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RECYCLE SLURRY OIL CHARACTERIZATION

Technical Report for October 1, 1982–March 31, 1983

By
R. A. Winschel
F. P. Burke

February 1984

Work Performed Under Contract No. AC22-80PC30027

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Section 1

INTRODUCTION

BACKGROUND

This is the technical report for the period October, 1982 through March, 1983, under the subject contract reporting the results of analyses of process and product oils in direct liquefaction processing. In this contract, the majority of samples have come from the integrated two-stage liquefaction (ITSL) process development unit (PDU) operated by the Lummus Co. at New Brunswick, New Jersey. The Lummus unit (Figure 1) consists of a short contact time (SCT) coal liquefaction, followed by Lummus antisolvent deashing (ASDA) of the short residence time product. The deashed coal liquids are then upgraded in a Lummus-Cities Service LC-Fining unit. The Lummus work is being conducted under DOE contract DE-AC22-79ET-14804. DOE has also provided us with samples from other sources, notably the H-Coal PDU in Trenton, New Jersey, the H-Coal pilot plant in Catlettsburg, Kentucky, and the Solvent Refined Coal (SRC) pilot plant in Wilsonville, Alabama.

Our intention in this work is not simply to provide analytical data, but to use recycle slurry and process oil characteristics to evaluate the relationship between coal liquid properties and process performance.

In making interpretations of process performance based on product and recycle oil characteristics, we are somewhat constrained by the operational characteristics of the liquefaction unit in question. The pertinent example is the Lummus ITSL PDU, in which sizing of the three major components (SCT, ASDA and LC-Finer) makes it impossible to consistently follow any conceptual piece of coal fed through the unit from feed to product and recycle. This difficulty is not unique to Lummus, and is largely dictated by their scale of operations and the different feed rates of the three component units. It does mean, however, that conclusions regarding integrated unit operation, such as the effect of changing SCT operating conditions on ASDA or LC-Finer performance, are necessarily imprecise. It is possible to treat each unit

as a quasi-independent entity and establish feed/product/operating condition relationships for each individually. This ultimately allows some synthesis of the whole, and will be seen to be our prevalent mode of interpretation in this research work.

DESCRIPTION OF PROCESS AND SAMPLES

The Integrated Two-Stage Liquefaction (ITSL) process development unit (PDU) is operated by the Lummus Co. in New Brunswick, NJ. (Figure 1). The short contact time (SCT) unit is the usual first stage of the Lummus ITSL process. One recent first-stage run was made using a back-mixed soaker reactor in addition to the SCT reactor. Pasting solvents (PS), consisting of distillate, soluble and insoluble resid, and ash are used to slurry coal (30-36% MF) which is then liquefied in a tubular reactor at residence times of 2-5 min, pressures of 2000-2400 psig and final temperatures of 830-865°F. The total liquid product (TLP) consists of ca. 97% heavy oil (500°F⁺) and ca. 3% light oil (500°F⁻). We received twenty-one samples of pasting solvent (PS) and twenty-three samples of heavy oil product (HO) from short contact time Runs 2SCT12 to 2SCT18 (March, 1982, to February, 1983). All runs (except Run 2SCT14) were made in the fully integrated mode, feeding Burning Star Illinois 6 coal. Run conditions during the sampling periods, as supplied by Lummus, are given in Table 1. Coal space rate varied by over a factor of two, from 82 to 182 lbs/hr/ft³ but because of the "slug" flow dynamics in the coil, residence times were little affected. Since gas hold-up is on the order of 60-80% of the coil volume, residence times were changed by varying the gas rate from 52 to 165 SCFH. MF coal concentration ranged from 30 to 36% for the coal runs. Preheater outlet temperature was ca. 840°F for all the SCT coal runs and 800-820°F for the soaker run. Total preheater pressure remained fairly constant for the SCT coal runs at 2000-2080 psig inlet and 2000-2060 psig outlet. The soaker run was operated at pressures of 1080-2080 psig inlet and 1040-2060 psig outlet. The earlier Illinois 6 coal SCT runs were all made in the coil-only mode at 36% MF coal concentration and 165 SCFH gas rate. Temperature and pressure

covered a wider range from 830-860°F and 2000 to 2400 psig, respectively. The Indiana 5 coal runs, SCT12 to SCT24, were similar except that all periods were run at the more severe conditions, i.e., 860-865°F and 2430-2540 psig inlet pressure.

The antisolvent deashing (ASDA) unit typically operates on first-stage heavy oil product (nominally 500°F⁺) to remove solids by agglomeration and settling. One of several proprietary antisolvents is added to the ASDA feed to promote solids agglomeration. The antisolvent is recovered by batch distillation from the atmospheric-flashed clean overflow (650°F⁻) and the vacuum-flashed dirty underflow (850°F⁻) streams and recycled. A nominal 500 x 850°F fraction is recovered from both streams as batch distillation bottoms to supply about 30% of the first-stage pasting solvent. The clean 650°F⁺ overflow fraction is sent to the LC-Fining unit. The vacuum-flashed underflow stream (850°F⁺) is drummed in the PDU but may be gasified or fluid coked in a commercial unit. During part of the period covered by this report, the PDU was operated in the modified configuration, i.e., second-stage product instead of first stage product was deashed. The feed sources for the ASDA runs analyzed by us are given in Table 13. We received nine samples each of antisolvent deasher (ASDA) feed (F), overflow (O), and underflow (U) and one "cone" (C) sample from Runs ASDA 154 to 165 with Illinois 6 coal-derived material and Run 3ASDA100 with Wyodak coal-derived material. No operating conditions were supplied for the ASDA runs.

The LC-Finer unit is the second stage of the ITSL process. The unit consists of three expanded-catalyst bed reactors in series. Deashed first-stage product (650°F⁺) is converted into high-grade light oil net product (650°F⁻) and a suitable recycle oil for the first stage consisting of heavy oil (650°F⁺). The LC-Finer operated on non-deashed first-stage product for part of Run 3LCF9. The catalyst in use is Shell 324M (Ni/Mo on alumina, promoted with P).

ITSL Run 3LCF9 was made with Burning Star (Illinois 6) coal in the integrated mode. We received a total of forty-two samples each of feed (F)

and heavy oil product (P) from Run 3LCF9 (March, 1982, to February, 1983). Two periods were operated on Indiana 5 coal-derived feed in order to produce material for coal/oil agglomeration work by Conoco. Run conditions during periods from which we have samples are given in Table 19. Control temperatures, which are the maximum temperatures in the non-isothermal LC-Finer, ranged from 750 to 780°F. Effective temperatures, which are the average temperatures in all three reactors, ranged from 717 to 756°F. Pressure was constant at 2700 psig. The linear hourly space velocity (LHSV) ranged from 0.21 to 0.61 lbs 850°F⁺/lb catalyst-hr. The feeds to the LC-Finer included deashed and non-deashed SCT product and deashed soaker product.

Feed Coals

Our analyses of the Burning Star, Illinois 6 coal and the Old Ben No. 1, Indiana 5 coal used in the ITSL PDU appear in Tables 34 and 35, respectively. These samples were received from Lummus.

Section 2

SUMMARY

This report includes the results of our analysis of the ITSL PDU runs using Illinois 6, Burning Star coal from March, 1982, to February, 1983. One hundred fifty-six samples were analyzed. In addition, we performed several sets of experiments designed to answer specific questions related to ITSL, we analyzed a set of samples from H-Coal Pilot Plant Run 9 and we worked on the development of analytical methods. Our principal conclusions derived from this work follow.

- SCT Runs 2SCT12-2SCT18: Illinois 6 Coal
 - The pasting solvent (PS) in these runs generally contained less distillate and was less highly hydrogenated than pasting solvent distillates generated for earlier SCT runs. Reasons for these differences include an improved procedure for the separation of the LC-Finer heavy-oil product and the continued purging of the system of the all-distillate start-up oil.
 - SCT coal conversions (THF solubles) and 850[°]F⁻ yields for these runs (840[°]F) were intermediate between previous higher temperature (850-860[°]F) and lower temperature (830-850[°]F) Illinois 6 coal SCT runs. This confirms the conclusion that increasing temperature increases both THF conversions and 850[°]F⁻ yields in the first stage.
 - The gas rate was reduced from 165 to 95 SCFH in the PDU to increase residence times, but this had no significant effect on calculated yields or on donor hydrogen depletion. This may indicate that residence times did not change with gas rate as supposed.
 - An SCT run (Run 2SCT14) made in the absence of coal (pasting solvent only) showed that: 1) the distillate becomes partially

depleted of donatable hydrogen, 2) part of the insoluble organic matter (IOM) is converted to solubles, 3) some preasphaltenes are converted to benzene solubles, and 4) a portion of the resid is converted to 850°F^- distillate and gas. Therefore the highly hydrogenated pasting-solvent resid is the partial source of the first-stage distillate products.

- Operations with the soaker reactor (Run 3SCT17) consumed more solvent hydrogen and produced more 850°F^- and less preasphaltenes than coil-only operations. Reduction in soaker temperature from 820 to 800°F reduced solvent hydrogen depletion and lowered the yield of 850°F^- .
- Antisolvent Deashing Runs ASDA154-ASDA165
 - Runs ASDA154 to ASDA165 confirm earlier conclusions that there is only minor selectivity for soluble-resid components in the ASDA system. It is primarily the THF-insoluble/pyridine-soluble preasphaltenes that precipitate in the ASDA process and concentrate in the underflow stream.
- LC-Finer Run 3LCF9: Illinois 6 Coal
 - Over the course of the run, 850°F^+ conversions decreased from about 42% to about 17% presumably as a result of catalyst deactivation.
 - Conversions obtained in Run 3LCF9 were generally comparable to those obtained at comparable conditions in Run 3LCF8 (Illinois 6 coal) and Run 3LCF7 (Indiana 5 coal).
 - 850°F^+ conversion activity, as measured by the first-order kinetic pre-exponential factor, declined rapidly in the first part of Run 3LCF9 until a catalyst age of about 350 lbs 850°F^+ /lb catalyst and thereafter declined at a relatively modest rate. The processing of non-deashed feed did not significantly

affect conversion activity. Activity for conversion of the product from the soaker reactor is the same as for the product from the SCT reactor to within the scatter of the data.

- First order kinetics calculations, using our analytical data on Run 3LCF9 samples, agree well with results reported by Lummus and indicate the general equivalence of our analytical methods.
- With increasing catalyst age there was less hydrogenation of the resid and less deoxygenation of the distillate.

- Reactions of ITSL Resids

- 15 to 25% of the pasting solvent resid can be converted to distillate and gas at SCT conditions in the absence of coal and molecular hydrogen. The pasting solvent resid is the source of a large portion of first-stage distillate yields.
- Based on batch experiments simulating LC-Finer conditions, but without a catalyst, 850°F^{+} conversions of up to 27 wt % are obtained by non-catalytic reactions. This compares to PDU conversions of 50% with a fresh catalyst and 35% with an aged catalyst (1000 lbs 850°F^{+} /lb catalyst). All the net thermal conversion in the batch experiments is from the preasphaltene and IOM fractions and increases with residence time from 1 to 1.5 to 2 hours. The results indicate that a significant portion (as much as 75%) of the conversion of coal-derived resid to distillate in the catalytic second stage of the Lummus ITSL unit may in fact be achieved under non-catalytic conditions. This "thermal" conversion is promoted by the superior hydrogen donor characteristics of the distillate portion of the reactor inventory. These results support our hypothesis that preasphaltene conversion is essentially all thermal, asphaltene conversion is at least partially thermal and that the primary role of catalyst in two-stage liquefaction is de-functionalization and hydrogenation of distillate and "light" resid.

- Effect of Boiling Point on Solvent Quality
 - A set of twelve microautoclave runs at simulated SCT conditions was made using distillation fractions of an ITSL pasting solvent to augment earlier work on the relationship of solvent quality and boiling point. Earlier conclusions were generally reinforced, i.e., the light ends boiling below about 350°C (662°F) are fairly poor donor solvents compared to the heavier material. At conditions where donor concentration is the parameter limiting conversion, the 350 x 425°C (662 x 797°F) cut is the optimum donor solvent.
- H-Coal Pilot Plant Run 9: Wyodak Coal
 - We analyzed a small set of samples from H-Coal Pilot Plant Run 9 (Wyodak, Syncrude). The samples analyzed are higher quality materials than PDU Run 10 (Wyodak, Syncrude) samples taken at near steady-state conditions. The few Pilot Plant Run 9 samples analyzed were not taken at equilibrium. The usefulness of the data is limited by the small size of the sample set and because they are not steady-state samples.
- ¹H-NMR Methods of Resid Analysis
 - Three different ¹H-NMR methods for determining proton distributions of 850°F⁺ resid samples were compared and found to give equivalent results as long as care was taken during integration.

Section 3

DISCUSSION

ITSL OPERATIONS WITH ILLINOIS 6 COAL: FIRST-STAGE OPERATIONS

Comparisons of Runs 2SCT12 to 18 with Earlier Illinois 6 Runs

The first-stage runs described in this report (Runs 2SCT12 to 2SCT18) were made between April, 1982, and February, 1983, in association with LC-Finer Run 3LCF9 using Illinois 6 Burning Star coal. In all, we analyzed twenty-one pasting solvents (PS) and twenty-three heavy-oil products (HO). The start-up solvent used in Run 2SCT12 was produced in Run 3LCF8. These SCT runs differed from an earlier set (Runs 2SCT1 to 10) in several ways. The temperature, which had earlier been reduced from 860 to 830°F, was increased to 840°F. Pressure was 2000 psig for these runs; most of the earlier runs employed 2400 psig. Finally, the gas rate, typically 165 SCFH in earlier runs, was decreased in order to increase residence times. Complete run conditions are given in Table 1. Run 2SCT14 was made without coal in order to study the effect of SCT conditions on the pasting solvent. Run 2SCT17 was made with the soaker reactor. These two special runs are discussed separately from the others.

Pasting Solvent Composition

The pasting solvent used for Runs 2SCT12 to 18 generally contained less distillate and more resid than that from earlier Illinois 6 SCT runs. The ranges of the pasting solvent components are compared below for the recent and earlier SCT runs.

Runs	Range, wt % of Pasting Solvent			
	850°F Dist	THF Sol Resid	IOM	Ash
2SCT12-18	41 - 62	32 - 55	<1 - 4	<1 - 4
2SCT3-10	57 - 70	27 - 38	<1 - 8	<1 - 3

Complete data are given in Table 2 and Reference 1. Two potential causes of the "heavier" pasting solvent are: 1) The component of the pasting solvent produced in the LC-Finer (60-80%) was vacuum flashed to remove essentially all 650°F⁻ material for the latest SCT runs.⁽²⁾ The LC-Finer product component used in the earlier pasting solvents had been collected in high-temperature separators which did not remove all the 650°F⁻ material. 2) The start-up solvent used in the earlier runs was an all-distillate creosote oil that was increasingly replaced with heavier coal-derived solvent with time.⁽²⁾

The pasting solvent distillate used in the latest runs was less highly hydrogenated as measured by its hydrogen aromaticity.

<u>Runs</u>	<u>Range, Proton Distributions</u>		
	<u>Aromatic</u>	<u>Cyclic</u>	<u>Alkyl</u>
2SCT12-18	27-36	33-37	29-36
2SCT3-10	19-29	40-44	26-40

Complete data are given in Table 6 and Reference 1. The reasons for this difference are not clear; however, the points noted above may be factors.

The differences discussed above would suggest that the pasting solvents used in Run 2SCT12-18 were poorer hydrogen donors than those from the earlier runs. However, the difference is not great enough to reduce conversion in the standard microautoclave test (Table 10) below its usual high level of 90-92%.

Conversions and Yields

THF conversions and 850°F⁻ distillate yields calculated from our analytical data are given in Tables 11 and 12. These yields are roughly intermediate between the previous higher temperature (850-860°F) and lower temperature (830-850°F) Illinois 6 coal SCT runs as shown below.

Runs	Temp, °F	wt % MAF Coal	
		THF Conversion	850°F Yield
2SCT12-18	840	84 $\pm$ 2	14 $\pm$ 11
2SCT3-6	860-850	88 $\pm$ 4	34
2SCT7-10	850-830	80 $\pm$ 3	16

The methods used to calculate yields and conversion are given in Appendix 1. The large standard deviation (above table) in the 850°F yield of Runs 2SCT12-18 arises at least partially from the widely ranging conditions (coal concentration, space rate, gas rate, etc.) employed. No standard deviation is given for the 850°F yield of Runs 2SCT3 to 10 because the averages shown are single points calculated from average feed and product compositions. The new data generally confirm earlier conclusions⁽¹⁾ that decreasing SCT temperatures reduce THF conversions and 850°F yields.

Little Effect of Gas Rates on SCT Performance

The "slug" flow characteristics of the coil along with the fact that gas typically occupies about 60-80% of the coil volume⁽²⁾ cause slurry residence time to be relatively insensitive to coal space rate.⁽³⁾ Therefore, during Runs 2SCT12 to 18, the gas rate was reduced from the typical value of 165 SCFH down to 52 SCFH in order to increase the slurry residence time in the first stage.

The effect of gas rate on residence time (at current run conditions), as reported by Lummus⁽²⁾ is shown below.

<u>Gas Rate, SCFH</u>	<u>Residence Time, Min</u>
165	2.0
129	2.7
106	3.1 (Interpolated)
95	3.3
52	4.1 (Interpolated)

One might expect, therefore, that a decrease in gas rate would increase 850°F⁻ yield by increasing the extent of thermal cracking. However, our results do not support this conclusion.

<u>Period</u>	<u>Gas Rate, SCFH</u>	<u>wt % MAF Coal</u>	
		<u>850°F⁻ Yield</u>	<u>THF Conversion</u>
2SCT12-0418	165	12.3	83.6
2SCT16-1212	158	21.7	80.2
2SCT12-0531	129	12.2	84.4
2SCT13-0708	129	2.5	83.5
2SCT18-0223	106	4.2	82.2
2SCT12-0701	95	13.5	85.4
2SCT13-0712	52	35.2	88.1

Complete data are shown in Table 12.

Additionally, there appears to be no consistent relationship between gas rate and the depletion of solvent hydrogen as measured by ¹H-NMR. The relative depletion of solvent hydrogen can be gauged by the relative increase in aromaticity between a pasting solvent distillate and its corresponding heavy-oil product distillate.

<u>Period</u>	<u>Gas Rate, SCFH</u>	<u>% Aromatic H</u>		
		<u>PS Dist</u>	<u>HO Dist</u>	<u>Δ</u>
2SCT12-0418	165	26.7	29.9	3.2
2SCT16-1212	158	38.2	41.2	3.0
2SCT12-0531	129	29.2	33.2	4.0
2SCT13-0708	129	31.9	32.5	0.6
2SCT18-0223	106	35.9	43.8	7.9
2SCT12-0701	95	26.4	31.1	4.7
2SCT13-0712	52	36.3	37.8	1.5

Complete data are shown in Tables 6 and 7. The run periods compared above were operated at nearly the same conditions ($T = 836\text{--}840^{\circ}\text{F}$, $P = 2000\text{--}2060$ psig, coal space rate = $141\text{--}182$ lbs MF/hr-ft³ and coal concentration = 30-36% MF).

Both the 850°F yield and the change in aromaticity are sensitive to fairly large changes in residence time (see section on "Soaker Operations"). That there is no consistent relationship between these variables and gas rate may indicate that residence times do not change with gas rates in the manner reported.

Pasting Solvent Reactions at SCT Conditions

Run 2SCT14 was made with no coal feed in order to determine the effect of SCT reaction conditions on the pasting solvent. The first of three periods (0916) was operated at conditions typical of SCT operations, i.e., 840°F and 2000 psig. The temperature was decreased to 800°F for the next two periods (0917A and 0917B) and the pressure was decreased to 1000 psig for the last period (0917B).

The product yields from these periods, calculated on a hypothetical 36% MF coal feed bases, are shown below.

Period	THF Conversion	Yields, wt % MAF Coal				Ash Balance
		Total 850°F	Oils	Asphaltenes	Pre- asphaltenes	
2SCT14-0916	2.3	3.0	0.1	1.0	-0.4	88.5
0917A*	1.5	2.7	4.6	-1.8	-3.4	95.0
0917B	2.7	5.6	1.9	-0.4	-3.9	95.0

*Feed assumed average of 0916 and 0917B.

The yields do not show enough of a difference to determine the effects of temperature and pressure. However, in each case there is a small conversion of IOM, a small net yield of 850°F and a small net loss of preasphaltenes.

The proton distributions of the pasting solvent and heavy oil product distillates of Run 2SCT14 differ in such a way as to indicate a mild loss of donatable hydrogen upon passing through the coil. As shown below, the distillates became more aromatic and lost cyclic-aliphatic protons. The change is greatest for period 0916, the only 840°F period.

<u>Period</u>	<u>Proton Distributions</u>			
	<u>PS Dist</u>		<u>HO Dist</u>	
	<u>Aromatic</u>	<u>Cyclic Aliphatic</u>	<u>Aromatic</u>	<u>Cyclic Aliphatic</u>
23CT14-0916	26.5	38.0	31.4	35.0
0917A	-	-	29.8	36.5
0917B	27.3	37.9	29.3	36.3

In a later section of this report, we detail a set of microautoclave experiments designed to test the effect of SCT conditions on the pasting solvent. Those experiments were operated at 860°F for 5 minutes (3 min at temp) in the absence of hydrogen. At those more severe conditions, the extent of reaction was greater than that found in Run 2SCT14 (14% vs 4% MAF 850°F⁻ yield).

At SCT conditions, in the absence of coal, the pasting solvent distillate donates hydrogen to the heavier portions of the pasting solvent. SCT reaction conditions cause some of the IOM to convert to THF solubles, some of the preasphaltenes to convert to benzene solubles and some of the 850°F⁺ to convert to 850°F⁻ distillate and gas.

Soaker Operations

Run 2SCT17 used a partially-back-mixed soaker reactor in addition to the SCT coil. The soaker reactor was designed for use in later Wyodak coal runs, but Illinois 6 coal was used for the "shake-down" run. The soaker reactor increases the first stage residence time to 10-15 min compared to 2-3 min with the coil alone, and was generally operated at lower temperatures. Run 2SCT17 was made at 820-800°F and at 2000-1000 psig. Coal space

rate, coal concentration and gas rate were kept constant throughout Run 2SCT17. Complete run conditions are given in Table 1.

Comparison of soaker and SCT Operations

Other than the use of the additional soaker reactor, Run 2SCT17 differed from typical SCT operations in its lower temperature, lower space rate, generally lower gas rate and for some periods its lower pressure conditions. Calculated yields for each first-stage operating period with good ash balances are shown in Table 12. The average yield structures ($\pm$ standard deviation) are compared below for SCT and soaker runs.

	THF Conversion	Yields, wt % MAF Coal			
		Total 850°F ⁻	Oils	Asphaltenes	Preasphaltenes
SCT	84 $\pm$ 2	14 $\pm$ 11	20 $\pm$ 7	16 $\pm$ 3	33 $\pm$ 3
Soaker	84 $\pm$ 3	24 $\pm$ 7	19 $\pm$ 6	17 $\pm$ 2	24 $\pm$ 2

The large standard deviations for the yields of some of the products are certainly influenced by the wide range of conditions over which each set of operations was made. They are included to show the experimental range and do not reflect reproducibility.

The average soaker period yielded significantly (three standard deviations) less preasphaltenes and a correspondingly greater amount of 850°F⁻. Therefore, the extent of the thermal reaction was increased by the use of the soaker reactor, yielding a "lighter" product slate.

The average increase in hydrogen aromaticity between the pasting solvent distillate and the corresponding heavy oil product distillate was 7.6 $\pm$ 1.4% for soaker periods and 3.6 $\pm$ 2.6% for coil-only periods (Tables 6 and 7). This indicates that a greater amount of hydrogen was lost from the distillate solvent during soaker operations and is consistent with the "lighter" product slate.

Since soaker operations produced more net 850°F⁻, the heavy oil product distillate contained more coal-derived, as opposed to pasting-solvent derived, distillate. It would be expected that distillates produced directly from the coal would contain more hetero-compounds than pasting solvents because the latter is largely produced by the catalytic LC-Finer. As shown in Table 9, the heavy oil product distillate from the soaker run, Run 2SCT17, does in fact have a higher concentration of phenolics than the SCT runs (0.69 ± 0.07 vs 0.052 ± 0.03 meq/g).

Effect of Soaker Temperature

The soaker reactor was operated at 820°F for five periods of Run 2SCT17 and at 800°F for two periods. Decreasing the temperature at constant pressure caused the distillate product to be slightly less depleted of hydrogen as evidenced by the lower aromaticity of the product distillate at lower temperatures.

	<u>% Aromatic Hydrogen in HO Dist</u>	
	<u>2000 psig</u>	<u>1000 psig</u>
820°F	41.8	41.7
800°F	39.0	40.3

Complete data are given in Table 7.

The decrease in soaker temperature also caused a reduction in 850°F⁻ yield (Table 11). Averages are shown below.

	<u>850°F⁻ Yield, wt % MAF Coal</u>	
	<u>2000 psig</u>	<u>1000 psig</u>
820°F	22.5	29.0
800°F	13.2	23.1

This is consistent with a lower level of thermal cracking and the lower depletion of solvent hydrogen at lower temperatures. These same temperature

effects were seen earlier in coil-only operations. (See Reference 1 and that section of this report).

Effect of Soaker Pressure

During soaker operations (Run 2SCT17), two pressure levels were tested at each of two temperatures. The yield structure for each run period is shown in Table 11. As shown by the table of average 850°F⁻ yields above, a decrease in pressure tended to increase 850°F⁻ yields at both temperature levels but to decrease residual oils (850°F⁺, hexane soluble) yields by nearly as much.

<u>P, psig</u>	<u>Oils Yield, wt % MAF Coal</u>	
	<u>800°F</u>	<u>820°F</u>
2000	27.9	20.6
1000	14.0	15.4

The apparent relationship between pressure and soaker yields indicated by the data is unusual. Some of the data on which the relationship is based are derived from single run periods and future runs would be desirable to establish the effect of pressure on soaker yields.

Since the decrease in pressure during Run 2SCT17 tended to decrease residual oil yields, it therefore tended to increase the relative amount of preasphaltenes in the soluble resid product (as shown below) even though their absolute concentration in the pasting solvent was unchanged.

<u>P, psig</u>	<u>wt % of Soluble Resid</u>			
	<u>PS</u>		<u>HO Product</u>	
	<u>Oils</u>	<u>Pre-</u>	<u>Oils</u>	<u>Pre-</u>
		<u>asphaltenes</u>		<u>asphaltenes</u>
2000	52.0	23.0	45.4	28.0
1000	52.8	22.3	42.0	31.6

Complete data are given in Tables 4 and 5.

A similar pressure effect was seen earlier in coil-only operations.⁽¹⁾

ITSL OPERATIONS WITH ILLINOIS 6 COAL: DEASHING RUNS

We received samples from nine antisolvent deashing (ASDA) runs made in the ITSL PDU between May, 1982, and March 1983. Eight of the runs were made during Illinois 6 coal operations and one during Wyodak coal operations. The feed source for each run is listed in Table 13. Runs ASDA 157 to 160 were made in the modified configuration, i.e., the feed to the ASDA unit was LC-Finer (second stage) product. Runs 165 and 3-100 were made with product from the soaker reactor. For each run, we received a sample of feed (F), overflow (O) and underflow (U). We also received a "cone" (C) sample from Run ASDA160. Cone samples are instantaneous underflow samples; most underflow samples are composited for three hours.

Component distributions of the sample are given in Table 14. The removal of ash and tetrahydrofuran (THF) insoluble organic matter (IOM) from the overflow and their concentration in the underflow (relative to the feed) is apparent in most cases though deashing was marginal during Run 154.

Solvent fractionation analyses of the THF-soluble resids are given in Table 15. With the exception of the Wyodak coal run, the feed, overflow and underflow samples from the same run generally give similar solvent fractionation results. This confirms earlier results from Indiana 5⁽³⁾ and Illinois 6⁽¹⁾ coal runs that showed that THF-soluble preasphaltenes are not consistently concentrated in the underflow, e.g., by precipitation, and that therefore this phenomenon was not necessary for deashing.

The liquid column fractionation method for solvent fractionation analysis was modified to handle whole resids (soluble and insoluble organics and ash) and to give a preasphaltene fraction that was benzene-insoluble/pyridine soluble. Results of these analyses (Table 16) show that the pyridine-soluble preasphaltenes do concentrate in the underflow.

The extent of the selective concentration of preasphaltenes in the underflow can be taken as the ratio:

Preasphaltenes in Underflow Resid
Preasphaltenes in Overflow Resid

This ratio is presented in Table 18 for preasphaltenes determined by both THF solubility and by pyridine solubility. The deashing efficiency of each ASDA run is also given. As the data illustrate, there was no significant concentration of THF-soluble preasphaltenes in the underflow except for Run 160 and the Wyodak coal run. The average for all the Illinois 6 coal runs is within one standard deviation of unity. The average for the pyridine-soluble preasphaltenes from the Illinois 6 coal runs is greater than two standard deviations above unity showing that the pyridine-soluble preasphaltenes do concentrate in the underflow.

These data, therefore, confirm earlier work that show that deashing is effected largely by precipitation of the THF-insoluble/pyridine-soluble preasphaltenes. The lone Wyodak coal run showed extensive concentration of preasphaltenes in the underflow.

ITSL OPERATIONS WITH ILLINOIS 6 COAL: RUN 3LCF9

The second-stage run described in this report, Run 3LCF9, was made between March, 1982, and February, 1983, in association with first-stage Runs 2SCT12 to 2SCT18 with Illinois 6 Burning Star coal. The start-up solvent was generated in Run 3LCF8. Run conditions are given in Table 19.

Through the first 500 lbs 850°F⁺/lb cat processed, the PDU was operated in its normal configuration, i.e., the LC-Finer processed deashed SCT product. From 500 to 1500 lbs 850°F⁺/lb cat processed, the PDU was operated in the modified configuration, i.e., the LC-Finer processed non-deashed SCT product. During the period following 1500 lbs 850°F⁺/lb cat, the LC-Finer processed deashed soaker-reactor product. During two periods in the middle of Run 3LCF9, 1005 and 1011, deashed SCT product from the Indiana 5 coal runs was fed to the LC-Finer to make material for other studies.

850°F⁺ Conversion vs Catalyst Age -- Run 3LCF9

Over the course of Run 3LCF9, 850°F⁺ conversion decreased from about 42% to about 17% apparently as a result of catalyst deactivation. Conversions, calculated by the method in Appendix 2, are given in Table 31 and are shown graphically in Figure 2. Figure 2 clearly shows the decrease of 850°F⁺ conversion with catalyst age and also illustrates the inverse relationship of space velocity and 850°F⁺ conversion.

Conversions in Runs 3LCF7, 3LCF8 and 3LCF9 Were Similar

The conversion levels in Runs 3LCF7, 3LCF8 and 3LCF9 were generally comparable at comparable operating conditions. The feed to Run 3LCF7⁽³⁾ was made with Indiana 5, Old Ben No. 1 coal, while that to Runs 3LCF8 and 3LCF9 were made with Illinois 6, Burning Star coal. All three runs used Shell 324M catalyst. The early part of Run 3LCF7, up to 400 lbs 850°F⁺/lb cat, was made at an overall space velocity of 1.0, relative to Lummus' proprietary reference value. The remainder of the run was made at an overall space velocity of 1.67R, an 850°F⁺ space velocity of 0.44 to 0.59 vol 850°F⁺/vol of settled catalyst, and at 2700 psig and 780°F control temperature.

Run 3LCF8 was made at similar conditions except that the control temperature was 770°F for most of the run.

Both the beginning and end of Run 3LCF9 were operated at lower control temperatures ($\leq 760^\circ\text{F}$). Other parts of the run were made at space velocities of 1.0R and 2.3R. However, a large portion of the middle of the run was made at comparable conditions to Runs 3LCF7 and 3LCF8.

Conversions of 850°F⁺ and of the benzene-insoluble fraction (preasphaltenes-plus-10M) were relatively well-behaved in all three runs during periods of constant operating conditions (Figure 2 and References 1 and 3). The similarity in the performance of the three runs in terms of conversions is shown below.

Run	Coal	Control Temp, °F	Run Periods	Catalyst Age	Avg Conversion, wt %	
					Total 850°F ⁺	Benzene Insolubles
3LCF7	Ind 5	780	58-90	527-1025	32 ± 4	56 ± 3
3LCF8	III 6	770	27-46	299-503	33 ± 2	56 ± 2
3LCF9	III 6	770-780	0826-1214*	697-1340	29 ± 6	56 ± 7

Constant Conditions: 2700 psig, feed S.V. = 1.67 relative;
LHSV ≈ 0.3-0.5 vol 850°F⁺/vol cat hr

*Period 1016 omitted

Over the range of catalyst ages averaged in the above table, 850°F⁺ conversions during the shorter Runs 3LCF7 and 3LCF8 were relatively steady. However, 850°F⁺ conversions decreased in the longer Run 3LCF9 giving rise to the lower average 850°F⁺ conversion. Conversions of benzene insolubles (preasphaltenes and IOM) remained remarkably steady at near 56% for the major part of all three runs. Evidence is presented in a later section ("Thermal Contribution to 850°F⁺ Conversion in the LC-Finer") that preasphaltenes conversion has only an indirect dependence on catalyst activity, so that its relative invariance with catalyst age is expected.

850°F⁺ Conversion Activity -- Run 3LCF9

First-order kinetics is an imperfect model to describe catalyst activity for 850°F⁺ conversion in the LC-Finer. Run 3LCF7 demonstrated several non-first-order phenomena such as sensitivity of the rate constant to space velocity and sensitivity of 850°F⁺ conversion to 850°F⁺ concentration.⁽³⁾ Additionally, there is considerable evidence that much of the 850°F⁺ conversion that occurs in the LC-finer is not directly catalytic. (See section on "Thermal Contribution of 850°F⁺ Conversion in LC-Finer", this report.) Despite these provisions, a first-order kinetic model may be useful to follow the performance of long runs in which space velocity and temperature are varied.

Lummus routinely calculates first-order rate constants (k) and pre-exponential factors (A) based on their analyses for LC-Finer runs. Their results for the first part of Run 3LCF9 indicate an initially rapid decline in 850°F⁺ conversion activity followed by a more moderate decline in activity.⁽²⁾

We applied first-order kinetics to Run 3LCF9 using Conoco's sample analyses and the liquid hourly space velocities (LHSV), weighted hourly space velocities (WHSV) and effective temperatures supplied by Lummus in order to compare results and to check our experimental reproducibility. The calculation assumes a first-order rate expression for three perfectly mixed reactors in series (Table 32). The temperature dependence of the rate constant is assumed to obey the Arrhenius expression

$$k(T) = A \exp \left(- \frac{E_{act}}{RT} \right)$$

The pre-exponential factor, A , is then calculated from the observed rate constant $k(T)$. The behavior of A as a function of catalyst age can be taken as an indicator of 850°F⁺ conversion activity.

Values of k and A based on LHSV and WHSV (850°F⁺) are tabulated in Table 32. Our values agree well with the limited set reported by Lummus⁽²⁾ which are shown in Table 33. Lummus' calculations are based on solids-free 850°F⁺ while Conoco's are based on total (soluble and insoluble) organic 850°F⁺. This agreement indicates the general equivalence of our analytical methods.

A plot of the pre-exponential factor (A) versus catalyst age is shown in Figure 3. The data suggest an initial rapid decline in conversion activity until a catalyst age of about 350 lbs 850°F⁺/lb catalyst, followed by a more gradual decline out to the end of the run. Lummus reported similar behavior for Run 3LCF8.⁽²⁾ The region of rapid initial decline contains only eight data points and is therefore less well defined than the later region.

Spent catalyst analyses⁽³⁾ have indicated that carbon laydown is initially extremely rapid whereas metals deposition is less rapid. The initial period of rapid laydown is followed by a period of much slower deposition. Other workers have concluded that the deposited carbon reduces pore volume and that both the carbon and metals deposition deactivates the LC-Finer catalyst.⁽⁴⁾ therefore, it seems highly likely that the initial rapid decline in the calculated pre-exponential factor (A) with catalyst age is caused by a rapid deposition of carbon and metals on the catalyst.

Taking the pre-exponential factor (A) as a measure of 850°F^{+} conversion activity, there is no evidence that the decline in conversion activity was more severe during operations in the modified configuration of the PDU, i.e., with the LC-Finer processing non-deashed feed. Perhaps this should be expected since even the "deashed" feeds (prior to period 0712 and following period 0129) contained significant levels of ash as shown in Table 21.

Based on the data shown in Figure 3, it is not apparent that the first-stage soaker product (fed beginning 1500 lbs 850°F^{+} /lb cat) gave lower 850°F^{+} conversion activity than the coil-only product.

Additional Effects of Catalyst Age

With increasing catalyst age, not only did more resid remain unconverted but the unconverted resid became more aromatic. Resid proton distributions are given in Tables 27 and 28. The feed resid had hydrogen aromaticities of $47 \pm 3\%$ over the entire run. The product resid had 25% aromatic hydrogen at a catalyst age of 25 lbs 850°F^{+} /lb cat. This increased to about 45% aromatic hydrogen at a catalyst age of about 1750 lbs 850°F^{+} /lb cat. Increasing aromaticity indicates decreasing hydrogenation. The trend of increasing aromaticity (decreasing hydrogenation) with catalyst age is certainly not solely the result of temperature changes; effective temperatures increased then decreased in Run 3LCF9 (Table 19). It must be presumed, therefore, that the decreasing hydrogenation of the resid was at least partially the result of catalyst deactivation.

The concentration of phenolic hydroxyl (-OH) groups in the LC-finer heavy-oil product tended to increase with catalyst age (Table 29). The concentration of these species in the feed also increased, but to a much lesser extent. This indicates that deoxygenation decreased with catalyst age; presumably because of catalyst deactivation.

NON-CATALYTIC REACTIONS AT ITSL CONDITIONS

The ITSL process is designed to produce a distillate product, and the LC-Finer catalyst plays an important role in the production of distillate. Based on data from Runs 3LCF7⁽³⁾ and 3LCF8⁽¹⁾ it appears that a significant amount of the resid conversion is not a direct effect of the catalyst. The catalyst surely is required to maintain solvent quality but in the presence of an adequate hydrogen donor solvent, ITSL resids will convert to distillable material in the absence of catalyst. We performed two sets of microautoclave experiments to examine this non-catalytic component of overall resid conversion which we term "thermal" resid conversions. This work was designed to give a qualitative understanding of thermal effects and to obtain an order-of-magnitude quantitation of thermal conversion in a high-quality donor solvent.

Thermal Conversion of Pasting Solvent Resids

A microautoclave study was made to investigate the thermal conversion behavior of LC-Finer product (pasting solvent) resids at short contact time (SCT) conditions. The operation of the ITSL PDU has demonstrated a relatively large (13%⁽³⁾ to 28%⁽⁵⁾ MAF coal basis) production of 850°F⁻ distillate and gas (including hetero gases) from Indiana 5 coal in the first stage (SCT) unit. We suggested⁽³⁾ that this yield of light products, which is not usually obtained from bituminous coals at this reaction conditions (860°F 2-5 min), was primarily the result of thermal cracking of the highly hydrogenated residual component (850°F⁺) of the pasting solvent (PS).

To study the contribution of thermal conversion of the PS resid to SCT yields, a rapidly heated/cooled microautoclave batch reactor was used to

model the SCT coil reactor using similar temperature and residence time conditions. The details of the experimental procedure are given in the "Experimental" section. Because we were interested in determining the thermal cracking behavior of pasting solvent resids, all reactions were run in the absence of coal and hydrogen. The microautoclave is believed to be a reasonably good model of the slug-flow SCT coil reactor.

Three well-characterized⁽³⁾ pasting solvent resids (850°F^{+}) from ITSL operations with Indiana 5 coal were selected for testing. The samples selected spanned a wide range of quality, as measured by oils, asphaltenes, preasphaltenes, and IOM content.

The compositions of the three pasting-solvent resids tested are shown in Table 36. They are ordered by increasing quality, i.e., increasing oils content and decreasing preasphaltenes and IOM content and span a large portion of the range of pasting solvent resid quality.

Yields and conversions of each fraction, calculated from the known feed (IN) and product (OUT) compositions, are shown in Table 37. These data are summarized below. Negative conversions indicate that more of the fraction in question was produced than converted.

<u>Resid Sample</u>	<u>% Conversion</u>				<u>Corresponding MAF Coal Conversion to 850°F^{-}</u>
	<u>Oils</u>	<u>Asphaltenes</u>	<u>Benzene Insolubles</u>	<u>Total 850°F^{+} Organic</u>	
SCT16-6PS3381	-3.4	-10.9	54.3	22.4	14.8
SCT14-PS51280	25.9	8.2	31.8	24.3	16.0
SCT14-PS31280	20.4	11.8	12.2	16.9	11.1

Total 850°F^{+} conversions range from 24 to 17%. The lowest quality resid (SCT16-6PS) gave very high benzene-insolubles conversion, and net yields of oils and asphaltenes. With decreasing feed preasphaltene content,

benzene-insolubles conversion decreases. This may indicate that the pre-asphaltenes and IOM present in high quality resids are less thermally reactive.

The table above also lists 850°F^{+} conversion on a wt % MAF coal basis. This is the contribution of the pasting solvent to the 850°F^{-} yield which would be measured if the given pasting solvent were fed in a slurry with 36% coal to the SCT unit and the PS resid reacted as in the microautoclave tests. This value ranges from 11 to 16%. As stated previously, actual SCT operations with Indiana 5 coal in the ITSL PDU give 850°F^{-} conversions of 13 to 27% MAF. These experiments demonstrate that a large portion of this can be accounted for as thermal conversion of the pasting solvent resid.

In the FCC-Fliner, the resids become highly hydrogenated but cracking reactions are moderated by the relatively low operating temperature (ca. 750°F). The hydrogenated resids then pass to the high temperature (860°F) SCT unit where they are thermally cracked to lighter materials.

Proton distributions of the feed and the three product distillates are shown in Table 38. The product distillates are all more aromatic than the feed distillate. This is caused by hydrogen donation and cracking reactions involving the distillate. The compositions of the product distillates are, of course, also affected by the 850°F^{+} material that cracked into the distillate range.

The feed distillate is an excellent donor solvent. It may be necessary to employ such a high quality solvent in order to prevent retrograde reactions from producing coke from the resids at SCT conditions.

Proton distributions of the THF soluble feed and product resids are shown in Table 39. The product resids are all significantly more aromatic than the respective feed resids. This indicates that the material that cracked to the 850°F^{-} range was relatively more highly hydrogenated and less aromatic than the remaining non-converted material.

Thermal Contribution of 850°F⁺ Conversion in the LC-Finer

A microautoclave study was made to determine the contribution of thermal effects to 850°F⁺ conversion in the LC-finer and by difference, the catalyst effects. Three microautoclave experiments were performed at conditions simulating those in the LC-Finer except that catalyst was absent. It is recognized that a batch microautoclave can only approximate the performance of the LC-finer continuous unit. The LC-Finer has three stages, each an expanded catalyst bed and the kinetics of conversion are better in the "plug flow" microautoclave than in the back-mixed LC-Finer unit. On the other hand, the hydrogen charge to the microautoclave is also done batchwise so that hydrogen partial pressure declines throughout a run as hydrogen is consumed. In the LC-finer, hydrogen partial pressure can be maintained through the rate of hydrogen feed.

The results of the three experiments are shown in Table 40. Table 41 is a summary of conversions versus residence time for the three runs and for comparison include results from the ITSL PDU during period 3LCF7-48-6381 when the same resid was processed and from periods with a relatively fresh and a relatively aged catalyst. ⁽³⁾

With increasing residence time, total 850°F⁺ organic conversion increased from 19.9 to 26.6%. The PDU 850°F⁺ conversion obtained on the same resid was 46.8%. ⁽³⁾ PDU 850°F⁺ conversions during Run 3LCF7 ranged from about 50% with a fresh catalyst to about 35% with an aged (1000 lbs 850°F⁺/lb catalyst) catalyst. ⁽³⁾ During that run, space velocity was also varied. Using the data from the two hour run, it can be seen that the thermal/donor effect can account for 50-75% of the total 850°F⁺ conversion obtained in the (catalytic) LC-Finer.

The LC-Finer is typically fed 100 to 150 lbs 850°F⁺ for every 100 lbs MAF coal fed to the integrated process. ⁽⁶⁾ Therefore, the 20 to 27% 850°F⁺ conversion obtained in the autoclave tests represents a 26 to 35% conversion of MAF coal to 850°F⁺ distillate in the conceptual integrated unit based on a feed rate of 133 lbs 850°F⁺/100 lbs MAF coal. ⁽⁷⁾

Conversions of hexane insolubles (preasphaltenes plus IOM and asphaltenes) also increased with residence time. Oils conversion was relatively constant. Negative conversions indicate that more of the material in question was created than converted.

Preasphaltenes plus IOM (benzene insolubles) conversion in the microautoclave tests closely approaches the conversion obtained in the PDU. This supports the contention that this conversion is largely thermal as previously concluded from continuous unit data.⁽³⁾ The conversions of the oils and asphaltenes are significantly lower in the microautoclave experiments than in the LC-Finer, indicating that they are more affected by the catalyst. Presumably, the generally smaller oil and asphaltene molecules are better able to enter the catalyst pores and reach the active sites than the preasphaltenes or IOM which are generally larger molecules.

The fact that the asphaltene conversion does increase with time, i.e., becomes less negative, indicates that the asphaltenes are capable of thermal conversions.

Proton distributions of the feed and product distillates from the microautoclave runs are given in Table 42. The product distillates are all more aromatic and have a lower concentration of cyclic protons than the feed. This is the expected result of hydrogen donation from the distillate. In the LC-Finer PDU, the hydrogenated product distillate is less aromatic and contains higher donor concentrations than the feed. The decreased concentration of alkyl and protons in the products probably result from cracking reactions. The proton distributions of the "spent" distillates are all similar indicating similar levels of donor hydrogen depletion. The interpretation of the distillate composition data is somewhat confused by the fact that some of the product distillate was created from cracking of the feed resid.

The proton distributions of the feed and product THF soluble resids from the microautoclave experiments are given in Table 43. As with the

distillates, the soluble resid products are all more aromatic than the corresponding fraction of the feed indicating that they are less hydrogenated. In the LC-Finer, hydrogenation produces a less aromatic resid.

The solvent fractionation data of the THF soluble feed and product resids, given in Table 44, show that the product soluble resids have a higher concentration of oils and asphaltenes and a lower concentration of preasphaltenes than the corresponding feed component. This is similar to what occurs in the LC-Finer and is probably largely caused by cracking.

Oils are generally less highly hydrogenated, less aromatic, less functional and of lower molecular weight than preasphaltenes. The conversion of preasphaltenes in a resid then should cause the material to become either more highly hydrogenated or lower molecular weight. The ^1H -NMR data given above show that the product resids from the microautoclave runs are less highly hydrogenated than the feed and therefore are most likely lower molecular weight. On the other hand, the LC-Finer produces a resid that is both more highly hydrogenated and lower molecular weight than the feed. The reasons for the differences are probably two-fold. First, the LC-Finer catalyst keeps a high concentration of donors in the distillate solvent that hydrogenates the resid whereas in the microautoclave, the distillate becomes relatively depleted of donors because of the absence of the catalyst. Second, the LC-Finer has a continuous hydrogen feed whereas the microautoclave has a single hydrogen charge that decreases in partial pressure with time. These results support the contention that the primary role of the catalyst in ITSL other than defunctionalization is hydrogenation of the distillate and "light" resid. Cracking of the heavy resid is a less important function of the catalyst because it is largely accomplished by thermal means.

Potential Extent of Thermal Yields in Integrated Process

The modeling experiments described above resulted in 11 to 16% (first stage) and 26 to 35% (second stage) yields of total 850°F^- on an MAF coal basis. Simple addition of these gives 37 to 51% total thermal 850°F^- yields by weight of MAF coal. This is 50 to 75% of the 75%⁽⁷⁾ yield given by the

total ITSL process. Of course, in actuality there is certainly a trade-off in obtainable thermal yields between the two stages. However, the 37 to 51% range is a useful measure of the significance of the thermal reactions.

Solvent Quality at SCT Conditions vs Boiling Point

A study relating the solvent quality of ITSL pasting solvent fractions to boiling point was previously reported.⁽⁸⁾ In that study, a pasting solvent sample was fractionated in a low efficiency distillation and the fractions were tested by extracting Old Ben No. 1, Indiana 5 coal in the microautoclave at our standard kinetic and equilibrium test conditions under N₂ pressure. Proton distributions and hydrogen contents were also determined on the fractions. It was concluded that the heavier fractions nominally boiling above 375°C (707°F) were better quality donor solvents than the light fractions. It was also concluded that the quantity of donatable hydrogen decreased with boiling point above 415°C (797°F) but that the donor activity remained high.

The microautoclave test conditions used in the previous study were chosen to determine the relative quantity and activity of donors present in the solvent fractions. It was deemed desirable to test the fractions at conditions more closely resembling first stage reactor conditions. Therefore, several additional microautoclave tests were performed using the same distillation fractions and coal but at simulated short contact time (SCT) conditions under nitrogen pressure, i.e., 860°F, 5 min total time (ca. 3 min @ 860°F), 9 g solvent, 5 g coal and 1000 psig cold N₂ pressure.

Results are listed in Table 45 and plotted in Figure 4. Reproducibility is ca. $\pm 1\%$ absolute. The two lowest boiling distillation fractions were unable to sustain the coal in solution and gave noticeable coke formation. No fraction tested boiling above 311°C gave coke formation. THF conversions increased with boiling point up to fraction 7 or 10 (349 or 383°C) then decreased. This relationship between conversion and boiling point is similar to that obtained in the earlier work using the equilibrium test.

The cause of the maximum in the plot of conversion versus boiling point is probably the same for both the SCT and equilibrium tests and can be explained with the data plotted in Figure 5. Hydrogen content decreases continuously with boiling point. Condensed aromatic hydrogen content increases with boiling point through at least 450°C. Cyclic aliphatic hydrogen content, which is related to donor hydrogen content, increases slightly through 350°C, then decreases. Both cyclic aliphatic and condensed aromatic hydrogen are beneficial to solvent quality. From the initial boiling point (IBP) of the sample to the 350°C fraction, both parameters increase, thus increasing solvent quality. The maximum conversion is reached at a boiling point of 350–425°C while the condensed aromatic hydrogen content continues to increase but the cyclic aliphatic hydrogen content begins to decrease. Above a boiling point of 450°C, both cyclic aliphatic hydrogen and condensed aromatic hydrogen decrease causing conversions to decrease at the SCT and equilibrium conditions. The highest quality donor solvent, therefore, is the 350–425°C fraction.

At the SCT conditions, the 470°C⁻ (878°F⁻) composite gave 87% conversion whereas most pasting solvent distillates from Indiana 5 coal give ca. 90% conversion.⁽³⁾ This is not surprising since ¹H-NMR showed this sample to be less hydroaromatic than most pasting solvent distillates. The absence of nitrogen over-pressure had no significant effect. The composite sample gave somewhat higher conversion than the weighted average of the individual fractions.

Actual boiling point distributions of several of the distillation fractions were determined by GC/MS using a methyl silicone capillary column and are given in Table 46.

These experiments in addition to those reported earlier clearly show that the heavier distillable portion of the pasting solvent is a superior donor solvent to the light ends boiling below 350°C (662°F). Under certain reaction conditions such as those where donor concentration is the limiting

parameter, the 350 x 415°C (662 x 797°F) cut is the optimum donor solvent for conversion.

H-COAL PILOT PLANT RUN 9

H-Coal Pilot Plant Run 9 was made with Wyodak coal in the Syncrude mode (835°F, 25 lbs/hr/ft³) from February 13 to May 5, 1982. Coal feed commenced on March 17. Shut-down procedures were begun on April 25, 1982. During Run 9 there were three sustained periods of coal feed as follows:

<u>Dates of Sustained Coal Feed, 1982</u>	<u>Cause of Coal Feed Suspension</u>
March 27 - March 24	Questionable Catalyst Inventory
March 31 - April 8	Repair Leak in High Pressure System
April 15 - April 25	Let-down Valve Failures

The report by Ashland Synthetic Fuels, Inc. (ASFI)⁽⁹⁾ covers Run 9 in detail.

We received five hydroclone overflow (HO) samples, SP-213, and two fractionator bottoms (FB) samples, SP-209, from Pilot Plant Run 9 from ASFI. With such an incomplete set of samples, an in-depth analysis of the run is impossible. It is apparent, however, that these samples are somewhat different from the "near steady-state" recycle oils produced in PDU Run 10, also made with Wyodak coal in the syncrude mode. The data concerning PDU Run 10 are contained in another report under this contract.⁽³⁾

Component distributions of the hydroclone overflow samples are given in Table 47. The higher IOM/Ash ratios observed for the first two samples compared to the last two reflect the lower conversions reported by ASFI (ca. 85 vs ca. 90%)⁽⁹⁾ for those days.

The compositions of the total recycle oil calculated from ASFI's flow rates⁽⁹⁾ and our hydroclone overflow component distributions are given in Table 48. It can be seen that these samples were not taken from periods of steady operations.

The data in Table 48 also show that the solvent/coal ratio during the periods of Pilot Plant Run 9 which we have studied was considerably higher than during near steady state operations in PDU 10. This indicates that these samples are not directly comparable to PDU 10 because of differing run conditions at the time of sampling.

Solvent fractionation analyses of the hydroclone overflow soluble resid are shown in Table 49. Generally, there is a trend of decreasing oils and increasing preasphaltenes with time, presumably reflecting catalyst deactivation. Because of the low preasphaltene content, the product resid from the early part of the run could not be flaked at the Pilot Plant. Flaking was successful only after April 21, 1982, presumably because catalyst deactivation had caused a greater production of preasphaltenes. The resid samples we obtained were found to be tar-like and non-friable.

The soluble resids we have from Pilot Plant Run 9 are of much higher quality (more oils, less preasphaltenes) than those made at near steady state in PDU 10. Resid quality in PDU Run 10 decreased continuously over the first twenty-eight days and then levelled-off. Most likely, the quality of the Pilot Plant Run 9 resids are so different from the PDU10 "steady-stage" resids because these pilot plant samples were taken prior to equilibrium.

Proton distributions of the hydroclone overflow distillates are given in Table 50. The Pilot Plant Run 9 samples we analyzed contain considerably more donors (cyclic and protons) and significantly fewer paraffins (alkyl and protons) than the "near steady-state" PDU Run 10 samples. This indicates that the pilot plant distillates are the better donor solvents. The pilot plant samples also have a greater ratio of condensed/uncondensed aromatics indicating that they are better physical solvents and more amenable to further hydrogenation without over-hydrogenation.

As was demonstrated above, the pilot plant was not at steady state throughout the time these samples were taken. As the recycle oils evolved during Pilot Plant Run 9, they approached the composition of the near steady-state PDU

10 samples. The proton distributions of the hydroclone overflow distillates show this trend as they become more aromatic, more paraffinic and as donor concentrations decrease with run period, presumably because of catalyst deactivation. PDU Run 10 oils evolved in a similar manner.

The two fractionator bottoms samples we analyzed are certainly not equilibrium samples. Run 9 used hydrogenated U.S. Steel creosote oil as a start-up solvent. Our two samples were taken five and six days into the run and show evidence that the hydrogenated creosote oil had not yet been replaced with process-derived oil. Hydrogenated creosote oil is less paraffinic, contains more hydroaromatics and is therefore a much better donor solvent than Wyodak coal-derived oils. This difference is evident in the ^1H -NMR data shown in Table 51. As a result, the fractionation bottoms samples were studied no further.

The hydroclone overflow distillate samples were tested in the microautoclave at standard conditions. The results shown in Table 52 confirm the NMR data. The pilot plant samples tested are better donor solvents than the "near steady-state" PDU Run 10 samples. For comparison, H-Coal hydroclone overflow distillates from bituminous coals generally give conversions of 80-85% at these test conditions.⁽³⁾

ANALYTICAL METHODS DEVELOPMENT

Microautoclave Tests At Very Short Residence Times

Several microautoclave runs were made at varying reaction times with Old Ben No. 1 coal and our composite ITSL pasting solvent (Indiana 5 coal). At our simulated SCT conditions (860°F, 5 min, 1/1.8 solvent/coal), many pasting solvents give 90% THF conversion in the microautoclave. Since this is about the maximum obtainable THF conversion with this coal using donor extraction, the microautoclave test cannot always differentiate the solvent quality of various excellent donor solvents. Therefore, we made several runs at very low residence times to see if conversions would decrease to a more meaningful region. The data are shown below.

<u>Residence Time at 860°F</u>	<u>THF Conversion wt % MAF</u>	<u>Gas Make, wt % MAF</u>
0	85.4	≤ 3.7
1	89.4	1.6 - 10.5
3	89.9	2.7 - 8.5
8	89.5	---

Conditions: 860°F, 5 g Old Ben No. 1 coal, 9 g composite pasting solvent,
2 min heat-up time

Only the 0 min run gave a conversion significantly below 90%. The heat-up curves for these runs were all similar: 800°F at 1 min, 845°F at 1.5 min and 860°F at 2 min.

The 0 min residence time microautoclave test may be a reasonable model of the SCT reactor since the conditions are nearly the same. The "slug flow" characteristics of the SCT coil are closely approximated by a rapidly heated/cooled batch reactor. Liquid residence time in the SCT reactor is approximately 2-3 minutes⁽²⁾ at above 600°F with only about half of that at above 800°F. The approximation of the microautoclave to the SCT reactor could be improved by using whole pasting solvent as opposed to distillate only, and by charging the autoclave with 1000 psig cold H₂; but since the coal conversion obtained in the SCT unit is routinely 92% to hot quinoline solubles, a more perfect model would be of no greater use for differentiating solvent quality.

At very short residence time, precision is lost in the autoclave test. Because the only one of these tests that gave significantly below 90% conversion was at such short residence times, we will not routinely use these conditions to test ITSL solvents.

Comparison of Methods for Obtaining Proton Distributions of Resids

Through the course of this contract, two slightly different ¹H-NMR methods have been used to obtain proton distributions on resids. These two methods

and one additional one were used on two samples to determine their equivalence. A short description of the methods is given below.

The one procedure uses pyridine- d_5 with 99.5% deuterium as the solvent. The residual protons in the pyridine (0.5%) give large signals that must be subtracted from the sample spectrum. This is done manually on the chart. Integrations are then performed in triplicate with a planimeter.

The second method is similar to the first but employs pyridine- d_5 with 99.96% deuterium as the solvent. The residual pyridine proton peaks are small enough to ignore and so integrations are performed electronically by the instrument.

For comparison of integration methods, the analyses using the second method were also integrated in triplicate by planimetry, resulting in a third method. All three methods were used on two samples. The data, given below show that the different methods give generally equivalent results.

COMPARISON OF ^1H -NMR METHODS FOR SOLUBLE RESIDS

Sample	Method No.	Proton Distributions			
		Aromatics	Alpha	Beta	Gamma
3LCF8-27F21281	1	42.5	29.8	21.0	6.6
	2	41.2	30.3	21.8	6.7
	3	41.3	31.2	21.7	5.9
3LCF8-49P2282	1	37.7	31.0	25.2	6.2
	2	35.5	29.5	26.9	8.1
	3	36.6	29.5	26.9	7.0

We found that it was necessary to take care in getting an accurate spectral baseline when planimetry was used for integration.

We normally consider proton distribution data to be precise to $\pm 1\%$ absolute. Since the various methods reproduce each other to about this level, we consider the methods essentially equivalent. Most of our future work will

use method 1 above because the cost of the pyridine is significantly reduced compared to the other methods.

Microautoclave Round-Robin Comparison

We participated in a round-robin microautoclave comparison with Oak Ridge National Laboratory (ORNL) and Cities Service at the request of DOE. The round-robin was performed because ORNL consistently obtained surprisingly low conversions with the coal (Kentucky 9) and solvent (3LCF7-92H0) they were using. Our results indicate that the cause of the low conversions experienced by ORNL was a combination of the use of an unreactive or oxidized coal and the use of a relatively poor quality LC-Finer product as a solvent. Complete details of this work were reported to DOE and ORNL.⁽¹⁰⁾

Distillation Round-Robin Program

We also participated in the third and final stage of a round-robin program to develop a standardized distillation procedure for the quantitation of process solvent in coal liquefaction samples. The program was coordinated by Air Products and Chemicals, Inc. (APCI) under DOE program 12.1.10. The other participating laboratories were APCI, Kerr-McGee, Lummus, ORNL and Wilsonville. Conoco was also a participant in the first two stages of the program. We found the distillation procedure tested to be fairly easy and straightforward and to give smooth and reasonably fast distillations. Reproducibility was within 1% absolute. Complete details were reported to DOE and APCI.⁽¹¹⁾

Section 4

EXPERIMENTAL

Most of the experimental techniques used in this work were detailed in the Second Annual Report ⁽¹⁾ for this contract and will not be presented here.

The procedures used in the section "Non-Catalytic Reactions at ITSL Conditions" are presented below.

The experiments used to examine the thermal conversion of pasting solvent resids proceeded as follows. The 30 ml microautoclave was charged with 5.25 g of the pasting-solvent resid and 9.75 g of our composite pasting solvent distillate from the Indiana 5 coal runs. This ratio of distillate and resid is approximately equal to the average composition of pasting solvents. The composite distillate was used to limit the variables to one; namely the resid composition. No hydrogen was used. The reconstituted pasting solvent charge was reacted at 860°F for 5 min total time (ca. 3 min at temperature). The products were filtered through a Whatman 42 paper with freshly distilled tetrahydrofuran (THF). The insolubles were combusted to determine IOM and ash. The THF was removed from the solubles by evaporation and the solubles were distilled in a micro-still to our usual end point (320°C pot/270°C column/5 torr) to give an 850°F⁻ distillate and a THF soluble 850°F⁺ resid fraction.

The experiments used to study the "Thermal Contribution of 850°F⁺ Conversion in the LC-Finer" were carried out as follows. The 30 ml microautoclave was charged with 9 g of LC-Finer feed resid (850°F⁺) from Run 3LCF7-48F6381 and 6 g of our composite LC-Finer product distillate (850°F⁻), both produced from Indiana 5 coal. The vessel was charged with 1000 psig cold H₂ (<2300 psig @ 780°F). Residence times were 1.0, 1.5 and 2.0 hr at 780°F. LC-Finer residence times are proprietary information but are believed to be on the order of two hours. The feed resid used was chosen because it has approximately the average composition of LC-Finer feed resids from Run 3LCF7.

The resid-to-distillate charge ratio used in the microautoclave tests is a typical value for Run 3LCF7.⁽³⁾ The reaction temperature of 780°F used in the microautoclave was the control temperature for most of Run 3LCF7 though the effective temperature was normally 750–760°F. The LC-Finer is generally run at 2700 psig total pressure; therefore, the hydrogen partial pressure should be close to the initial hydrogen partial pressure used in the microautoclave experiments.

COMPOSITION OF 3LCF7-48F6381 RESID

<u>Component</u>	<u>wt %</u>	
Oils	27.3	} 84.1
Asphaltenes	17.0	
Preasphaltenes	39.8	
IOM	11.1	
Ash	4.8	

An LC-Finer product distillate was used in the microautoclave charge because it contains a higher concentration of donor hydrogen than a donor-depleted feed distillate. The donor hydrogen in the LC-Finer inventory must promote whatever thermal conversion occurs and the LC-Finer product distillate better models the donor hydrogen content of the LC-Finer distillate inventory since the reactor is back-mixed.

After each microautoclave run, the microautoclave contents were extracted with THF to separate the IOM and ash. The solubles were distilled to 320°C pot/270°C column/5 torr (850°F) using a microstill. The soluble product resids were analyzed by LCF solvent fractionation. Proton distributions were determined on the feed and product soluble resids and distillates. Conversions and yields were determined from known feed and product compositions. Ash was assumed to carry through unchanged. 850°F⁻ was determined by difference and includes both distillate and total gas make.

Section 5

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APPENDIX 1

Calculation of First-Stage THF Conversions and Yields

We define THF conversion as the amount of MAF coal plus insoluble organic matter (IOM) fed that is converted into THF-soluble material. It is usually expressed as a weight percent of MAF coal fed. To calculate this, it is necessary to know the amount of MAF coal fed, the amount of IOM fed in the pasting solvent, and the amount of IOM in the total product.

The amount of MAF coal fed is calculated from the solvent to MF coal ratio (1.6/1 = 2.6/1 total feed/MF coal) and the ash content of the coal (10.55% MF for Burning Star, Illinois 6 coal).

The amount of IOM fed is calculated from the IOM content of the pasting solvent (Table 2) and the solvent/MF coal ratio (1.6/1 = 2.6/1.6 total feed/solvent).

The amount of IOM in the total product is calculated from the IOM content of the heavy oil product (Table 3) and the ratio of heavy oil product to feed (0.967, average from Lummus). Conversion is then calculated as:

$$\frac{\text{MAF Coal (IN)} + \text{IOM (IN)} - \text{IOM (OUT)}}{\text{MAF Coal (IN)}} \times 100\%$$

Ash balances, ash (OUT)/ash (IN), are calculated in a related manner. Data are shown in Table 11.

Ash balances for several run periods were much poorer than the analytical precision. Lummus also experiences the same problem and attributes it to solids settling prior to sampling. Table 12 shows conversions and yields for only those periods with ash balances of $100 \pm 15\%$.

850°F⁻ distillate yields and yields of the solvent fractions are calculated in a related manner as follows.

$$\frac{\text{Total 850°F}^+ \text{ (IN)} - \text{Total 850°F}^+ \text{ (OUT)}}{\text{MAF Coal (IN)}} \times 100$$

APPENDIX 2

Calculations of LC-Finer Conversions

Conversions of 850°F⁺ resid in the LC-Finer were calculated as follows: LC-Finer (heavy oil) product data are adjusted to a total product basis by applying the ratio of heavy oil to feed for that period as supplied by Lummus (Table 20). When the ratios were applied, ash balances were generally very good. For the run periods studied, the average value of (% ash IN - % ash OUT) equalled +0.1% ± 0.4. This indicates reliable data. For conversion calculations, 850°F⁺ material is defined as the sum of the THF soluble resid plus IOM. 850°F⁺ conversion, defined as

$$850^{\circ}\text{F}^{+} \text{ Conversion} = \frac{850^{\circ}\text{F}^{+}(\text{IN}) - 850^{\circ}\text{F}^{+}(\text{OUT})}{850^{\circ}\text{F}^{+}(\text{IN})} \times 100\%$$

represents the portion of residual organic matter converted to distillate and gas (including hetero-gases). By including solvent fractionation data in the calculations, conversions were calculated for oils, asphaltenes and the sums oils-plus-asphaltenes and preasphaltenes-plus-IOM in a like manner. Calculated conversion data are listed in Table 31 and plotted in Figure 2. Negative conversion values indicate that more of the material in question was produced than was converted to other products.

TABLE 1

SHORT CONTACT TIME RUN CONDITIONS
ITSL OPERATIONS WITH ILLINOIS 6 COAL

Run Period	Temp., °F		MF Coal Concentration, wt % (nom)	Total Preheater Pressure, psig		Coal Space Rate, lb MF/hr-ft ³	Gas Rate, SCFH
	Coil Outlet	Soaker		Inlet	Outlet		
2SCT12-0418	840	-	36	2010	2000	182	165
0420	839	-	36	2010	2000	163	165
0531	840	-	36	2000	2000	146	129
0629	840	-	36	2000	2000	146	129
0701	836	-	36	2020	2010	172	95
0702	841	-	36	2010	2010	171	95
2SCT13-0708	840	-	30	2020	2020	173	129
0712	842	-	30	2040	2030	141	52
2SCT14-0916	840	-	0	-	2000	170(a)	70
0917A	800	-	0	-	2000	170(a)	70
0917B	800	-	0	-	2000	170(a)	70
2SCT15-1014	830	-	30	2030	2030	82	70
2SCT16-1122	840	-	33	~2060	~2050	~158	158
1212	840	-	33	2060	2050	158	158
2SCT17-0123	320	820	36	2080	2050	84(b)	106
0124	320	820	36	2080	2050	89(b)	106
0127	318	819	36	1080	1080	89(b)	106
0129	320	821	36	1080	1080	89(b)	106
0210	320	822	36	1100	1090	84(b)	106
0214A	800	804	36	1080	1040	85(b)	106
0214B	800	805	36	2080	2040	80(b)	106
2SCT18-0223	837	-	36	2060	2040	158	106
0225	840	-	36	2050	2040	178	106

(a) Run 2SCT14 was made with undashed LC₃ Finer product as feed (no coal). The residence time for this run was equivalent to 170 lb MF/hr-ft³ using a 36% MF coal feed.

(b) Calculated using both coil and soaker reactor volumes.

/ls

TABLE 2

COMPONENT DISTRIBUTIONS OF SCT PASTING SOLVENTS
 Illinois 6 Coal Runs

Sample	Wt % of Total Sample			
	850°F ⁻ Distillate	THF Soluble Resid	IOM	Ash
2SCT12-0418PS	59.7	39.2	0.5	0.3
0420PS	59.9	38.8	0.5	0.3
0531PS	62.0	32.4	2.1	3.0
0629PS	58.8	35.9	2.2	2.7
0701PS	53.2	37.9	4.3	4.0
0702PS	49.7	42.1	3.5	4.0
2SCT13-0708PS	58.1	35.6	3.2	2.2
0712PS	52.8	40.7	4.4	1.8
2SCT14-0916PS	45.7	42.8	5.1	5.9
0917BPS	46.2	41.9	5.7	5.7
2SCT15-1014PS	42.3	50.7	4.2	2.5
2SCT16-1122PS	41.2	54.8	2.5	1.2
1212PS	45.6	49.0	3.1	2.0
2SCT17-0123PS	57.6	38.4	2.0	1.1
0124PS	60.6	37.0	0.7	0.6
0127PS	61.7	36.5	0.7	0.6
0129PS	62.5	36.2	0.6	0.5
0210PS	54.2	43.8	0.9	0.5
0214PS	50.2	47.5	1.2	0.5
2SCT18-0225PS	62.1	34.8	1.9	0.9
0225PS	61.2	37.0	1.2	0.3

/Is

TABLE 3
COMPONENT DISTRIBUTIONS OF SCT HEAVY OIL PRODUCTS
Illinois 6 Coal Runs

Sample	Wt % of Total Sample			
	850°F ⁻ Distillate	THF Soluble Resid	IOM	Ash
2SCT12-0418HO	40.1	49.8	5.8	4.0
0420HO	39.4	47.6	7.4	5.0
0531HO	41.4	45.8	6.6	5.6
0629HO(a)	50.1	38.7	2.3	2.6
0701HO	35.6	18.9	7.7	6.7
0702HO(a)	48.2	44.0	3.0	4.5
2SCT13-0708HO	38.6	48.3	6.9	4.8
0712HO	44.5	44.4	6.5	4.3
2SCT14-0916HO	45.2	44.6	4.1	5.4
0917AHO	45.1	43.5	4.8	5.7
0917BHO	47.4	42.1	4.5	5.6
2SCT15-1014HO(a)	46.3	47.9	3.4	2.0
2SCT16-1122HO	37.6	49.1	9.4	3.6
1212HO	34.6	51.1	8.2	5.7
2SCT17-0123HO	42.9	42.9	7.1	6.6
0124HO	43.9	46.4	4.9	4.4
0127HO	48.2	42.0	5.0	4.3
0129HO	46.7	43.7	5.0	4.1
0210HO	41.2	46.6	6.8	4.9
0214AHO	37.6	50.1	7.8	4.2
0214BHO	34.2	55.7	5.3	4.4
2SCT18-0223HO	38.4	49.4	7.2	4.1
0225HO	41.1	44.7	8.5	5.2

(a) Sample is suspect; we believe it to be pasting solvent.

/ls

TABLE 4

SOLVENT FRACTIONATION OF SCT PASTING SOLVENT
THF-SOLUBLE RESIDS
Illinois 6 Coal Runs

Sample	Wt % of Soluble Resid		
	Oils	Asphaltenes	Preasphaltenes
2SCT12-0418PS	55.0	22.1	22.9
0420PS	55.9	21.8	22.3
0531PS	56.1	23.7	20.2
0629PS	51.4	25.3	23.4
0701PS	50.3	26.2	23.5
0702PS	53.3	23.6	23.1
2SCT13-0708PS	51.1	26.7	22.3
0712PS	53.0	23.1	23.9
2SCT14-0916PS	51.6	26.7	21.7
0917BPS	47.9	27.6	24.5
2SCT15-1014PS	47.7	26.7	25.6
2SCT16-1122PS	50.7	25.2	24.1
1212PS	50.8	26.9	22.3
2SCT17-0123PS	54.1	25.2	20.7
0124PS	54.2	24.2	21.5
0127PS	51.5	24.9	23.6
0129PS	52.7	24.8	22.5
0210PS	53.9	24.8	21.3
0214PS	50.0	25.3	24.7
2SCT18-0223PS	49.1	24.7	26.2
0225PS	48.0	26.8	25.2

/Is

TABLE 5

SOLVENT FRACTIONATION OF SCT HEAVY OIL PRODUCT
THF-SOLUBLE RESIDS
Illinois 6 Coal Runs

Sample	Wt % of Soluble Resid		
	Oils	Asphaltenes	Preasphaltenes
2SCT12-0418HO	41.9	22.7	35.3
0420HO	42.6	23.1	34.3
0531HO	39.4	24.7	35.8
0629HO(a)	49.9	25.4	24.6
0701HO	41.7	24.4	33.9
0702HO(a)	52.6	24.9	22.6
2SCT13-0708HO	45.1	21.5	33.4
2SCT13-0712HO	40.7	24.0	35.3
2SCT14-0916HO	51.3	27.7	21.1
0917AHO	55.7	25.2	19.2
0917BHO	51.7	27.9	20.4
2SCT15-1014HO(a)	51.2	25.1	23.7
2SCT16-1122HO	47.0	26.1	26.9
1212HO	43.2	25.6	31.1
2SCT17-0123HO	45.5	27.1	27.4
0124HO	45.7	26.5	27.8
0127HO	41.8	26.5	31.7
0129HO	41.3	26.4	32.3
0210HO	44.3	26.3	29.4
0214AHO	40.7	26.2	33.1
0214BHO	44.9	26.2	28.9
2SCT18-0223HO	40.0	24.9	35.1
0225HO	44.4	26.0	29.6

(a) Sample is suspect; we believe it to be a pasting solvent.

/ls

TABLE 6
PROTON DISTRIBUTIONS OF SCT PASTING SOLVENT DISTILLATES
Illinois 6 Coal Runs

Sample	Proton Distributions						
	Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
2SCT12-0418PS	19.7	7.0	16.5	9.2	20.2	16.6	10.8
0420PS	20.5	6.3	17.0	9.0	20.2	17.0	9.9
0531PS	22.5	6.7	17.2	9.2	19.0	16.1	9.4
0629PS	21.1	6.7	17.4	8.8	19.9	16.2	9.9
0701PS	20.5	5.9	17.9	8.8	20.4	16.6	9.8
0702PS	19.9	5.9	17.1	9.1	20.6	17.0	10.3
2SCT13-0708PS	26.4	5.5	16.7	8.6	18.8	14.8	9.2
0712PS	30.4	5.9	16.3	8.3	16.6	14.1	8.5
2SCT14-0916PS	20.0	6.5	17.5	8.5	20.5	16.6	10.3
0917B-PS	21.1	6.2	17.9	8.5	20.0	16.5	9.9
2SCT15-1014PS	28.4	6.7	17.8	8.1	17.0	13.7	8.3
2SCT16-1122PS	30.3	7.8	16.2	7.9	16.5	12.8	8.5
1212PS	30.6	7.6	17.0	8.0	16.8	12.2	7.8
2SCT17-0123PS	28.4	6.7	16.6	8.0	17.8	14.0	8.6
0124PS	26.7	7.0	17.1	8.2	18.3	14.0	8.7
0127PS	28.7	6.2	17.3	8.4	17.7	13.5	8.1
0129PS	26.4	6.5	17.5	8.5	18.6	13.9	8.5
0210PS	26.2	6.0	16.0	8.7	17.7	14.8	9.9
0214PS	27.0	6.4	16.1	8.6	17.6	14.8	9.5
2SCT18-0223PS	31.2	4.7	18.6	8.0	17.9	12.6	7.1
0225PS	24.5	5.1	17.9	8.8	19.4	15.2	9.1

/ls

TABLE 7

PROTON DISTRIBUTIONS OF SCT HEAVY OIL PRODUCT DISTILLATES
Illinois 6 Coal Runs

Sample	Proton Distributions						
	Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
2SCT12-0418HO	21.8	8.1	14.2	9.9	17.4	16.8	11.6
0420HO	23.9	6.2	16.3	9.1	18.3	16.4	9.9
0531HO	26.7	6.5	16.1	9.6	16.7	15.3	9.1
0629HO(a)	21.2	6.9	17.3	9.0	19.7	16.4	9.6
0701HO	25.0	6.1	16.0	9.4	17.0	16.1	9.6
0702HO(a)	16.0	5.7	18.9	8.7	22.6	17.9	10.2
2SCT13-0708HO	26.3	6.2	16.0	9.0	18.2	15.3	9.1
0712HO	30.8	7.0	15.4	9.0	14.9	14.2	8.7
2SCT14-0916HO	24.7	6.7	16.9	8.6	18.1	15.2	9.9
0917A-HO	23.6	6.2	17.5	8.3	19.0	15.7	9.7
0917B-HO	23.1	6.2	17.4	8.5	18.9	16.2	9.8
2SCT15-1014HO(a)	36.8	5.1	16.2	7.9	15.0	12.1	6.8
2SCT16-1122HO	40.0	8.2	14.5	8.5	11.5	10.6	6.8
1212HO	33.0	8.2	15.6	8.5	14.4	12.4	7.9
2SCT17-0123HO	34.5	7.6	14.7	9.1	12.9	12.9	8.3
0124HO	32.7	7.7	16.4	8.8	13.0	13.2	8.3
0127HO	35.8	6.2	15.5	9.0	12.9	12.9	7.7
0129HO	34.1	6.9	14.9	9.4	13.5	13.2	8.2
0210HO	36.3	5.9	15.1	9.4	12.2	13.1	8.0
0214A-HO	32.8	7.5	14.2	9.2	13.3	13.9	9.0
0214B-HO	32.5	6.5	15.4	9.5	13.5	14.1	8.5
2SCT18-0223HO	37.3	6.5	15.1	8.3	12.9	12.8	7.1
0225HO	30.7	7.4	13.9	9.4	14.3	14.5	9.8

(a) Sample is suspect; we believe it to be pasting solvent.

/Is

TABLE 8
PROTON DISTRIBUTIONS OF PYRIDINE-SOLUBLE SCT RESIDS
Illinois 6 Coal Runs

<u>Sample</u>	<u>Proton Distribution</u>			
	<u>Aromatic</u>	<u>Alpha</u>	<u>Beta</u>	<u>Gamma</u>
<u>Pasting Solvents</u>				
2SCT12-0418PS	36.8	27.7	27.4	8.0
0701PS	40.9	28.2	23.5	7.4
2SCT13-0708PS	47.8	25.5	20.3	6.3
2SCT14-0916PS	41.4	29.7	22.5	6.4
0917B-PS	42.1	29.5	22.6	5.8
2SCT15-1014PS	45.6	29.2	19.6	5.7
2SCT16-1212PS	48.8	28.0	18.7	4.5
2SCT17-0127PS	46.1	28.8	20.1	5.0
0214PS	46.8	29.4	18.9	5.0
<u>Heavy Oil Products</u>				
2SCT12-0418HO	41.2	30.0	22.3	6.5
0701HO	44.5	29.7	19.9	5.9
2SCT13-0708HO	45.8	28.7	20.0	5.5
2SCT14-0916HO	43.1	28.6	21.9	6.3
0917A-HO	42.1	29.1	22.3	6.5
0917B-HO	40.9	30.2	22.7	6.1
2SCT15-1014HO(a)	49.5	26.2	18.9	5.4
2SCT16-1212HO	47.6	29.3	18.0	5.1
2SCT17-0127HO	47.9	30.2	17.3	4.6
0214A-HO	48.7	29.1	17.5	4.7
0214B-HO	47.4	29.7	17.8	5.1

(a) Sample is suspect; we believe it to be a pasting solvent.

Samples dissolved in pyridine-d₅ (99.5%), integrated by planimetry.

/ls

TABLE 9
PHENOLIC -OH CONCENTRATIONS OF SCT DISTILLATES
Illinois 6 Coal Runs

<u>Sample</u>	Phenolic -OH Concentration, meq/g	
	<u>Pasting Solvent</u>	<u>H.O. Product</u>
2SCT12-0418	0.16	0.49
0420	0.16	0.52
0531	0.19	0.49
0629	0.19	0.20(a)
0701	0.16	0.55
0702	0.17	0.10(a)
2SCT13-0712	0.13	0.48
2SCT14-0916	0.13	0.17
0917A	----	0.14
0917B	0.17	0.10
2SCT15-1014	0.25	0.12(a)
2SCT16-1212	0.15	0.56
2SCT17-0124	0.13	0.65
0214	0.22	----
0214A	----	0.64
0214B	----	0.77
2SCT18-0225	0.22	0.54
Avg SCT Coal Period	0.18 ± 0.04	0.52 ± 0.03
Avg Soaker Period	0.18 ± 0.06	0.69 ± 0.07

(a) Sample is suspect; we believe it to be a past-
ing solvent.

/ls

TABLE 10

MICROAUTOCLAVE TESTS WITH SCT DISTILLATES
MODIFIED EQUILIBRIUM CONDITIONS
Illinois 6 Coal Runs

Sample	THF Conversion, wt % MAF Coal	
	Pasting Solvent	H.O. Product
2SCT12-0418	90.3	85.4
0420	90.8	87.2
0531	90.3	85.9
0629	89.6	89.5(a)
0701	89.3	84.4
0702	90.1	----
2SCT13-0708	88.7	85.2
0712	87.4	80.8
2SCT14-0916	90.9	90.2
0917A	----	90.3
0917B	90.4	90.9
2SCT15-1014	87.5	87.9(a)
2SCT16-1122	88.8	72.9
1212	89.8	83.2
2SCT17-0123	89.3	73.0
0124	92.4	77.8
0127	91.8	75.4
0129	91.6	75.6
0210	87.7	69.4
0214	88.9	----
0214A	----	74.6
0214B	----	81.4
2SCT18-0223	89.8	71.1
0225	89.9	78.6

Avg $\pm$ Std Dev 89.8 $\pm$ 1.3

Conditions: 9 g solvent, 6 g coal, 750°F, 30 min

Coal: Old Ben No. 1, Indiana 5

(a) Sample is suspect; we believe it to be a past-
ing solvent

/ls

TABLE 11

CALCULATED THF CONVERSIONS AND YIELDS
SCT RUN PERIODS
Illinois 6 Coal Runs

Period	THF Conversion	Yields, wt % MAF Cca				Ash Balance
		Total 850°F ⁻	Oils	Asphaltenes	Preasphaltenes	
2SCT12-0418	83.6	12.3	19.8	16.7	34.9	96.9
0420	78.8	10.3	17.8	16.2	31.8	121.2
0531	84.4	12.2	18.1	18.7	36.2	94.7
0701	85.4	13.5	23.3	16.1	32.1	101.9
2SCT13-0708	83.5	2.5	31.0	12.6	37.4	98.7
0712	88.1	35.2	8.8	13.9	31.1	94.0
2SCT14-0916(a)	2.3	3.0	0.1	1.0	-0.4	88.5
0917A(a,b)	1.5	2.7	4.6	-1.8	-3.4	95.0
0917B(a)	2.7	5.6	1.9	-0.4	-3.9	95.0
2SCT16-1122	74.9	41.1	12.5	10.6	13.3	81.2
1122	80.2	21.7	15.8	12.9	27.3	114.3
2SCT17-C123	82.7	24.3	17.3	15.7	19.5	141.8
C124	86.7	20.7	23.8	19.1	22.9	101.7
0127	86.4	32.9	14.7	15.2	23.6	99.4
0129	86.2	27.4	16.3	16.8	26.2	96.3
0210	81.4	26.6	15.1	15.2	22.6	115.1
0214A	79.0	23.1	14.0	15.5	26.5	98.6
0214B	86.5	13.2	27.9	19.9	25.0	103.3
2SCT18-C223	82.2	4.2	25.4	19.9	33.9	90.6
C225	76.9	12.9	24.3	15.2	21.2	126.0

(a) No coal in feed for Run 2SCT14, conversion and distillate yields prorated to a 36% MF coal feed.

(b) No pasting solvent sample received, average PS of 0916 and 0917B used in calculation.

/ls

TABLE 12

CALCULATED THF CONVERSIONS AND YIELDS
SCT COAL PERIODS WITH ASH BALANCES OF 100 $\pm$ 15%
Illinois 6 Coal Runs

Period	THF Conversion	Yields, wt % MAF Coal				Ash Balance
		Total 850°F	Oils	Asphaltenes	Preasphaltenes	
2SCT12-0418	83.6	12.3	19.8	16.7	34.9	96.9
0531	84.4	12.2	18.1	18.7	36.2	94.7
0701	85.4	13.5	23.3	16.1	32.1	101.9
2SCT13-0708	83.5	2.5	31.0	12.6	37.4	98.7
0712	88.1	35.2	8.8	13.9	31.1	94.0
2SCT16-1212	80.2	21.7	15.8	12.9	27.3	114.3
2SCT17-0124	86.7	20.7	23.8	19.1	22.9	101.7
0127	86.4	32.9	14.7	15.2	23.6	99.4
0129	86.2	27.4	16.3	16.8	26.2	96.3
0210	81.4	26.6	15.1	15.2	22.6	115.1
0214A	79.0	23.1	14.0	15.5	26.5	98.6
0214B	86.5	13.2	27.9	19.9	25.0	103.3
2SCT18-0223	82.2	4.2	25.4	19.9	33.9	90.6
Avg SCT Period	84 $\pm$ 2	14 $\pm$ 11	20 $\pm$ 7	16 $\pm$ 3	33 $\pm$ 3	
Avg Soaker Period	84 $\pm$ 3	24 $\pm$ 7	19 $\pm$ 6	17 $\pm$ 2	24 $\pm$ 2	

/ls

TABLE 13
FEED SOURCES
ANTISOLVENT DEASHER RUNS
Illinois 6 Coal Runs

<u>Run - Mo/Yr</u>	<u>Feed Source</u>
ASDA154-0582	Short Contact Time Product
ASDA155-0682	Short Contact Time Product
ASDA157-1082	LC-Finer Product
ASDA158-1082	LC-Finer Product
ASDA159-1182	LC-Finer Product
ASDA160-1182	LC-Finer Product
ASDA164	Soaker Product
ASDA165	Short Contact Time Product
3ASDA100-0383	Soaker Product, Wyodak Coal

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TABLE 14
COMPONENT DISTRIBUTIONS - ASDA SAMPLES
Illinois 6 Coal Runs

Sample	wt % of Sample			
	850°F ⁻ Distillate	THF Soluble Resid	IOM	Ash
ASDA154-F0582	44.8	45.1	5.0	4.8
O	35.7	53.7	5.4	4.6
U	41.3	45.3	6.6	6.3
ASDA155-F0682	42.0	47.6	5.4	4.5
O	51.4	47.8	0.2	0.1
U	35.3	16.6	32.7	14.7
ASDA157-F1082	55.4	34.2	5.0	4.8
O	51.1	48.3	0.1	0.1
U	49.8	42.5	3.9	3.4
ASDA158-F1082	48.8	43.3	3.6	4.0
O	39.4	59.2	0.5	0.6
U	31.7 (a)	30.2	20.2	17.4
ASDA159-F1182	59.7	31.7	4.1	3.8
O	50.8	48.3	0.1	0.1
U	37.8	21.6	22.6	17.4
ASDA160-F1182	58.9	35.0	2.8	2.8
O	58.9	40.3	0.1	0.1
U	38.0	14.9	27.4	18.4
C	33.0 (b)	11.2	33.8	21.0
ASDA164-F	44.5	53.6	0.9	0.5
O	43.0	55.6	<0.1	<0.1
U	26.8	25.5	24.8	22.3
ASDA165-F	40.3	45.7	8.9	4.3
O	34.7	61.7	2.7	0.2
U	32.6	31.2	22.9	12.7
3ASDA100-F0383 (c)	60.6	25.4	8.9	4.5
O	66.4	32.9	0.1	<0.1
U	43.6	20.0	24.2	11.4

(a) Includes 0.3% H₂O, absolute

(b) Includes 0.4% H₂O, absolute

(c) Wyodak Coal Run²

/Is

TABLE 15
SOLVENT FRACTIONATION OF THF-SOLUBLE ASDA RESIDS
Illinois 6 Coal Runs

Sample	wt % of THF-Soluble Resid		
	Oils	Asphaltenes	Preasphaltenes
ASDA154-F0582	46.2	24.0	29.8
O	44.5	24.7	30.8
U	42.1	25.9	32.0
ASDA155-F0682	45.8	24.0	30.2
O	41.9	25.3	32.8
U	38.6	25.4	36.0
ASDA157-F1082	56.1	25.6	18.3
O	57.4	23.1	19.4
U	58.3	22.9	18.8
ASDA158-F1082	53.8	28.4	17.8
O	56.4	28.5	15.4
U	54.0	27.6	18.4
ASDA159-F1182	53.3	29.2	17.5
O	54.2	29.2	16.5
U	52.8	27.5	19.7
ASDA160-F1182	52.5	29.5	18.2
O	54.8	29.9	15.3
U	52.6	26.7	20.7
C	55.8	23.8	20.4
ASDA164-F	47.6	27.8	24.6
O	44.8	25.8	29.3
U	47.6	23.3	27.1
ASDA165-F	41.0	24.3	34.7
O	40.8	26.8	32.4
U	39.7	25.5	34.8
3ASDA100-F0383(a)	69.3	12.7	18.0
O	74.8	13.4	11.7
U	65.1	12.4	22.5

(a) Wyodak coal run

/Is

TABLE 16

SOLVENT FRACTIONATION OF PYRIDINE-SOLUBLE ASDA RESIDS
Illinois 6 Coal Runs

Sample	wt % of Pyridine-Soluble Resid		
	Oils	Asphaltenes	Preasphaltenes
ASDA154-F0582	46.9	19.4	33.7
O	49.7	20.0	30.3
U	39.3	20.5	40.2
ASDA155-F0682	39.3	20.9	39.8
O	38.9	22.6	38.5
U	27.9	15.2	56.8
ASDA157-F1082	50.8	25.1	24.1
O	53.0	23.8	23.2
U	52.2	21.5	26.3
ASDA158-F1082	50.2	31.5	18.2
O	51.9	28.7	19.4
U	41.5	27.2	31.3
ASDA159-F1182	50.2	29.2	20.6
O	49.5	30.4	20.2
U	43.5	24.9	31.6
ASDA160-F1182	52.3	27.8	19.9
O	53.5	29.4	17.1
U	46.2	22.6	31.3
C	49.0	19.7	31.2
ASDA164-F	39.7	23.6	36.7
O	42.9	23.0	34.1
U	33.4	15.2	51.4
ASDA165-F	34.2	20.1	45.7
O	37.4	22.4	40.2
U	31.3	19.1	49.6
3ASDA100-F0383(a)	58.8	13.1	28.1
O	68.6	15.1	16.3
U	46.2	9.0	44.8

(a) Wyodak coal run

/ls

TABLE 17
PROTON DISTRIBUTIONS OF ANTISOLVENT DEASHER DISTILLATES
Illinois 6 Coal Runs

Sample	Proton Distributions						
	Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
ASDA154-F0582	30.3	7.2	16.1	8.9	15.5	13.5	8.5
O	23.5	8.3	14.8	11.5	16.3	15.4	10.2
U	23.3	8.4	14.7	11.6	16.2	15.6	10.1
ASDA155-F0682	30.0	6.9	15.9	8.7	15.7	14.0	8.9
O	27.0	7.5	16.6	11.0	16.2	13.7	8.0
U	26.5	8.1	15.6	11.9	15.7	13.7	8.4
ASDA157-F1082	25.3	7.1	15.9	8.3	18.7	15.2	9.4
O	32.8	7.5	14.1	8.1	14.2	13.6	9.7
U	30.5	7.5	15.1	9.3	15.2	14.0	8.5
ASDA158-F1082	18.5	7.5	16.3	8.3	20.7	17.3	11.4
O	23.3	7.2	17.3	8.4	17.8	15.8	10.3
U	19.3	6.6	15.6	8.4	18.9	18.1	12.9
ASDA159-F1182	20.7	7.7	16.1	8.0	20.1	16.4	11.0
O	27.5	7.6	17.5	8.1	16.6	13.8	8.9
U	19.9	6.8	14.9	8.4	18.5	18.0	13.4
ASDA160-F1182	20.0	7.1	17.6	8.0	21.4	15.8	10.1
O	24.1	7.6	17.9	8.8	18.6	14.0	8.9
U	18.6	7.4	15.0	10.9	17.5	18.3	12.4
C	14.2	7.3	12.1	11.9	16.3	22.4	15.9
ASDA164-F	33.2	7.4	15.5	8.9	14.0	12.9	8.1
O	32.6	7.6	14.9	11.4	12.4	13.0	8.1
U	29.9	8.7	14.5	13.0	12.0	13.3	8.5
ASDA165-F	32.6	8.8	14.4	8.9	14.0	13.6	9.7
O	33.2	8.0	14.9	10.6	12.3	12.7	8.3
U	28.4	8.7	13.4	13.3	12.4	14.4	9.4
3ASDA100-F0383(a)	31.7	8.0	15.5	9.3	14.0	13.6	8.0
O	38.0	6.9	15.0	8.5	11.7	12.4	7.4
U	31.0	7.6	14.0	9.2	12.5	15.5	10.2

(a) Wyodak coal run

/Is

TABLE 18

SELECTIVE CONCENTRATION OF PREASPHALTENES
ASDA RESID SAMPLES
Illinois 6 Coal Runs

Run	Preasphaltenes (Underflow)/ Preasphaltenes (overflow)		Deashing Efficiency (a)
	THF-Soluble Basis	Pyridine-Soluble Basis	
ASDA154-0582	1.04	1.33	15
ASDA155-0682	1.10	1.48	98
ASDA157-1082	0.97	1.13	-- (c)
ASDA158-1082	1.19	1.61	88
ASDA159-1182	1.19	1.56	98
ASDA160-1182	1.35	1.83	100
ASDA164	0.92	1.51	80
ASDA165	1.07	1.23	97
3ASDA100-0383(b)	1.92	2.75	100
Average (Ill. 6 coal)	1.10 $\pm$ 0.13	1.46 $\pm$ 0.22	

(a) Deashing Efficiency Calculated as $1 - \frac{\text{Ash (Overflow)}}{\text{Ash (Feed)}} \times 100\%$
based on forced ash balance.

(b) Wyodak Coal Run

(c) Ash balance not possible, feed sample had more ash than both Overflow and Underflow samples.

/Is

TABLE 19
LC-FINER RUN CONDITIONS
Run 3LCF9

Sample Run - Date	Control Temp., °F	Effective Temp., °F	Pressure, psig	WHSV lbs Feed lb cat hr	Relative WHSV of Feed	LHSV, Vol 850°F ⁺ Vol Cat ltr	Catalyst Age, lb 850°F ⁺ lb Catalyst	Feed Source
3LCF9-0321	750	737	2700	1.09	1.67	-	25	Deashed-SCT
0323	750	738	2700	1.09	1.67	0.346	40	Deashed-SCT
0417	750	733	2700	1.15	1.67	0.409	56	Deashed-SCT
0419	750	731	2700	1.12	1.67	-	89	Deashed-SCT
0429	750	719	2700	1.18	1.67	0.408	106	Deashed-SCT
0501	750	725	2700	1.22	1.67	0.448	135	Deashed-SCT
0503	750	725	2700	1.22	1.67	0.462	166	Deashed-SCT
0513	760	738	2700	1.26	1.67	0.472	201	Deashed-SCT
0517	760	739	2700	1.03	1.67	0.388	239	Deashed-SCT
0524	760	741	2700	0.94	1.67	0.366	287	Deashed-SCT
0526	760	743	2700	0.98	1.67	0.377	310	Deashed-SCT
0607	770	744(a)	2700	1.10	1.67	0.430	343	Deashed-SCT
0609	770	741(a)	2700	1.15	1.67	0.476	379	Deashed-SCT
0617	770	745	2700	1.08	1.67	0.402	398	Deashed-SCT
0620	770	746	2700	1.15	1.67	0.411	432	Deashed-SCT
0622	770	746	2700	1.33	1.67	0.487	462	Deashed-SCT
0704	770	739	2700	1.15	1.67	0.417	486	Deashed-SCT
0712	770	737	2700	0.64	1.0	0.223	512	Ashy-SCT
0715	770	743	2700	0.62	1.0	0.220	538	Ashy-SCT
0717	770	741	2700	0.63	1.0	0.227	557	Ashy-SCT
0720	770	741	2700	0.67	1.0	0.228	581	Ashy-SCT
0821	770	732	2700	1.01	2.3	0.607	627	Ashy-SCT
0824	770	736	2700	1.66	2.3	0.590	661	Ashy-SCT
0826	770	733	2700	1.15	1.67	0.421	697	Ashy-SCT
0925	780	746	2700	1.10	1.67	0.270	720	Ashy-SCT
0928	780	746	2700	-	1.67	0.340	754	Ashy-SCT
1001	780	748	2700	1.34	1.67	0.335	785	Ashy-SCT
1005(b)	780	756	2700	-	1.67	0.490	851	Deashed-SCT(b)
1011(b)	780	746	2700	1.15	1.67	0.430	878	Deashed-SCT(b)
1016	780	743	2700	1.21	1.67	0.440	908	Ashy-SCT
1020	780	746	2700	1.22	1.67	0.471	905	Ashy-SCT
1029	780	749	2700	1.07	1.67	0.310	1064	Ashy-SCT
1101	780	746	2700	1.12	1.67	0.332	1102	Ashy-SCT
1109	780	719	2700	1.27	1.67	0.43	1136	Ashy-SCT
1114	780	723	2700	1.79	1.67	0.51	1179	Ashy-SCT
1118	780	728	2700	1.21	1.67	0.29	1228	Ashy-SCT
1121	780	726	2700	1.19	1.67	0.33	1266	Ashy-SCT
1211	770	744	2700	1.14	1.67	0.29	1289	Ashy-SCT
1214	770	748	2700	1.29	1.67	0.49	1340	Ashy-SCT
0110	750	734	2700	1.14	1.67	0.31	1362	Ashy-SCT
0114	750	733	2700	1.24	1.67	0.35	1398	Ashy-SCT
0116	750	733	2700	1.29	1.67	0.42	1401	Ashy-SCT
0118	750	732	2700	1.28	1.67	0.37	1428	Ashy-SCT
0121	750	731	2700	1.13	1.67	0.30	1462	Ashy-SCT
0122	750	730	2700	1.11	1.67	0.34	1474	Ashy-SCT
0129	750	736	2700	1.31	1.67	0.47	1500	Deashed-Soaker
0202	750	736	2700	1.31	1.67	0.51	1547	Deashed-Soaker
0205	750	734	2700	1.28	1.67	0.47	1597	Deashed-Soaker
0208	750	730	2700	1.67	1.67	0.44	1644	Deashed-Soaker
0213	750	729	2700	1.14	1.67	0.40	1699	Deashed-Soaker
0217	750	724	2700	1.17	1.67	0.44	1743	Deashed-Soaker
0220	750	723	2700	0.58	1.0	0.21	1778	Deashed-Soaker
0224	750	717	2700	0.59	1.0	0.21	1802	Deashed-Soaker

(a) Actual effective temperature may be higher.

(b) Feed was deashed SCT product from Indiana 5 coal made for oil agglomeration/liquetfaction tests at Conoco and Gulf.

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TABLE 20

PRODUCT DISTRIBUTIONS FOR RUN 3LCF9

<u>PERIOD</u>	<u>HO/TLP, wt %</u>	<u>TLP/Feed, wt %</u>
3LCF9-0321	94.51	98.87
0323	96.30	98.08
0417	94.88	98.14
0419	95.05	98.23
0429	97.09	98.12
0501	94.39	98.21
0503	96.04	98.06
0513	93.16	97.80
0517	93.71	96.45
0524	95.43	94.47
0526	89.79	96.41
0607	90.91	96.05
0609	93.38	97.17
0617	96.79	97.85
0620	94.32	98.17
0622	94.44	98.31
0704	95.32	97.48
0712	72.08	95.88
0715	75.98	96.01
0717	76.82	95.94
0720	75.85	96.45
0821	92.65	98.51
0824	92.62	98.17
0826	85.81	97.97
0925	82.92	97.42
0928	83.38	98.42
1001	83.09	97.93
1005	83.18	96.97
1011	82.85	97.79
1016	92.96	98.09
1020	93.44	97.96
1029	91.29	96.53
1101	91.76	96.05
1109	94.11	97.79
1114	98.12	98.14
1118	92.09	98.24
1121	92.57	98.38
1211	93.45	99.43
1214	93.08	98.13
0110	93.20	98.41
0114	94.74	99.45
0116	95.45	99.72
0118	94.64	99.34
0121	93.48	99.11
0122	93.41	90.04
0129	95.54	99.13
0203	96.82	----
0206	93.10	98.8
0208/9	93.94	98.7
0213	88.50	99.6
0217	92.4	99.0
0221	83.2	97.8

TLP - Total Liquid Product

HO - Heavy Oil Product, ca. 650°F+

/ls

TABLE 21

COMPONENT DISTRIBUTIONS OF LC-FINER FEEDS
Run 3LCF9

Sample	Wt % of Total Sample			
	850°F ⁻ Distillate	THF Soluble Resid	10M	Ash
3LCF9-0321F	50.9	45.3	2.3	0.8
0323F	49.7	48.1	1.6	0.4
0417F	43.5	46.7	6.6	2.8
0429/30F	47.1	45.6	4.6	2.0
0501F	43.0	53.1	2.3	1.1
0513F	39.2	50.6	5.7	3.9
0524/25F	38.6	49.7	7.0	4.1
0527F	39.2	50.1	6.5	3.4
0617/18F	40.2	46.7	7.8	4.5
0620F	38.5	53.7	5.0	2.4
0704/5F	42.9	50.9	4.2	1.9
0712F	39.3	43.5	10.0	6.8
0715F	40.3	44.7	9.1	5.4
0717F	38.9	46.8	8.6	5.3
0720F	38.8	45.8	9.4	5.2
0821/22F	39.4	44.6	8.9	6.1
0823/24F	39.9	44.5	9.2	5.9
0826F	38.7	44.9	9.6	6.0
0925F	55.6	33.8	6.8	3.2
0928F	53.1	35.8	6.7	3.9
1001F	57.2	31.5	7.1	3.9
1005F	35.6	58.2	3.9	1.7
1011/12F	42.2	52.1	3.8	1.6
1017F	37.1	52.6	6.4	3.2
1020F	36.3	52.1	7.5	3.7
1114F	51.0	34.9	9.3	3.9
1118F	51.7	34.9	8.8	3.9
1121F	52.7	33.1	9.5	4.1
1211F	59.1	35.4	3.5	1.5
1214F	37.3	50.7	7.2	4.4
0111F	52.7	36.9	6.0	4.0
0114F	48.1	40.5	6.5	4.4
0110F	48.8	42.1	5.0	3.6
0121F	53.9	38.5	2.8	4.4
0122F	50.0	41.1	3.7	4.5
0129F	41.3	56.0	1.4	0.7
0203F	40.8	57.1	1.1	0.4
0206F	43.2	55.2	0.4	0.4
0208/9F	47.7	51.0	0.5	0.3
0213F	43.3	55.0	0.7	0.6
0217F	43.2	55.4	0.5	0.2
0221F	42.5	55.2	1.4	0.4

/Is

TABLE 22

COMPONENT DISTRIBUTIONS OF LC-FINER HEAVY OIL PRODUCTS
Run 3LCF9

Sample	Wt % of Total Sample			
	850°F ⁻ Distillate	THF Soluble Resid	10M	Ash
3LCF9-0321P	68.6	29.5	0.6	0.7
0323P	68.5	29.6	0.5	0.6
0417P	64.6	30.2	2.1	2.7
0429/30P	66.1	30.0	1.6	2.1
0501P	61.1	35.9	0.9	1.3
0513P	56.2	34.3	2.6	4.4
0524/25P	55.9	35.6	2.9	4.8
0527P	55.5	35.9	2.9	4.9
0617/18P	54.6	37.0	2.8	4.8
0620P	54.0	41.0	1.5	3.1
0704/5P	57.4	39.0	1.3	1.8
0712P	54.6	33.1	4.2	7.5
0715P	54.1	34.6	4.1	6.8
0717P	54.4	34.5	3.9	6.7
0720P	51.6	36.4	4.1	7.0
0821/22P	47.0	41.1	4.8	6.5
0823/24P	47.9	40.5	4.7	6.4
0826P	48.7	39.9	4.3	6.5
0925P	65.7	27.0	3.5	3.4
0928P	61.8	30.0	3.3	4.4
1001P	62.9	28.0	4.0	4.7
1005P	46.6	49.3	2.0	2.1
1011/12P	49.8	46.3	1.7	1.9
1017P	39.7	51.4	3.6	4.0
1020P	47.8	44.5	3.5	3.9
1114P	59.2	32.4	4.1	3.9
1118P	59.5	32.1	3.2	4.1
1121P	60.9	31.0	3.8	3.8
1211P	66.3	26.7	4.9	1.6
1214P	45.2	46.0	3.3	4.7
0111P	61.0	32.0	2.7	3.8
0114P	59.2	32.6	3.1	4.4
0118P	59.3	33.2	2.7	4.4
0121P	63.1	29.0	2.6	4.6
0122P	61.0	30.9	2.8	4.6
0129P	50.7	47.2	0.6	0.8
0203P	46.2	48.1	3.2	1.8
0206P	49.6	49.4	0.1	0.3
0208/9P	51.9	46.6	0.4	0.3
0213P	46.6	51.7	0.7	0.7
0217P	43.3	55.3	0.6	0.2
0221P	46.2	51.9	1.0	0.5

/Is

TABLE 23
SOLVENT FRACTIONATION OF THF-SOLUBLE LC-FINER FEED RESIDS
Run 3LCF9

Sample	Wt % of THF Soluble Resid		
	Oils	Asphaltenes	Preasphaltenes
3LCF9-0321F	42.9	20.3	36.8
0323F	40.4	19.1	40.5
0417F	48.4	18.1	33.4
0429/30F	42.3	19.2	38.5
0501F	44.2	18.9	37.0
0513F	42.6	18.7	38.6
0524/25F	45.1	19.4	35.5
0527F	42.9	18.8	38.4
0617/18F	44.2	20.0	35.8
0620F	43.3	21.9	34.8
0704/5F	43.9	20.1	36.0
0712F	40.5	21.5	38.0
0715F	43.2	24.6	32.2
0717F	42.9	24.2	32.9
0720F	42.7	22.9	34.4
0821/22F	44.2	24.5	31.3
0823/24F	42.9	21.8	35.3
0826F	48.7	24.8	26.5
0925F	40.3	21.7	38.0
0928F	39.7	24.0	36.3
1001F	41.5	22.8	35.7
1005F	33.2	26.0	40.8
1011/12F	33.0	27.4	39.6
1017F	53.1	23.3	23.6
1020F	49.8	26.3	23.9
1114F	42.0	24.2	33.7
1118F	37.8	22.9	39.3
1121F	39.5	24.3	36.2
1211F	37.7	21.6	40.6
1214F	42.9	23.2	33.8
0111F	30.4	19.3	50.3
0114F	36.4	21.1	42.4
0118F	39.7	22.3	38.0
0121F	43.4	25.5	31.1
0122F	42.2	24.8	33.0
0129F	46.0	23.7	30.3
0203F	42.9	25.6	31.5
0206F	43.8	25.6	30.5
0208/9F	41.0	25.9	33.1
0213F	42.3	25.6	32.1
0217F	40.8	26.7	32.4
0221F	42.8	26.2	31.0
Avg 111. 6 Period			
0417 to 0221	42.6 ± 3.8	22.8 ± 2.5	34.6 ± 4.9
/ls			

TABLE 24

SOLVENT FRACTIONATION OF THF-SOLUBLE
LC-FINER HEAVY OIL PRODUCT RESIDS
Run 3LCF9.

Sample	Wt % of THF Soluble Resid		
	Oils	Asphaltenes	Preasphaltenes
3LCF9-0321P	75.6	14.1	10.3
0323P	73.0	11.4	15.6
0417P	56.8	16.8	26.4
0429/30P	52.8	19.0	28.2
0501P	54.2	18.1	27.7
0513P	52.0	22.4	25.6
0524/25P	52.9	24.2	22.9
0527P	52.3	24.6	22.7
0617/18P	47.9	25.1	26.9
0620P	51.2	29.1	19.7
0704/5P	48.9	24.1	27.0
0712P	57.4	23.8	18.8
0715P	56.7	25.8	17.4
0717P	56.6	28.7	14.7
0720P	54.1	28.2	17.7
0821/22R	51.7	30.1	18.2
0823/24P	51.3	29.5	19.3
0826P	50.8	27.4	21.8
0925P	53.5	27.2	19.3
0928P	54.4	28.1	17.5
1001P	50.2	29.0	20.8
1005P	45.4	32.3	22.3
1011/12P	45.4	30.7	23.9
1017P	55.3	28.2	16.1
1020P	55.8	29.0	15.2
1114P	48.3	30.2	21.4
1118P	53.1	29.0	17.9
1121P	50.3	29.0	20.6
1211P	46.9	29.4	23.8
1214P	52.1	28.5	19.4
0111P	43.9	26.1	29.9
0114P	40.6	28.6	30.8
0118P	44.9	28.8	26.3
0121P	51.6	27.8	20.7
0122P	48.2	29.8	22.0
0129P	47.6	27.8	24.6
0203P	50.2	26.6	23.2
0206P	47.8	28.5	23.8
0208/9P	48.0	27.0	25.0
0213P	52.8	26.0	21.2
0217P	51.1	26.8	22.1
0221P	53.8	27.0	19.2

/Is

TABLE 25

PROTON DISTRIBUTIONS OF LC-FINER FEED DISTILLATES
Run 3LCF9

Sample	Proton Distributions						
	Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
3LCF9-0321F	34.6	5.9	15.1	8.3	14.4	13.0	8.7
0323F	34.1	5.0	15.8	8.2	15.4	13.3	8.1
0417F	35.8	8.3	15.0	8.6	13.2	11.5	7.6
0429/30F	35.0	7.3	15.8	8.7	14.2	11.7	7.3
0501F	30.2	7.5	16.6	8.3	16.4	12.9	8.1
0513F	28.3	7.9	16.0	9.3	16.3	13.5	8.7
0524/25F	29.3	6.8	17.3	8.4	16.6	13.2	8.3
0527F	31.9	6.9	16.5	8.8	14.8	13.1	8.0
0617/18F	32.6	7.4	16.0	8.9	14.5	12.4	8.2
0620F	34.0	6.8	16.1	8.6	14.7	12.6	7.2
0704/5F	33.6	7.3	15.6	8.7	14.2	12.5	8.0
0712F	28.1	7.1	15.8	8.6	16.7	14.5	9.2
0715F	30.2	6.9	15.9	8.5	15.7	14.1	8.7
0717F	29.4	7.6	14.7	9.1	15.5	14.3	9.4
0720F	30.3	6.5	15.4	8.6	15.9	14.1	9.2
0821/22F	25.5	6.6	15.7	8.7	17.2	15.8	10.5
0823/24F	26.8	6.3	16.1	8.2	17.5	15.6	9.6
0826F	26.0	7.1	15.1	8.6	17.0	15.8	10.4
0925F	27.7	8.0	17.5	9.2	17.4	12.5	7.8
0928F	27.5	6.5	18.3	8.7	19.0	12.7	7.3
1001F	26.7	6.6	19.0	8.7	19.1	12.7	7.2
1005F	42.3	7.6	13.7	9.7	9.4	10.7	6.5
1011/12F	40.8	7.8	13.2	15.3	8.0	9.4	5.4
1017F	31.9	6.1	16.3	8.5	16.1	13.0	8.1
1020F	35.4	5.8	15.9	8.7	14.0	12.9	7.4
1114F	26.9	6.9	18.0	8.3	19.8	12.5	7.6
1118F	29.1	7.3	17.3	8.5	18.8	12.3	7.0
1121F	26.6	8.4	16.5	8.7	18.6	13.0	8.1
1211F	24.5	7.0	19.0	8.8	20.8	13.1	6.9
1214F	30.2	7.6	16.3	8.9	16.4	12.6	8.0
0111F	27.6	6.9	18.0	8.8	18.7	12.4	7.6
0114F	26.9	7.4	16.9	9.3	18.9	12.8	7.8
0118F	27.0	7.5	16.3	9.1	18.3	13.5	8.2
0121F	23.4	8.3	16.8	9.8	18.6	14.3	8.8
0122F	24.1	8.0	16.7	9.5	19.0	13.8	8.8
0129F	38.7	7.5	16.1	8.0	12.3	10.5	7.0
0203F	42.6	5.3	15.7	8.3	11.6	10.4	6.1
0206F	32.7	5.3	15.2	15.3	11.9	12.2	7.3
0208/9F	38.6	7.5	14.8	10.4	10.4	11.1	7.2
0213F	29.0	9.1	14.0	13.6	11.5	14.0	8.8
0217F	32.2	8.8	14.2	12.4	11.3	12.9	8.4
0221F	36.9	7.9	13.9	11.7	10.2	11.9	7.5

/Is

TABLE 26

PROTON DISTRIBUTIONS OF LC-FINER HEAVY OIL PRODUCT DISTILLATES
Run 3LCF9

Sample	Proton Distributions						
	Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
3LCF9-0321P	10.4	6.0	17.6	9.7	25.7	18.6	11.9
0323P	10.0	6.8	17.7	9.4	25.4	18.9	11.9
0417P	11.1	7.8	16.8	9.4	24.0	18.9	11.9
0429/30P	10.9	7.3	17.8	10.0	24.4	18.2	11.3
0501P	9.9	7.8	17.0	9.6	24.7	18.5	12.5
0513P	9.7	7.8	15.2	8.9	23.8	20.8	13.8
0524/25P	9.7	6.8	14.7	8.5	23.9	21.5	14.8
0527P	11.6	7.3	15.8	8.8	22.1	20.5	13.9
0617/18P	12.4	7.5	15.9	8.6	22.5	19.5	13.6
0620P	15.3	6.9	16.5	8.3	22.3	18.9	11.8
0704/5P	15.1	7.1	17.1	8.7	21.8	18.1	12.0
0712P	11.2	7.6	15.9	9.2	25.9	23.2	15.3
0715P	11.9	6.6	15.7	8.3	23.3	21.0	13.3
0717P	12.0	6.0	16.6	8.6	23.8	20.5	12.5
0720P	12.3	6.4	15.9	8.2	23.7	20.3	13.3
0821/22P	15.5	7.0	18.0	8.5	22.2	18.0	10.9
0823/24P	14.9	7.0	16.9	8.9	21.6	18.5	12.2
0826P	12.5	6.5	17.0	8.8	23.4	19.6	12.3
0925P	13.8	6.9	18.5	9.1	23.4	17.6	10.6
0928P	12.8	7.3	17.2	8.4	24.7	18.3	11.2
1001P	13.3	7.1	17.4	8.6	24.3	17.9	11.3
1005P	19.2	7.9	16.6	8.7	19.6	17.2	10.8
1011/12P	18.7	7.6	17.4	10.6	19.6	15.9	10.2
1017P	16.1	5.8	17.8	8.7	21.3	18.6	11.7
1020P	18.2	6.7	17.0	8.0	20.5	18.5	11.1
1114P	21.1	6.7	18.3	8.3	21.6	15.1	8.9
1118P	20.2	7.2	17.4	8.1	21.2	16.2	9.8
1121P	19.5	7.5	17.3	8.1	21.3	15.8	10.4
1211P	16.5	6.9	17.1	8.7	23.2	17.0	10.7
1214P	19.0	6.6	17.6	8.4	21.0	16.7	10.7
0111P	15.3	7.2	16.4	9.2	22.9	17.3	11.7
0114P	15.0	7.3	17.8	8.9	23.5	17.3	10.2
0118P	14.8	7.6	17.0	8.8	23.3	17.4	11.1
0121P	13.1	7.4	17.4	9.3	23.5	17.9	11.4
0122P	13.8	7.5	17.1	9.3	23.3	17.7	11.3
0129P	25.7	7.7	16.1	8.1	17.4	14.8	10.4
0203P	28.0	6.6	14.9	8.3	19.2	13.9	9.1
0206P	23.0	7.0	16.2	9.4	18.2	15.6	10.5
0208/9P	26.6	7.3	15.7	9.0	16.6	14.8	10.0
0213P	22.0	7.7	15.1	10.4	17.0	16.4	11.4
0217P	23.3	7.2	14.6	9.7	17.2	16.4	11.6
0221P	20.1	5.9	15.7	8.8	20.0	17.7	11.7

/Is

TABLE 27
PROTON DISTRIBUTIONS OF PYRIDINE-SOLUBLE
LC-FINER FEED RESIDS
Run 3LCF9

Sample	Proton Distributions			
	Aromatic	Alpha	Beta	Gamma
3LCF9-0321F	47.0	28.2	19.1	5.6
0417(a)	49.1	26.7	19.2	5.1
0429/30F	48.4	28.0	18.8	4.0
0513F	43.5	29.4	22.1	5.0
0521F(a)	44.7	29.2	20.9	5.2
0620F	45.5	29.2	20.0	5.2
0715F	44.1	30.2	20.5	5.2
0720F	42.0	27.6	25.1	5.3
0821/22F	43.3	29.6	21.7	5.4
0925F	49.6	27.8	18.0	4.5
1001F	46.3	30.1	18.8	4.8
1005F	47.0	29.9	18.2	4.9
1020F	48.5	27.9	18.2	5.3
1121F	49.0	29.2	17.3	4.5
1214F	47.8	28.3	18.5	5.3
0111F	48.3	28.9	18.0	4.7
0121F	45.8	30.0	20.0	4.2
0129F	51.4	27.8	16.4	4.4
0206F	49.2	28.6	17.6	4.6
0213F	50.5	27.8	17.0	4.6
0221F	48.5	29.1	17.0	5.3

Samples dissolved in pyridine-d₅ (99.5%D), integrated by planimetry.

(a) Samples dissolved in pyridine-d₅ (99.96%D), integrated electronically.

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TABLE 28

PROTON DISTRIBUTIONS OF PYRIDINE-SOLUBLE
LC-FINER HEAVY OIL PRODUCT RESIDS

Run 3LCF9

Sample	Proton Distributions			
	Aromatic	Alpha	Beta	Gamma
3LCF9-0321P	24.6	29.4	36.4	9.6
0417P(a)	29.5	32.0	30.7	7.9
0429/30P	27.9	31.9	32.9	7.3
0513P	30.6	30.3	31.1	8.0
0527P(a)	32.8	30.7	28.5	8.0
0620P	37.4	32.4	24.2	6.1
0715P	34.3	31.5	26.6	7.6
0720P	36.4	32.0	25.7	5.9
0821/22P	36.5	31.9	25.4	6.2
0925P	35.5	32.2	25.7	6.7
1001P	36.0	32.4	24.7	6.9
1005P	41.9	32.8	20.7	4.6
1020P	41.0	31.1	22.0	5.8
1121P	43.0	31.8	20.8	4.5
1214P	40.6	31.8	22.1	5.5
0111P	38.6	33.2	22.6	5.6
0121P	36.8	32.5	25.0	5.7
0129P	45.7	31.3	18.8	4.2
0206P	47.1	29.6	18.6	4.7
0213P	45.7	30.8	18.7	4.8
0221P	44.4	30.7	19.4	5.4

Samples dissolved in pyridine -d₅ (99.5%D), integrated by planimetry.

(a) Samples dissolved in pyridine -d₅ (99.96%D), integrated electronically.

/Is

TABLE 29
CONCENTRATIONS OF PHENOLIC -OH,
LC-FINER DISTILLATES
Run 3LCF9

Sample	Phenolic -OH Concentration meq/g Sample	
	Feeds	H.O. Products
3LCF9-0321	0.26	0.06
0323		0.07
0417	0.20	0.09
0429/30		0.09
0501	0.23	0.08
0513	0.22	0.07
0524/25		0.14
0527		0.11
0617/18	0.16	0.14
0620		0.18
0704/5	0.19	0.12
0712		0.08(a)
0720	0.34	0.10(a)
0821/22		0.18(b)
0826		0.18
1001	0.23	0.13
1017		0.19
1118	0.28	0.29
1214		0.26
0118	0.32	0.23
0129		0.26
0208/9	0.31	0.28
0221	0.36	0.21(a)

Relative WHSV of Feed = 1.67R except for

(a) WHSV = 1.0R

(b) WHSV = 2.3R

/ls

TABLE 30
 MICROAUTOCLAVE TESTS WITH LC-FINER DISTILLATE SAMPLES
 AT THE MODIFIED EQUILIBRIUM TEST
 Run 3LCF9

<u>Sample</u>	<u>THF Conversion, wt % MAF Coal</u>	
	<u>Feed</u>	<u>H₂O Product</u>
3LCF9-0321	84.9	91.5
0417	84.2	91.6
0513	88.9	89.9
0704/5	86.3	91.3
0720	86.7	89.9
0826	85.8	90.5
1001	90.2	91.4
1005(a)	84.7	87.8
1020	81.8	89.8
1118	89.5	90.6
1214	87.1	90.5
0118	90.3	91.7
0129	79.8	89.1
0203	74.4	83.3
0208/9	74.8	87.3
0217	74.0	85.1
0221	71.5	89.5

Conditions: 9 g distillate solvent, 6 g coal, 750°F,
 30 min.

Coal: Old Ben No. 1, Indiana 5

(a) Indiana 5 coal period

/ls

TABLE 31

LC-FINER CONVERSIONS
Run 3LCF9

Run Period	Cat Age, lb 850°F ⁺ lb cat	Conversion, wt %				Total Organic 850°F ⁺
		Oils	Asphaltenes	Oils & Asphaltenes	Preasphaltenes & IOM	
3LCF9-0321	25	-7.2	57.6	13.6	82.1	40.8
0323	40	-5.2	65.2	17.5	76.8	42.7
0417	56	29.2	44.7	33.4	57.7	43.5
0429/30	106	21.8	38.6	26.7	56.8	40.0
0501	135	23.4	40.0	28.4	54.3	38.4
0513	201	24.5	26.3	25.1	58.7	40.1
0524/25	287	24.1	18.8	22.5	59.8	38.8
0527	310	24.2	19.1	22.6	62.6	40.6
0617/18	398	18.4	5.4	14.0	50.6	30.8
0620	432	16.7	6.8	13.4	62.4	32.9
0704/5	486	20.6	14.7	18.8	51.1	32.1
0712(b)	512	23.6	42.6	31.5	72.8	51.0
0715(b)	538	25.9	40.9	31.4	68.5	47.6
0717(b)	557	28.4	35.4	30.9	72.5	48.9
0720(b)	581	26.5	28.6	27.2	69.4	46.4
0821(c)	627	2.0	-3.7	0.0	51.1	21.7
0824(c)	661	1.0	-11.3	-3.1	54.2	22.9
0826	697	21.9	17.1	20.3	49.3	31.7
0925	720	14.7	19.2	16.3	64.3	39.4
0928	754	5.6	19.8	11.0	64.5	35.8
1001	785	12.2	8.3	15.8	56.3	32.6
1005(a)	851	6.2	15.2	10.2	62.0	33.3
1011(a)	878	1.2	19.6	9.5	57.4	30.4
1016	900	7.2	-7.3	2.7	42.6	15.1
1020	985	12.4	13.9	12.9	53.0	26.3
1114	1179	-2.0	-11.0	-5.2	49.0	20.6
1118	1228	-17.6	-5.0	-12.8	64.0	27.0
1121	1266	-8.4	-2.5	-6.2	55.9	24.7
1211	1289	12.8	5.3	10.0	41.3	24.4
1214	1340	-0.5	-1.7	-0.9	54.3	22.3
0110	1362	-15.2	-8.5	-12.6	54.1	25.9
0114	1398	15.6	-3.5	8.6	48.1	28.5
0118	1428	16.2	4.3	11.9	48.6	28.5
0121	1462	16.8	23.5	19.2	46.0	29.1
0122	1474	20.2	16.7	18.9	48.6	29.7
0129	1500	17.4	6.8	13.8	37.0	21.1
0203	1547	5.7	15.8	9.5	27.7	15.5
0206	1597	10.3	8.5	9.7	36.6	18.2
0208/9	1644	1.0	11.4	5.0	35.6	15.9
0213	1699	-3.4	15.6	3.7	44.0	17.1
0217	1743	-14.6	8.1	-5.6	36.4	8.6
0221(b)	1778	3.8	21.4	10.5	66.2	24.0

All periods made with Illinois 6 coal at WHSV = 1.67R except

(a) Indiana 5 coal periods

(b) WHSV = 1.0R

(c) WHSV = 2.3R

/Is

TABLE 32
FIRST ORDER KINETIC CONSTANTS
Run 3LCF9

Run Period	Catalyst Age, lb 850°F ⁺ lb cat	k(T), Vol 850°F ⁺ Vol cat-hr	A, Vol 850°F ⁺ x 10 ⁷ Vol cat-hr	k(T), lb 850°F ⁺ lb cat hr	A, lb 850°F ⁺ x 10 ⁷ lb cat-hr
3LCF9-0323	40	0.211	1.113	0.330	1.669
0417	56	0.257	1.460	0.385	2.188
0429	106	0.228	1.595	0.331	2.316
0501	135	0.236	1.510	0.356	2.278
0513	201	0.264	1.390	0.397	2.089
0524	287	0.195	0.983	0.284	1.432
0526	310	0.215	1.049	0.316	1.543
0617	398	0.158	0.748	0.231	1.095
0620	432	0.175	0.819	0.287	1.345
0704/5	486	0.172	0.895	0.257	1.336
0712	512	0.184	0.983	0.282	1.509
0715	538	0.159	0.775	0.241	1.175
0717	557	0.171	0.860	0.263	1.322
0720	581	0.158	0.795	0.256	1.290
0821	627	0.155	0.890	0.247	1.420
0824	661	0.160	0.869	0.242	1.313
0826	697	0.172	0.973	0.256	1.448
0925	720	0.147	0.688	0.243	1.138
0928	754	0.162	0.759	-	-
1001	785	0.142	0.643	0.219	0.993
1005	851	0.213	0.860	-	-
1011	878	0.166	0.775	0.248	1.159
1016	908	0.074	0.361	0.120	0.586
1020	985	0.152	0.709	0.235	1.094
1114	1179	0.122	0.572	0.189	0.887
1118	1228	0.096	0.588	0.175	1.072
1121	1266	0.098	0.619	0.150	0.951
1211	1289	0.085	0.410	0.130	0.627
1214	1340	0.129	0.585	0.197	0.892
0110	1362	0.098	0.545	0.176	0.860
0114	1398	0.124	0.705	0.206	1.174
0118	1428	0.131	0.754	0.214	1.229
0121	1462	0.109	0.637	0.170	0.991
0122	1474	0.120	0.709	0.186	1.102
0129	1500	0.120	0.652	0.192	1.043
0203	1547	0.088	0.478	0.132	0.715
0206	1597	0.097	0.544	0.147	0.824
0208	1644	0.079	0.466	0.118	0.698
0213	1699	0.077	0.465	0.122	0.738
0217	1743	0.040	0.260	0.060	0.386
0221	1778	0.060	0.398	0.094	0.622

Rate constant, $k(T) = 3(S.V.) \left(\frac{1}{([850^\circ F^+ \text{ OUT}]/[850^\circ F^+ \text{ IN}])^{1/3}} - 1 \right)$ for 3 CSTR's in Series

Pre-exponential Factor, $A = k_e \frac{E_{act}}{RT}$

S.V. = linear hourly space velocity of 850°F⁺ (vol 850°F⁺/Vol cat-hr) or weighted hourly space velocity of 850°F⁺ (lb 850°F⁺/lb cat-hr)

E_{act} = 23,500 cal/mole from Lummus

[850°F⁺] = Concentration (wt %) of total organic 850°F⁺ fraction

/ls

TABLE 33

COMPARISON OF FIRST ORDER KINETIC CONSTANTS - CONOCO AND LUMMUS
Run 3LCF9

Run Period	Catalyst Age, lb 850°F ⁺ lb cat	k(T), Vol 850°F ⁺ Vol cat-hr		A, Vol 850°F ⁺ x 10 ⁷ Vol cat-hr	
		Conoco	Lummus	Conoco	Lummus
3LCF9-0323	40	0.211	0.21	1.113	1.09
0417	56	0.257	0.24	1.460	1.32
0429	106	0.228	0.24	1.595	1.68
0501	135	0.236	0.23	1.510	1.45
0513	201	0.264	0.29	1.390	1.40
0524	287	0.195	0.19	0.983	0.94
0526	310	0.215	0.18	1.049	0.87
0617	398	0.158	0.14	0.748	0.67
0620	432	0.175	0.12	0.819	0.54
G704/E	486	0.172	---	0.895	0.90

/ls

TABLE 34

ANALYSES OF BURNING STAR, ILLINOIS 6 COAL

Proximate, wt % as determined

Moisture	4.61
Volatile Matter	40.51
Fixed Carbon (diff.)	44.82
Oxidized Ash	10.06
Low Temperature Ash	11.6

Ultimate, wt % dry basis

Carbon	70.70
Hydrogen	4.69
Nitrogen	1.42
Oxygen (diff.)	9.57
Sulfur, total	3.02
Pyritic	0.97
Sulfate	0.07
Organic (diff.)	1.98
Chlorine	0.05
Ash	10.55

Elemental, wt % oxidized ash

Na ₂ O	0.64
K ₂ O	1.92
CaO	6.62
MgO	1.02
Fe ₂ O ₃	16.39
TiO ₂	0.98
P ₂ O ₅	0.07
SiO ₂	47.18
Al ₂ O ₃	22.12
SO ₃	3.02
Unaccounted	0.04

Calorific Value, dry (HHV)	12,648 Btu/lb
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Wet Screen

<u>Tyler Mesh</u>	<u>wt %</u>
+48	< 0.1
48 x 100	0.9
100 x 200	14.7
200 x 325	20.9
-325	63.9

/ls

TABLE 35

ANALYSIS OF OLD BEN NO. 1 MINE, INDIANA 5 COAL

Proximate, wt % as determined

Moisture	2.34
Volatile Matter	40.59
Fixed Carbon (diff.)	46.78
Oxidized Ash	10.29

Ultimate, wt % dry basis

Carbon	70.80
Hydrogen	4.94
Nitrogen	1.49
Oxygen (diff.)	8.59
Sulfur, total	3.64
Pyritic	1.45
Sulfate	0.02
Organic (diff.)	2.17
Chlorine	<0.01
Oxidized Ash	10.54

Elemental, wt % Oxidized Ash

Na ₂ O	0.36
K ₂ O	2.44
CaO	3.39
MgO	0.86
Fe ₂ O ₃	22.60
TiO ₂	0.66
P ₂ O ₅	0.15
SiO ₂	43.34
Al ₂ O ₃	18.82
SO ₃	2.05
Unaccounted	5.33

<u>Calorific Value, dry (HHV)</u>	12,846 Btu/lb
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Wet Screen

<u>Tyler Mesh</u>	<u>wt %</u>
+100	0.0
100 x 200	10.8
200 x 325	20.0
-325	69.2

/ls

TABLE 36

COMPOSITION OF FEED RESIDS
THERMAL CONVERSION OF PASTING SOLVENT RESIDS

Resid Sample	wt % of Sample				
	Oils	Asphaltenes	Preasphaltenes	IOM	Ash
SCT16-6PS3381	32.5	18.0	34.1	11.0	4.5
SCT14-PS51280	47.4	18.3	22.6	6.0	5.8
SCT14-PS31280	55.4	20.9	14.5	3.8	5.4

/ls

TABLE 37

THERMAL CONVERSION OF PASTING SOLVENT RESIDS

	Gas	Total 850°F ⁻	Soluble Resid	IOM	Ash	Oils	Asphaltenes	Pre- asphaltenes	Preasph + IOM	Total 850°F ⁺ Organic
<u>SCT16-6PS3381</u>										
Total Feed Composition, g	0.0	9.76	4.44	.58	.23	1.70	.95	1.79	2.37	5.02
Total Product Composition, g	≤0.21	10.88	3.75	.13	.24	1.76	1.05	.95	1.08	3.89
Yield, g	≤0.21	1.12	-.67	-.44	.00	.06	.10	-.84	-1.28	-1.12
Conversion wt %	--	--	15.1	76.6	--	-3.4	-10.9	47.1	54.3	22.4
<u>SCT14-PS51280</u>										
Total Feed Composition, g	0.0	9.75	4.63	.32	.30	2.49	.96	1.19	1.50	4.95
Total Product Composition, g	≤0.20	10.95	3.43	.27	.31	1.84	.88	.75	1.02	3.75
Yield, g	≤0.20	1.20	-1.15	-.05	.00	-.64	-.08	-.43	-.48	-1.20
Conversion, wt %	--	--	24.9	14.6	--	25.9	6.2	36.4	31.8	24.3
<u>SCT14-PS51280</u>										
Total Feed Composition, g	0.0	9.75	4.77	.20	.28	2.91	1.10	.76	.96	4.97
Total Product Composition, g	≤0.20	10.58	3.89	.24	.29	2.32	.97	.60	.84	4.13
Yield, g	≤0.20	.83	-.88	.04	.01	-.59	-.13	-.16	-.12	-.84
Conversion, wt %	--	--	18.5	-22.1	--	20.4	11.8	21.1	12.2	16.9
vls										

TABLE 38

PROTON DISTRIBUTIONS OF FEED AND PRODUCT DISTILLATES
THERMAL CONVERSION OF PASTING SOLVENT RESIDS

	Proton Distribution						
	<u>Cond</u> <u>Arom</u>	<u>Uncond</u> <u>Arom</u>	<u>Cyclic</u> <u>Alpha</u>	<u>Alkyl</u> <u>Alpha</u>	<u>Cyclic</u> <u>Beta</u>	<u>Alkyl</u> <u>Beta</u>	<u>Gamma</u>
Feed Distillate	20.9	6.3	16.1	9.0	20.9	16.4	10.5
<u>Product Distillate</u>							
SCT16-6PS3381	25.3	5.7	16.6	8.1	19.2	15.5	9.6
SCT14-PS51280	24.0	6.1	16.9	8.4	19.9	15.4	9.3
SCT14-PS31280	24.0	5.9	17.4	8.4	19.8	15.2	9.2

/ls

TABLE 39

PROTON DISTRIBUTIONS OF THF SOLUBLE RESIDS
THERMAL CONVERSION OF PASTING SOLVENT RESIDS

	Proton Distributions			
	<u>Aromatic</u>	<u>Alpha</u>	<u>Beta</u>	<u>Gamma</u>
<u>Feed Resids</u>				
SCT16-6PS3381	35.4	27.0	29.4	8.2
SCT14-PS51280	27.0	27.7	34.0	11.4
SCT14-PS31280	27.8	28.1	34.0	10.2
<u>Product Resids</u>				
SCT16-6PS3381	41.5	28.4	24.1	6.0
SCT14-PS51280	37.3	29.5	26.9	6.2
SCT14-PS31280	35.7	30.0	28.1	6.2

Samples dissolved in pyridine -d₅ (99.5% D), integrated by planimetry.

/Is

TABLE 40

THERMAL CONVERSION OF LC-FINER FEED RESID: (@780°F, 1000 psi Cold H₂)
 Thermal Contribution of 850°F⁺ Conversion in the LC-Finer

	Gas	Total 850°F ⁻	Soluble Resid	IOM	Ash	Oils	Asphaltenes	Pre- asphaltenes	Preasph + IOM	Total 850°F ⁺ Organic
<u>Residence Time = 1 Hr</u>										
Total Feed Composition, g	0.0	6.01	7.56	.99	.43	2.46	1.53	3.58	4.57	8.56
Total Product Composition, g	≥0.63	7.71	6.29	.57	.43	2.57	1.98	1.74	2.31	6.86
Yield, g	--	1.70	-1.27	-.42	0	.11	.45	-1.84	-2.26	-1.70
Conversion wt %	--	--	16.8	42.4	--	-4.5	-29.4	51.4	49.4	19.9
<u>Residence Time = 1.5 Hr</u>										
Total Feed Composition, g	--	6.01	7.55	.99	.43	2.45	1.52	3.57	4.56	8.54
Total Product Composition, g	N.D.	8.05	5.99	.50	.43	2.64	1.87	1.49	1.99	6.50
Yield, g	--	2.04	-1.56	-.49	0	.19	.35	-2.08	-2.57	-2.04
Conversion wt %	--	--	20.7	49.5	--	-7.8	-23.0	58.3	56.4	23.9
<u>Residence Time = 2 Hr</u>										
Total Feed Composition, g	--	6.01	7.56	.99	.43	2.46	1.53	3.58	4.57	8.56
Total Product Composition, g	≥0.10	8.29	5.78	.50	.43	2.58	1.77	1.43	1.93	6.28
Yield, g	--	2.28	-1.78	-.49	0	.12	.24	-2.15	-2.64	-2.28
Conversion wt %	--	--	23.5	49.5	--	-4.9	-15.7	60.1	57.8	26.6
/ls										

TABLE 41

CONVERSION OF OILS, ASPHALTENES, PREASPHALTENES + IOM, AND TOTAL 850°F⁺
ORGANIC IN MICROAUTOCLAVE RUNS vs PDU

Thermal Contribution of 850°F⁺ Conversion in the LC-Finer

Run Description	Residence Time (hr)	Conversion, wt %			
		Oils	Asphaltenes	Preasph + IOM	Total 850°F ⁺ Organic
Microautoclave	1.0	-4.5	-29.4	49.4	19.9
Microautoclave	1.5	-7.8	-23.0	56.4	23.9
Microautoclave	2.0	-4.9	-15.7	57.8	26.6

	S.V. Vol 850°F ⁺ Vol Ca ⁺	Cat Age lb 850°F ⁺ lb Cat				
3LCF7-30	0.20	234	27.5	48.8	71.1	53.4
3LCF7-48	0.22	383	24.6	36.8	62.1	46.8
3LCF7-85	0.47	930	4.5	12.7	58.4	30.9

$$\text{Conversion} = \left(\frac{\text{IN}-\text{OUT}}{\text{IN}} \right) \times 100\%$$

/ls

TABLE 42

PROTON DISTRIBUTIONS OF DISTILLATES
FROM MICROAUTOCLAVE EXPERIMENTS

Thermal Contribution of 850°F⁺ Conversion in the LC-Finer

<u>Sample</u>	<u>Proton Distributions</u>						
	<u>Condensed Aromatics</u>	<u>Uncondensed Aromatics</u>	<u>Cyclic Alpha</u>	<u>Alkyl Alpha</u>	<u>Cyclic Beta</u>	<u>Alkyl Beta</u>	<u>Gamma</u>
Feed Distillate	10.6	5.9	14.9	8.6	25.7	20.5	13.8
Product Distillates							
1.0 hr run	23.1	5.0	15.7	9.0	19.1	17.4	10.7
1.5 hr run	24.8	5.3	14.6	9.2	17.1	18.1	10.9
2.0 hr run	23.4	5.0	15.3	9.3	18.8	18.0	10.3
Average Product	23.8	5.1	15.2	9.2	18.3	17.8	10.6
/ls							

TABLE 43

PROTON DISTRIBUTIONS OF THF SOLUBLE RESIDS
Thermal Contribution of 850°F⁺ Conversion
in the LC-Finer

<u>Sample</u>	<u>Proton Distributions</u>			
	<u>Aromatic</u>	<u>Alpha</u>	<u>Delta</u>	<u>Gamma</u>
Feed Resid	43.4	29.0	22.1	5.6
Product Resid				
1.0 hr run	48.1	28.5	18.7	4.7
1.5 hr run	49.0	27.9	18.4	4.7
2.0 hr run	49.6	28.8	18.0	3.6

Samples dissolved in pyridine-d₅ (99.5%D), integrated by planimetry.

/Is

TABLE 44
SOLVENT FRACTIONATION OF THF SOLUBLE RESIDS
Thermal Contribution of 850°F⁺ Conversion
in the LC-Finer

<u>Sample</u>	<u>Oils</u>	<u>Asphaltenes</u>	<u>Preasphaltenes</u>
Feed Resid	32.5	20.2	47.3
Product Resid			
1.0 hr run	40.9	31.4	27.7
1.5 hr run	44.0	31.2	24.8
2.0 hr run	44.7	30.5	24.8
/ls			

TABLE 45

MICROAUTOCLAVE TESTS AT SCT/N₂ CONDITIONS
Solvent Quality at SCT Conditions vs Boiling Point

Fraction No.	Nominal Boiling Point (end) °C	Conversion to THF Solubles, wt % MAF Coal
1	203	56.8*
2	311	76.5*
4	323	85.0
7	349	88.8
10	383	88.3
13	410	86.6
16	421	82.8
17	440	83.4
19	498	76.9
22	514	70.5
Comp., 1 + 18	470 ⁻	86.9
Same, no added N ₂	470 ⁻	88.1

SCT/N₂ Conditions: 860°F, 5 min, 9 g solvent, 5 g coal
 1000 psig cold N₂

Coal: Indiana 5, Old Ben No. 1 Mine

*Cokod

/ls

TABLE 46
COMPARISON OF ACTUAL AND NOMINAL
BOILING POINTS OF DISTILLATION FRACTIONS
Solvent Quality at SCT Conditions vs. Boiling Point

<u>Fraction</u>	<u>Nominal Boiling Point (end), C</u>	<u>Actual Boiling Point, °C</u>		
		<u>IBP</u>	<u>Mode</u>	<u>FPB</u>
4	323	220	315	390
7	349	260	340	390
10	383	290	380	440
13	410	340	395	450
19	498	400	465*	500*

*Assuming whole sample eluted

/Is

TABLE 47
COMPONENT DISTRIBUTION
HYDROCLONE OVERFLOW SAMPLES
H-Coal Pilot Plant Run 9

Sample		wt % of Total Sample			
Date (1982)	Time	290°C /1 torr Distillate	THF Soluble Resid	IOM	Ash
4/6	2030	55.1	20.5	14.9	9.2
4/7	1620	53.0	20.3	14.8	11.1
4/8	2400	54.8	22.2	12.1	9.9
4/21	1220	40.0	28.6	15.6	15.3
4/23	0430	34.2	33.9	16.7	14.9
PDU Run 10, Avg.		58.3	26.2	7.8	9.2

/Is

TABLE 48
TOTAL RECYCLE OIL COMPOSITION
H-Coal Pilot Plant Run 9

Sample Date (1982)	lb/lb MF Coal Basis				
	Total 995°F ⁻ Distillate	THF Soluble Resid	IOM	Ash	Total
4/6	1.94	0.12	0.09	0.05	2.20
4/7	2.03	0.19	0.14	0.11	2.48
4/8	2.07	0.25	0.14	0.11	2.58
4/21	1.66	0.44	0.24	0.24	2.59
4/23	1.61	0.48	0.24	0.21	2.53
PDU Run 10,	1.45	0.38	0.11	0.10	2.06
Avg., Periods 27 + 40					
/ls					

TABLE 49

SOLVENT FRACTIONATION OF HYDROCLONE OVERFLOW
THF SOLUBLE RESIDS

H-Coal Pilot Plant Run 9

Sample Date (1982)	wt % of Soluble Resid		
	<u>Oil</u> s	<u>Asphaltenes</u>	<u>Preasphaltenes</u>
4/6	74.3	20.7	5.0
4/7	72.3	21.5	6.2
4/8	70.4	24.8	4.8
4/21	67.0	24.3	8.7
4/23	66.8	26.1	7.1
PDU Run 10, Avg.	51.3	29.6	19.1
/ls			

TABLE 50

PROTON DISTRIBUTIONS
HYDROCLONE OVERFLOW DISTILLATES (975°F⁻)

H-Coal Pilot Plant Run 9

<u>Sample Date</u> <u>(1982)</u>	<u>Cond</u> <u>Arom</u>	<u>Uncond</u> <u>Arom</u>	<u>Cyclic</u> <u>Alpha</u>	<u>Alkyl</u> <u>Alpha</u>	<u>Cyclic</u> <u>Beta</u>	<u>Alkyl</u> <u>Beta</u>	<u>Gamma</u>
4/6	13.0	7.6	15.9	9.3	18.2	24.2	11.9
4/7	13.7	7.6	15.7	9.2	17.7	24.7	11.5
4/8	13.5	7.5	15.9	9.5	17.4	24.8	11.4
4/21	15.9	6.9	16.2	10.1	15.0	25.2	10.7
4/23	17.1	6.9	16.2	10.4	14.5	25.0	9.9
PDU Run 10, Avg.	16.1	8.0	13.6	10.7	13.0	26.9	11.8
/ls							

TABLE 51

PROTON DISTRIBUTIONS
FRACTIONATOR BOTTOMS SAMPLES

H-Coal Pilot Plant Run 9

<u>Sample</u>		<u>Cond</u>	<u>Uncond</u>	<u>Cyclic</u>	<u>A kyl</u>	<u>Cyclic</u>	<u>Alkyl</u>	<u>Gamma</u>
<u>Date (19E2)</u>	<u>Time</u>	<u>Arom</u>	<u>Arom</u>	<u>Alpha</u>	<u>A pha</u>	<u>Beta</u>	<u>Beta</u>	
3/22	1630	18.4	7.3	17.5	9.0	19.0	18.6	10.2
3/23	1516	16.8	7.0	17.5	9.2	13.5	20.7	10.3
PDU Run 10, Avg.		13.4	8.1	13.2	10.6	13.8	28.5	12.4

/Is

TABLE 52

MICROAUTOCLAVE TESTS
HYDROCLONE OVERFLOW DISTILLATES (975°F⁻)

H-Coal Pilot Plant Run 9

Sample Date (1982)	THF Conversion, wt % MAF Coal	
	KIN	EQ
4/6		76.3*
4/7		76.3*
4/8	76.5	79.7*
4/21		72.7*
4/23		73.1*
PDU Run 10, Composite	70.7	64.1

*Possible Coking

KIN: 10 min, 750°F, 8/1 Solvent/Coal

EQ: 30 min, 750°F, 2/1 Solvent/Coal

Coal: Old Ben No. 1, Indiana 5

/ls

Figure 1. ITSL PDU - Process Flow Diagram.

Figure 2. Lummus Run 3LCF9 - 850°F+ Conversion vs Catalyst Age.

Figure 3. First-Order Pre-exponential Factor for 850°F^+ Conversion vs Catalyst Age -- Run 3LCF9.

Figure 4. Coal Conversion at SCT/N₂ Conditions vs Boiling Point of Pasting Solvent Distillation Fraction.

Figure 5. Characteristics of Boiling Fractions of ITSL Pasting Solvent vs Boiling Point.