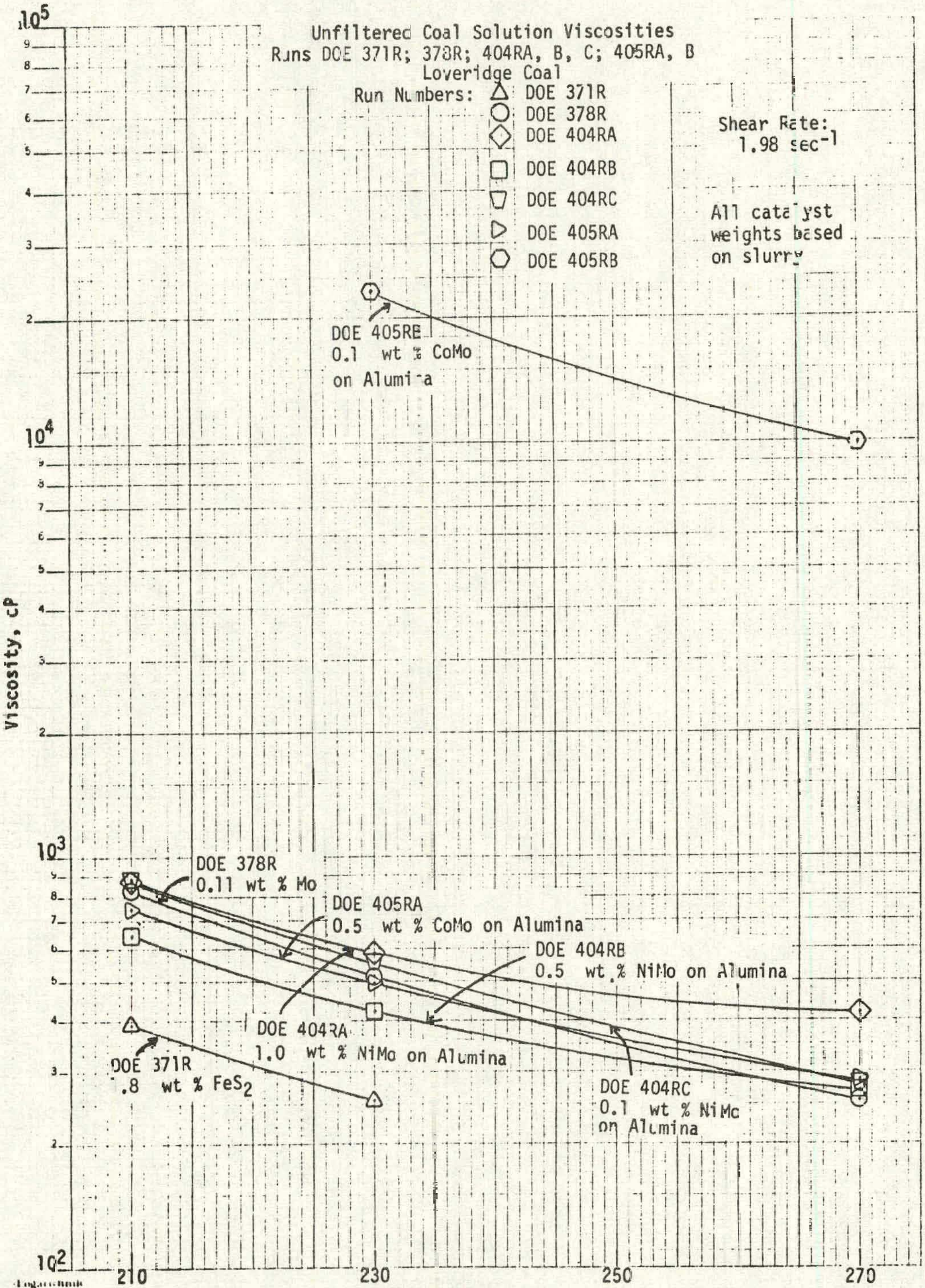


Figure 7
Unfiltered Coal Solution Viscosities
Runs DOE 392, 407, 408
Indiana V and Loveridge Coals

Figure 8



Unfiltered Coal Solution Viscosities
Runs DOE 371R; 378R; 404RA, B, C; 405RA, B
Loveridge Coal
Run Numbers: △ DOE 371R
○ DOE 378R
◇ DOE 404RA
□ DOE 404RB
▽ DOE 404RC
▷ DOE 405RA
⬡ DOE 405RB
Shear Rate: 1.98 sec^{-1}
All catalyst weights based on slurry
DOE 405RB 0.1 wt % CoMo on Alumina
DOE 378R 0.11 wt % Mo
DOE 405RA 0.5 wt % CoMo on Alumina
DOE 404RB 0.5 wt % NiMo on Alumina
DOE 404RA 1.0 wt % NiMo on Alumina
DOE 371R 1.8 wt % FeS$_2$
DOE 404RC 0.1 wt % NiMo on Alumina
Viscosity, cP
10^5
10^4
10^3
10^2
Temperature, °F
210
230
250
270
Logarithmic x 10 to the inch

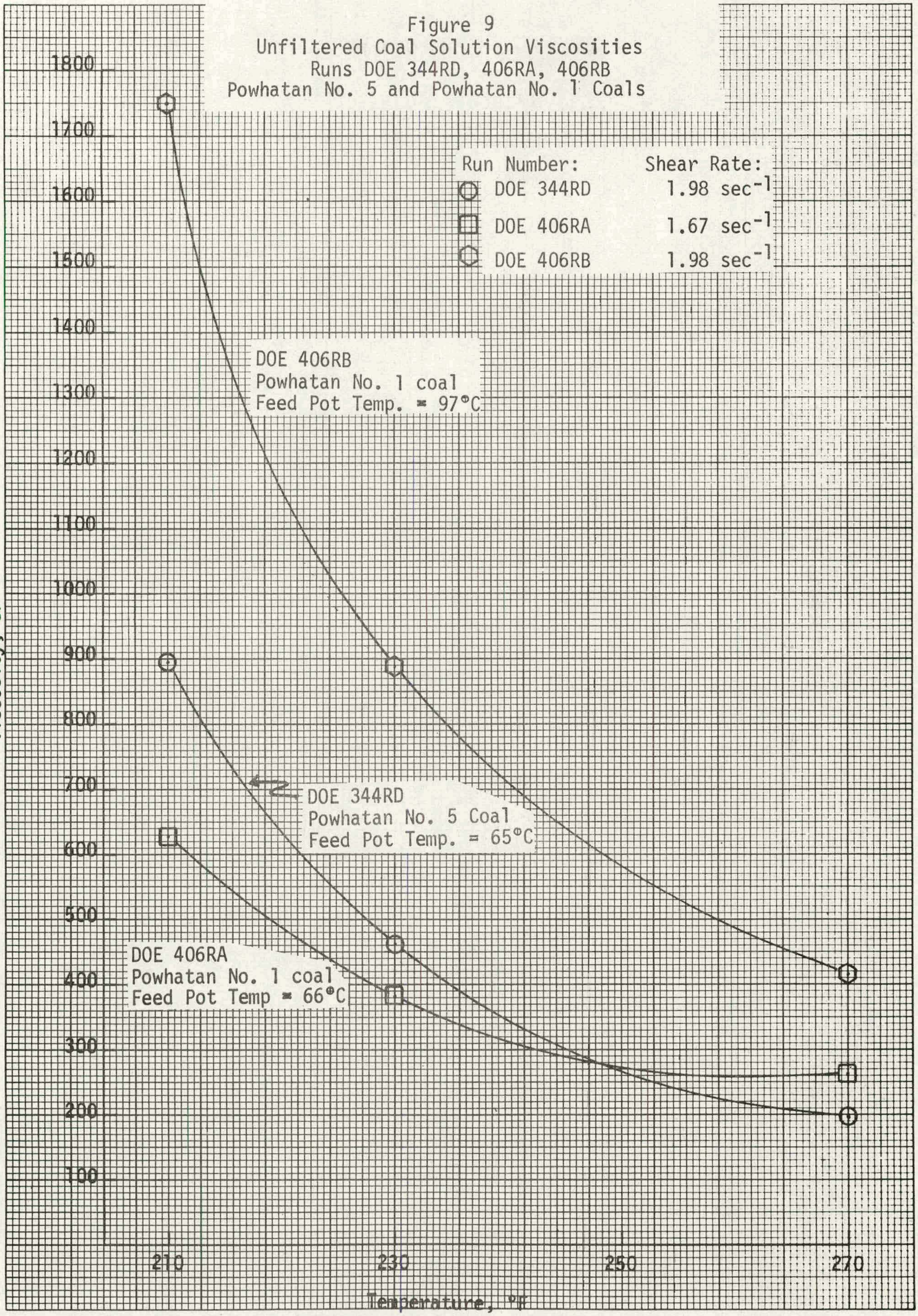
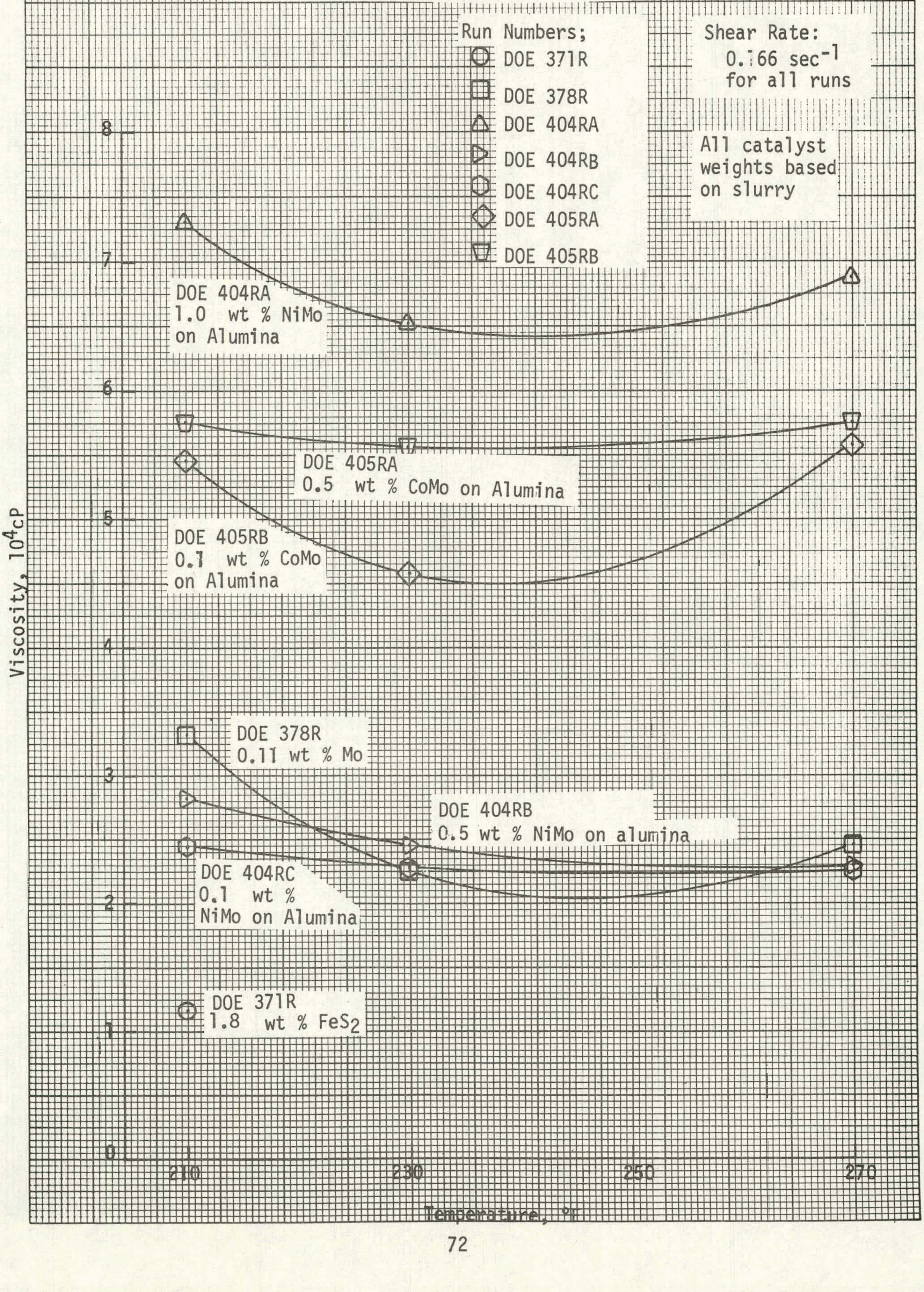


Figure 9
Unfiltered Coal Solution Viscosities
Runs DOE 344RD, 406RA, 406RB
Powhatan No. 5 and Powhatan No. 1 Coals

Figure 10
Feed Slurry Viscosities
Runs DOE 371R; 378R; 404RA, B, C; 405RA, B
Loveridge Coal



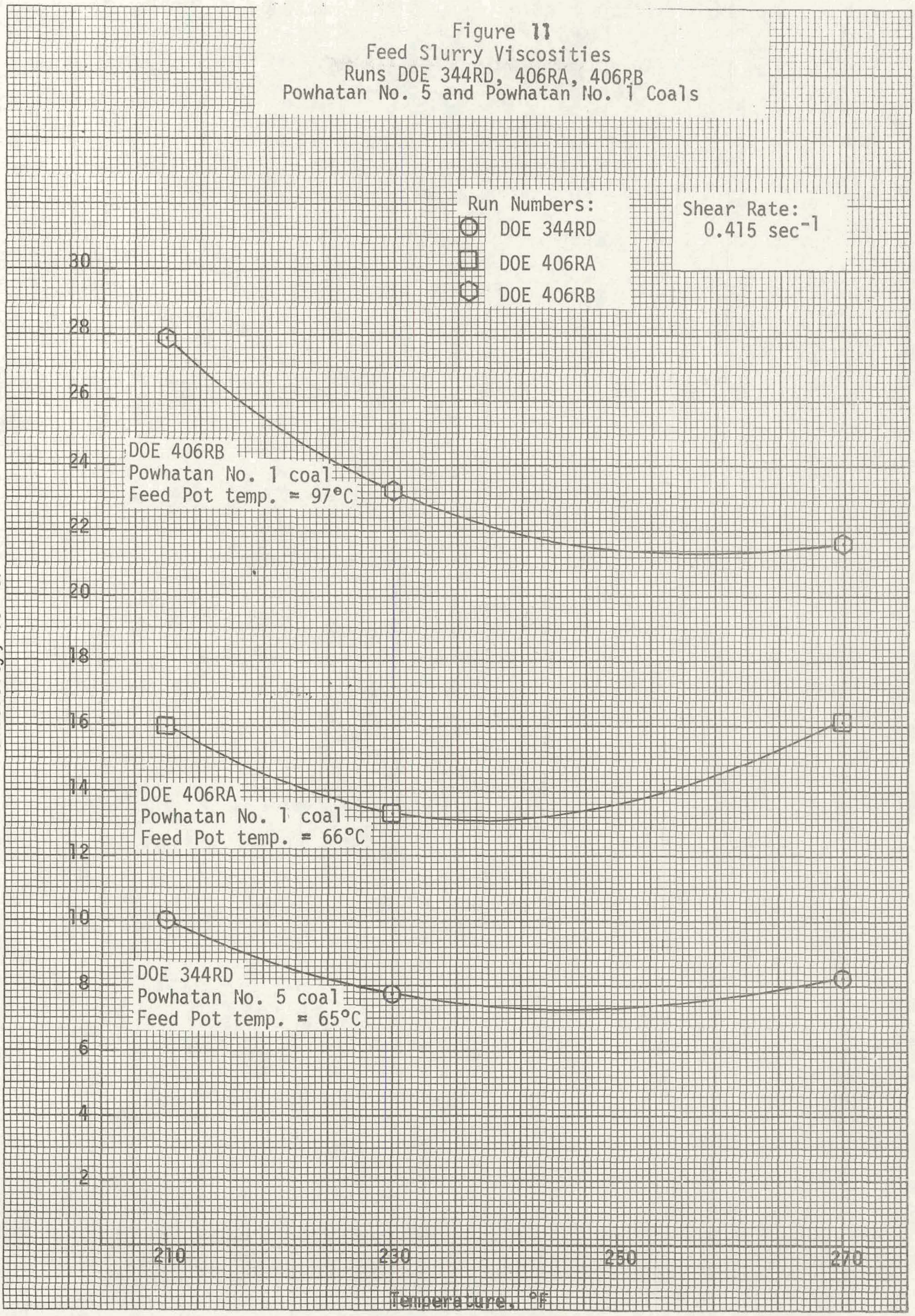
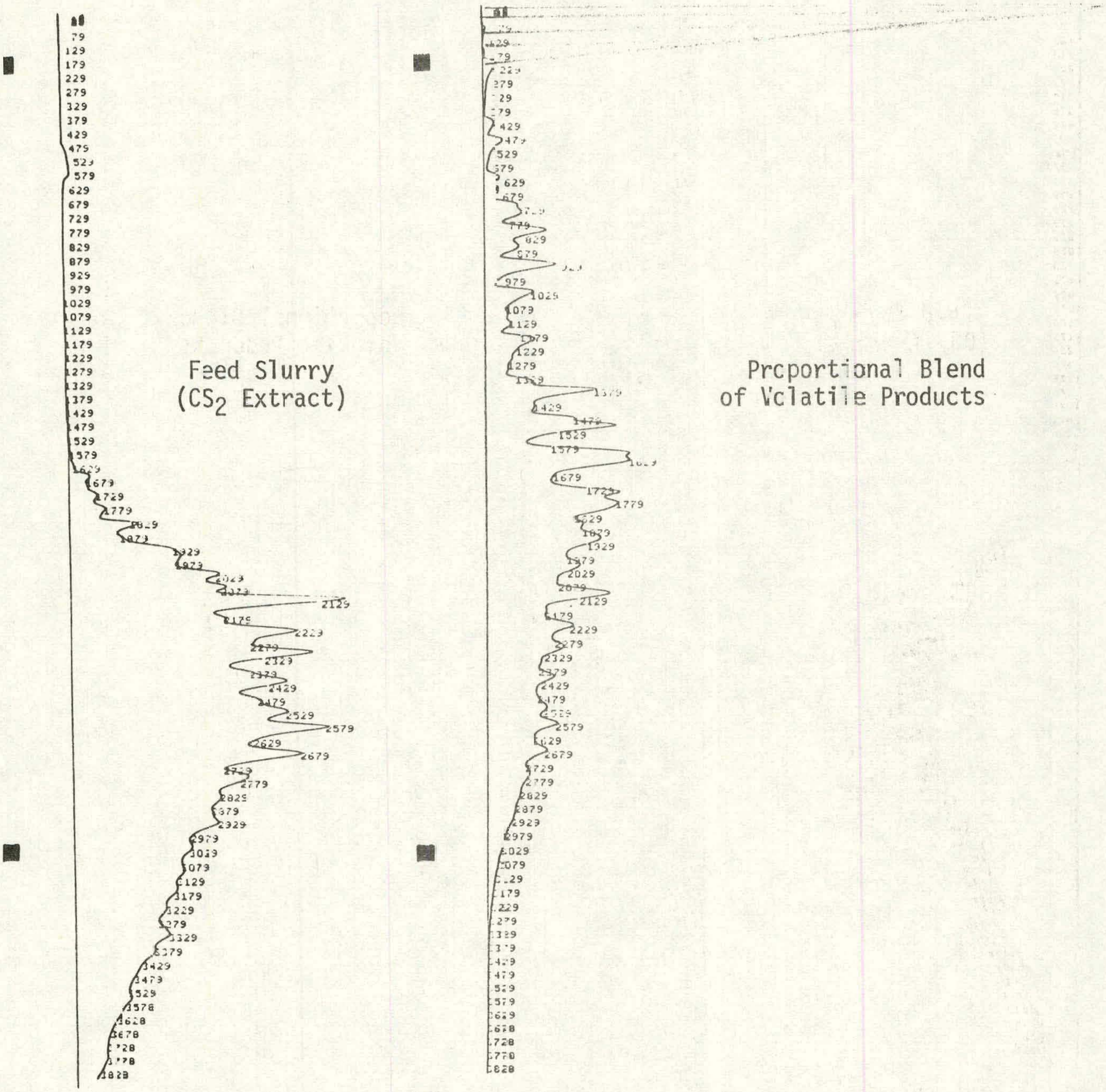


Figure 11
Feed Slurry Viscosities
Runs DOE 344RD, 406RA, 406RB
Powhatan No. 5 and Powhatan No. 1 Coals

Figure 12
Simulated Distillation of Feed Slurry and Product, Run DOE 405RA



Feed Slurry
(CS_2 Extract)

Proportional Blend
of Volatile Products

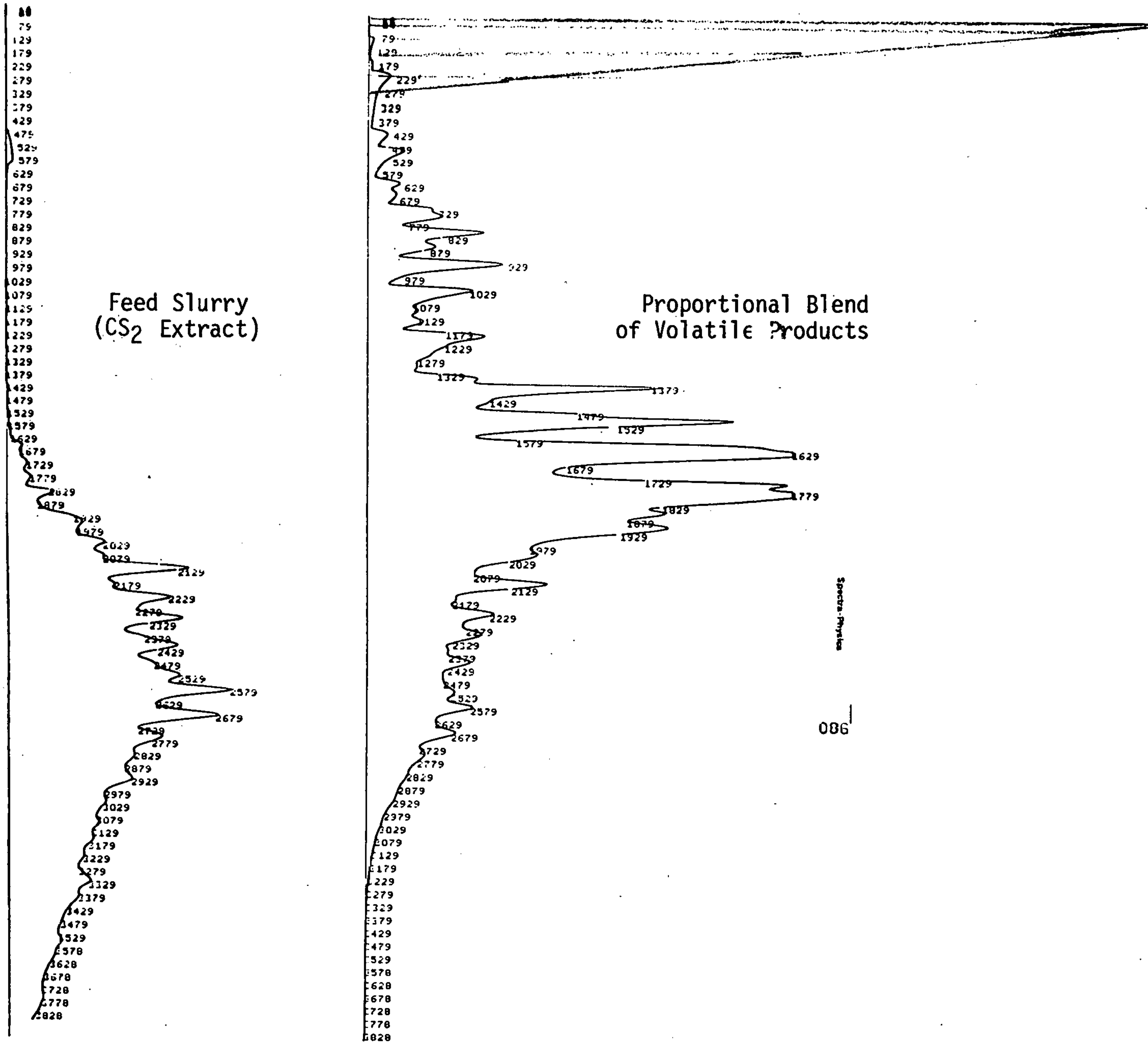
DOE 405 RA p. 70 CS2 ext.

```
SIMULATED DISTILLATION      1980 DEC 11 10 31 57

CHANNEL  1   RUN  12   FILE  1   METHOD  8

VIAL  20

INDEX   9   SAMPLE   7

  % OFF                 TEMP

    .5                   63.97
   5                    254.2
  10                    270.2
  15                    285.5
  20                    293.5
  25                    301.6
  30                    317.5
  35                    325.6
  40                    333.8
  45                    342.1
  50                    350.2
  55                    364.9
  60                    372.8
  65                    381
  70                    389.3
  75                    404.9
  80                    420.0
  85                    437.2
  90                    461.3
  95                    485.1
  99.5                  530.8
```

DOE 405 RA Prop Blend as made

```
SIMULATED DISTILLATION      1980 DEC 04 08 12 57

CHANNEL  1   RUN 216   FILE  1   METHOD  8

VIAL  26

INDEX 215   SAMPLE 214

  % OFF                 TEMP

    .5                    9.484
   5                     79.05
  10                    108.4
  15                    146.2
  20                    176.7
  25                    192.5
  30                    207
  35                    214.3
  40                    230
  45                    238.1
  50                    246.3
  55                    254.6
  60                    270.8
  65                    285.9
  70                    301.6
  75                    318.1
  80                    334.2
  85                    357.7
  90                    373.6
  95                    405
  99.5                  470
```

Figure 13
Simulated Distillation of Feed Slurry and Product, Run DOE 405RB



DOE 405RB P144-145 CS_2 Ext.

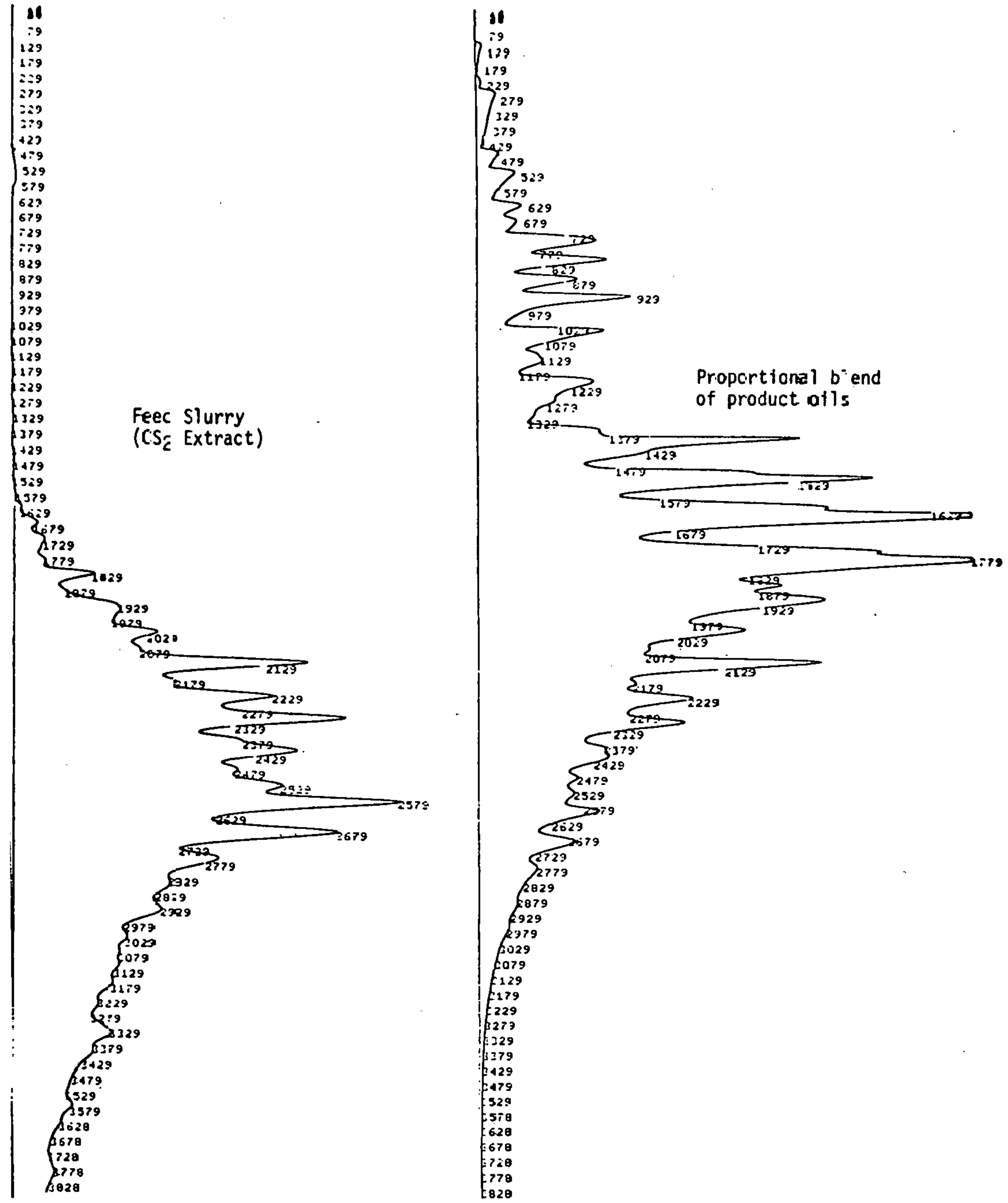
```
SIMULATED DISTILLATION       1980 DEC 11 11:27:09
CHANNEL   1    RUN  13    FILE   1    METHOD  8
VIAL  21
INDEX  10    SAMPLE   8
   % OFF                TEMP

     .5                  52.45
    5                   255.7
   10                   274.1
   15                   285.6
   20                   297.3
   25                   308.3
   30                   318.1
   35                   328.9
   40                   338:6
   45                   348.2
   50                   356.3
   55                   364.2
   60                   372.3
   65                   381.3
   70                   391.7
   75                   404.1
   80                   418
   85                   435.7
   90                   457.6
   95                   483.6
   99.8                 527.6
```

DOE 405RB P135-144 Prop. Blend

```
SIMULATED DISTILLATION       1980 DEC 11 12:22:20
CHANNEL   1    RUN  14    FILE   1    METHOD  8
VIAL  22
INDEX  11    SAMPLE   9
   % OFF                TEMP

     .5                   9.762
    5                    85:88
   10                   122.7
   15                   153.6
   20                   176.5
   25                   192
   30                   199.8
   35                   207.1
   40                   214.1
   45                   222.2
   50                   230
   55                   237.8
   60                   245.7
   65                   253.8
   70                   262.3
   75                   278
   80                   294
   85                   318.4
   90                   349.6
   95                   380.5
   99.5                 427.1
```

Figure 14

Simulated Distillation of Feed Slurry and Product, Run DOE 406RB



DOE 406 RB P-116 Feed Slurry 22.7% in CS$_2$

```
SIMULATED DISTILLATION      1980 DEC 18 12:09.04
CHANNEL   1    RUN  66    FILE   1    METHOD  8
VIAL  22
INDEX  62    SAMPLE  61
   % OFF                TEMP
      .5                192.5
     5                  254.3
    10                  274.8
    15                  289.6
    20                  300
    25                  306.8
    30                  317.2
    35                  325.1
    40                  333.3
    45                  341.5
    50                  345.6
    55                  357.3
    60                  364.9
    65                  372.7
    70                  380.7
    75                  389.2
    80                  404.6
    85                  420.8
    90                  444.9
    95                  478.7
    99.5                522.2
```

DOE 406 P116-123 Prop. Blend

```
SIMULATED DISTILLATION      1981 JAN 19 12:21 18
CHANNEL   1    RUN 189    FILE   1    METHOD  8
VIAL   9
INDEX 184    SAMPLE 184
   % OFF                TEMP
      .5                 14.2
     5                   85.67
    10                  116.6
    15                  155.1
    20                  178.2
    25                  193.2
    30                  208.2
    35                  215.9
    40                  223.8
    45                  232
    50                  239.8
    55                  247.6
    60                  255.8
    65                  264.3
    70                  280
    75                  288.4
    80                  303.6
    85                  326.9
    90                  350.5
    95                  382.5
    99.5                455.1
```

APPENDIX A

SETTLING EXPERIMENT

Introduction

Gravity settling can be used to separate a mixture of particles that have different densities or diameters. This experiment was undertaken to see what, if any, separation could be effected between the iron compounds and the rest of the ash material in unfiltered coal solution (UFCS) from the SRC process. If iron compounds in the UFCS could be concentrated, the level of catalytically active materials in the feed slurry could be increased while maintaining total solids at a constant level. Calculations using Stokes Law indicate that for 1 micron sized particles there will be no significant settling. However, since this equation does not take into account the effects of flocculation, particle size distribution, particle concentration or non-Newtonian liquids, it was felt that it would still be worthwhile to try an experiment using a gravity settler.

Theory (Stokes Law)

When a particle falls through a fluid, a drag force is generated. This force is given by

$$F_d = \frac{C A_p \rho u^2}{2 g_c}$$

C = drag coefficient
A_p = projected area of particle in direction of motion (L^2)
ρ = density of fluid (mass/L^3)
u = relative velocity (L/sec)
g_c = dimensional constant related to local gravitational acceleration $\frac{\text{mass}\cdot L}{\text{force}\cdot\text{sec}^2}$

The drag force will increase as the particle accelerates until the drag force plus the bouyant force equal the local gravitational force. At this point the velocity can be calculated as

$$u = \left[\frac{2 g\, m_p\, (\rho_p - \rho)}{\rho \rho_p\, A_p\, C}\right]^{1/2}$$

g = local gravitational constant
m_p = mass of particle
ρ_p = density of particle

Stokes Law is formulated by making certain assumptions about the drag coefficient. Because all of the assumptions in the law are not fulfilled by the unfiltered coal solution (UFCS), experiments need to be made to determine the applicability of gravity settling for concentrating the iron content of the UFCS.

Experimental

The experimental apparatus is shown in Figure A-1. It consists of a feed pot, mixer, two pumps, settling vessel, and siphon break. The feed pot and mixer were used to ensure that the unfiltered coal solution

(UFCS) was uniformly mixed. Pump A pumped UFCS into the settling vessel while pump B removed it at a lower rate. A siphon break was included in line B to prevent the siphoning of UFCS out of the settling vessel.

Experimental Procedure

UFCS from a standard SRC II run with Kentucky 9/14 coal was added to the feed pot and kept at a temperature of approximately 115°C. Once the settler was full, pumps A and B were calibrated to flowrates of 1043 g/hr and 761 g/hr, respectively, so that the percentage of the UFCS product normally recycled in the SRC II process was pumped out the bottom. To start the experiment, samples of UFCS were taken from the feed pot and line B. Additional samples were taken at various times so that ash and iron concentrations could be determined (refer to Table A-1). The temperatures of all the lines and the settling vessel were kept at 125°C to enhance settling.

During the startup, line B, between pump B and the feed pot, plugged and the experiment was shut down. The plug was believed to be caused by a hot spot from the heat tracing. Line B was changed from 1/4" SS tubing to 3/8" and the heat tracing on all the lines was changed to avoid further hot spots. The decrease in ash content in the feed pot indicates that there was deposition of ash material in the settling vessel and feed lines.

After the experiment was completed, the unit was drained of UFCS and flush oil added. The flush oil was circulated until the next day. The feed pot was then taken out of the system and drained. Upon inspection of the feed pot line, it was noted that the line was about half filled with residue that had settled out of the UFCS.

During the run, the flowrate through line B changed from 761 g/hr to 286 g/hr (see Table A-2) because the pump stroke length was not locked into place. This shift in the flowrates seemed to help the ash settling but will make quantitative interpretation of the results impossible.

Conclusions

The decrease in the bottoms flowrate during the run confounds interpretation of the data. However, settling of ash material did occur in this experiment. There is about a 3 percent difference between the ash content of the overhead and the bottoms streams and a similar difference in the iron content of the ash (Table A-1). However, the surprising result is that the percentage of iron compounds in the ash is greater in the overhead stream than in the bottoms stream. This result, if verified by further work, could mean a reduction of the amount of inert materials that need to be handled to obtain a given concentration of iron.

From the absolute levels of iron in the bottoms (3.98%) and overhead (3.81%) streams, it is apparent that very little settling of the iron compounds takes place, whereas the other compounds had a significant amount of settling. The iron compounds have densities significantly higher than the clays which indicates that the particle size distribution must be different for these materials. One possible explanation is that as the pyrites go through the reactor the particle size is broken down to such a degree that thermal and convective agitation keep the iron compounds in suspension in the settler.

Table A-1

Ash and Iron Analyses of Gravity Settler Streams

Date and Time of Sample		Stream					
		Bottoms		Overhead		Feed Pot*	
		% Ash	% Fe in Ash	% Ash	% Fe in Ash	% Ash	% Fe in Ash
11/10/80	2130	20.5	21.1			19.5	20.7
	2300	20.4	19.7	19.9	20.5		
11/11/80	0400	20.3	19.6	15.6	20.9		
		20.2	19.5	15.6	20.9		
	1000	20.7	18.9	17.0	23.1		
		20.7	18.7	16.9	22.7		
	1600	20.5	19.5	17.4	22.7		
		20.3	19.2	17.4	22.2		
	2400	20.3	19.4	17.5	22.3		
		20.4	19.4	17.6	22.3		
11/12/80	0600	20.0	19.6	17.8	22.0		
		20.0	19.7	17.2	22.3		
	1000			18.3	21.9	18.9	20.9
Average		20.4	19.5	17.3	22.0	--	--
Absolute Std. Dev.		0.23	0.53	1.13	0.80	--	--

* Average for 2 or 3 determinations.

Table A-2

Residence Time in the Settler†

Stream	Start	Finish
Overhead	11.4	4.3
Bottoms	1.3	3.4

† In hours.

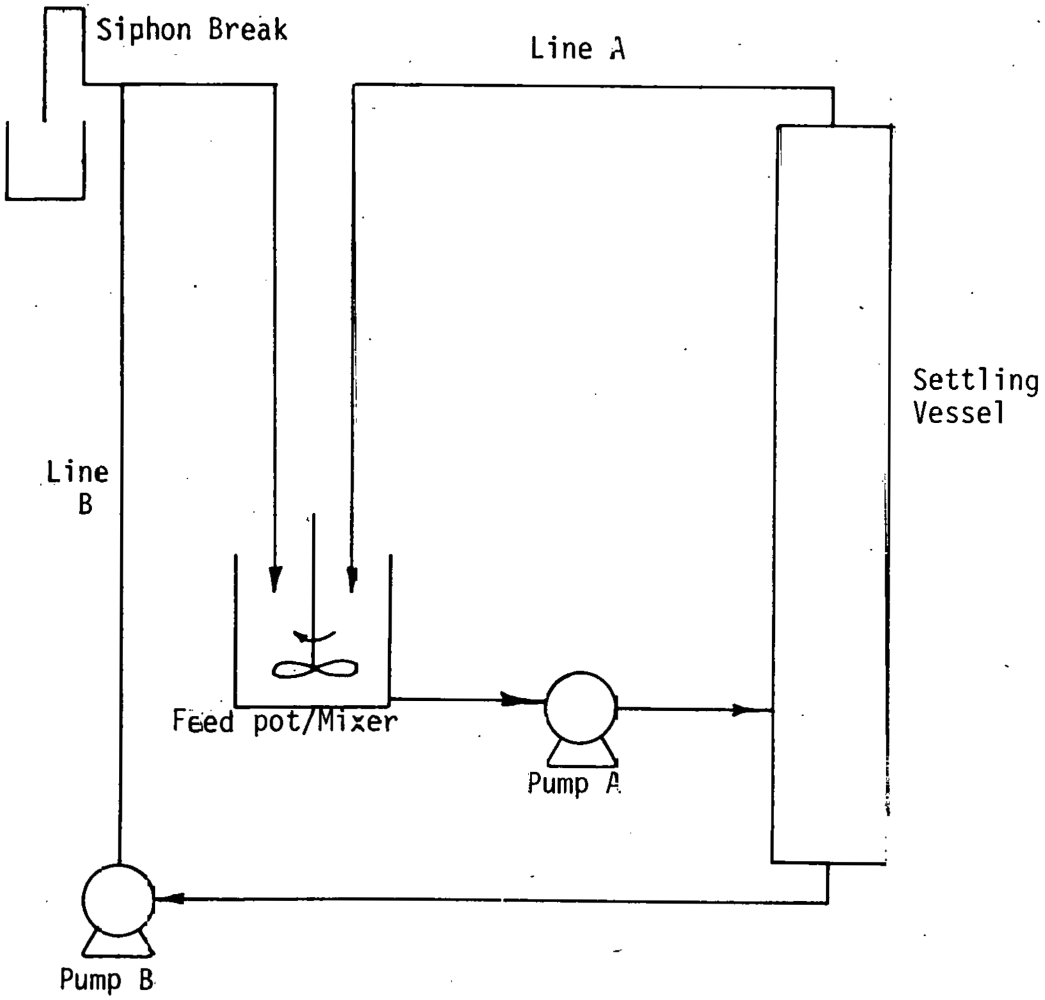


Figure A-1

Experimental Apparatus
for Settling Experiment

APPENDIX B

REPORT ON RUPTURE DISC FAILURE

Run DOE 406R
Powhatan No. 1 Coal

Summary

The rupture disc failed due to corrosion and pitting resulting in reduced wall thickness which lowered the pressure rating. This is a common problem with rupture discs which have been in service for extended periods of time.

Discussion

A rupture disc (Figure B-1) was submitted for failure analysis. The disc failed at normal operating conditions. The line was carrying dissolver effluent at 735°F and 1800 psig. The disc was located in a dead leg of the transfer line between the dissolver and the high temperature, high pressure separator. The dead leg temperature was estimated to be 300°F. The rupture disc had been in service for three months, with an average of two shutdowns per month. The disc was identified as Inconel 600 and had a pressure rating of 5,910 psi at 72°F.

The disc failed by rupturing (Figure B-2) which is characteristic of over-pressuring. Examination of the topography of the surface (Figure B-3) revealed pitting and metal loss.

No metallurgical abnormalities were observed in the structure of the disc material.

Conclusion

Corrosion and pitting caused the failure by reducing the wall thickness which lowered the pressure rating. Pitting was the major cause of the failure with general corrosion a minor factor. Sulfidation corrosion occurs in alloys which contain a high amount of nickel and are present in sulfur containing atmospheres around 350°F. The sulfur can be present as H_2S, organic sulfur compounds and free sulfur. Pitting is mainly caused by the buildup of chlorides on the metal surface. Premature failure is a common problem with rupture discs due to their thinned condition and susceptibility to corrosion in this environment.

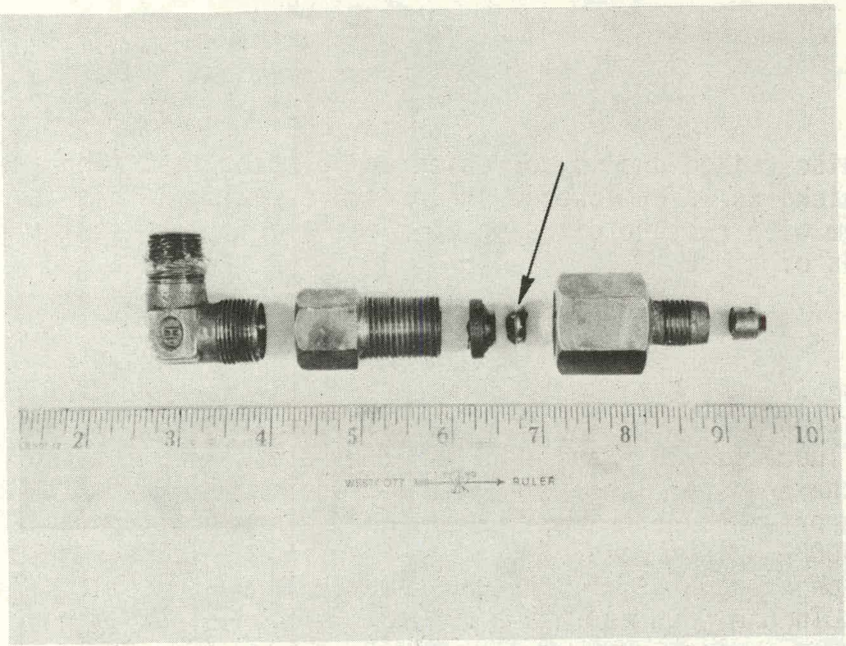
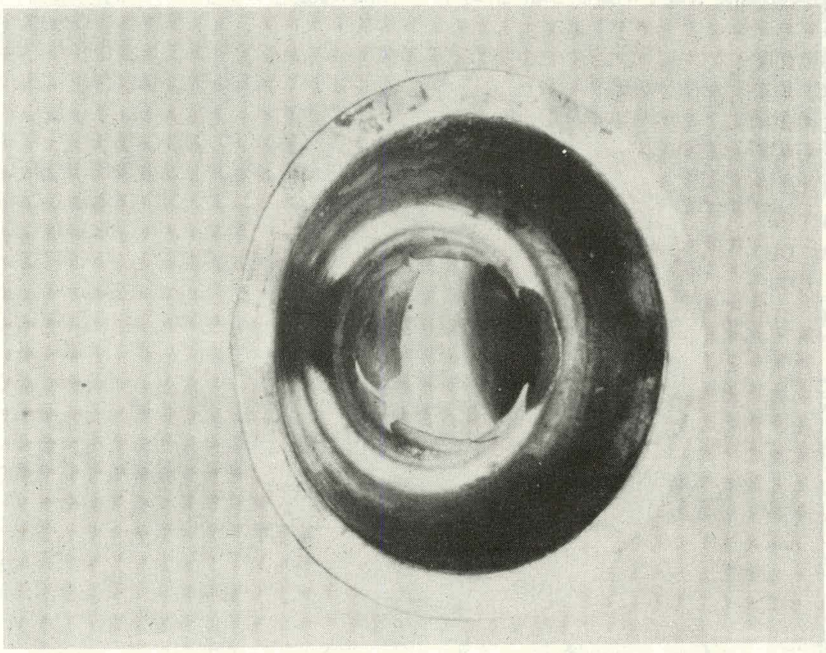


Figure B-1

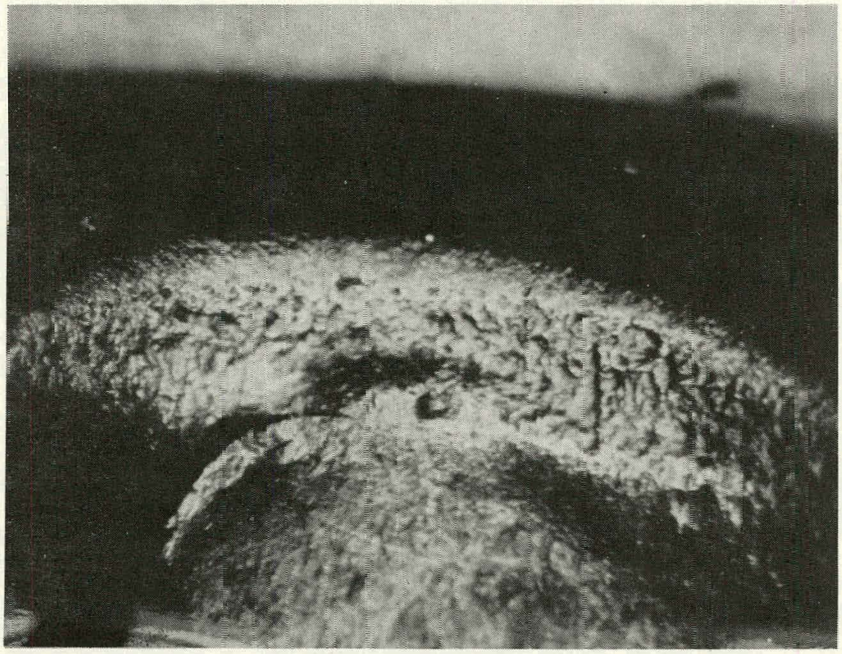
Rupture disc assembly. The failed Disc is shown by the arrow.



6X

Figure B-2

Closeup of failed rupture disc. Appearance is characteristic of over-pressuring.



40X

Figure B-3

Topography of the rupture disc surface. The topography shows pitting and metal loss. Note the disc blowout downward in the picture.

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