Exploratory Research on Novel Coal Liquefaction Concept

**Quarterly Report
April 1 - June 30, 1997**

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SUMMARY OF TECHNICAL PROGRESS - OVERALL

Task 3 experimental tests were concluded this quarter. Second-stage testing of first-stage distilled filtrates and of a Wilsonville resid from Run 263 were completed. The first-stage filtrates were found to have higher reactivity than the Wilsonville material.

Task 4 experimental testing was initiated. First-stage microautoclave tests were made at reduced catalyst loading and reduced solvent to coal ratio without negatively affecting coal conversion. Lower severity second-stage catalytic upgrading tests were begun. The single-pass resid conversion obtained in the first completed second-stage test was high (67-69 %).

The engineering evaluation of the first-stage tests made under Task 3 was completed. Evaluation of second-stage Task 3 tests was started. Comparison of a second-stage test made with Wilsonville Run 263J ashy resid and those made with first-stage distilled filtered products was begun.

SUMMARY OF TECHNICAL PROGRESS BY TASK

Task 3 - Flow Sheet Development

First-Stage Tests, Filtration and Distillation of First-Stage Products

Task 3 tests completed last quarter included four successful Run 23-LA series tests (through Run 23d-LA).¹ Distillation of the filtered products of these tests was completed this quarter to prepare feedstocks for second-stage testing (Table 1). Distillation of the composited filtrates of first-stage Runs 23-LA, 23a-LA and 23c-LA (produced last quarter) was carried out under vacuum with an atmospheric equivalent cut point temperature of 454 °C (850 °F), yielding 25.3% residue and 74.7% distillate. The residue had a softening point of 290 °C. This high softening point could be indicative of partially coked material. The next filtrate distillation (Run 23d-LA) was carried out at less severe conditions, at an atmospheric equivalent temperature of 416 °C (780 °F). The products collected were in the proportions 29.5 wt % residue and 70.5 wt % distillate. The residue had a softening point of 165 °C. Analysis by simulated distillation of the residue gave 24.9 wt % boiling <566 °C, as opposed to 6 wt % boiling <566 °C that was found in the residue of the composited filtrate.

Three one-liter autoclave tests were made this quarter (Runs 23f-LA, 23g-LA, and 23h-LA). All tests were made at the same conditions as Run 23-LA (375 °C, 60 min, 222 mmole KOH/kg dry coal, HI "E", solvent/dry coal = 1.9) to produce sufficient feedstock for second-stage catalytic upgrading tests and to provide information on repeatability of one-liter test data. The integrated filter unit was used for all tests; however, filtration was not successful for Run 23g-LA (see below).

Mass and elemental balances were obtained on most of the Run 23-LA series tests. The material balances achieved for three of the tests (Runs 23f-LA, 23g-LA and 23h-LA) were 110.0%, 104.1% and 98.4% respectively. The poor balances in Runs 23f-LA and 23g-LA are attributed to inaccuracies in the measured gas yields, which were 146 g, 133 g, and 114 g for Runs 23f-LA, 23g-LA, and 23h-LA, respectively.

Run 23f-LA was filtered in situ and yielded 165.08 g of filtrate (containing 0.3% quinoline insolubles (QI) and 3.5% tetrahydrofuran insolubles (THFI)) and 20.39 g of filter cake (40.4% QI and 44.9% THFI) (Table 2). As in the distillation of Run 23d-LA (performed at 416 °C), in order to provide a larger quantity of feed for second-stage tests and to reduce the possibility of cracking the first-stage product, a lower distillation cut point was chosen than 454 °C (the temperature used in Run 23-LA series tests through Run 23c-LA). The filtrate was distilled under vacuum to an atmospheric equivalent temperature of 426 °C. The products collected were in the proportions 27.9% residue and 72.1% distillate. The residue had the same softening point (165 °C) and a similar amount (19.9%) of <566 °C distillate (19.9% vs 24.9%) as the Run 23d-LA distillation residue.

Run 23g-LA was not successfully filtered in situ. Material lodged in the valve between the autoclave and the filter apparatus and prevented transfer of material to the filter. The product was sampled for elemental analyses and the remainder was filtered in the 200 mL filter apparatus off line (Table 2). Yield of filtrate was 143.37 g (0.2% QI, 2.0 % THFI). The filter cake yield was 15.61 g (40.8% QI, 42.1% THFI). Correcting these results for the product removed for analyses, the yields are 165.13 g of filtrate and 17.98 g of filter cake; overall material balance for the run was 104%. The filtration rate was calculated to be 225 kg/m²/h with a cake resistivity of 34×10^{10} m/kg. These data compare well with the 208 kg/m²/h and 33×10^{10} m/kg achieved in Run 23-LA. The filtrate was distilled under vacuum to an atmospheric equivalent temperature of 440 °C. The resulting products were in the proportions 25.5% residue and 74.5% distillate. The residue had a softening point of 255 °C. Analysis by simulated distillation of the residue gave 12.3% of the material boiling <566 °C.

Run 23h-LA was filtered in situ and yielded 165.15 g of filtrate and 19.64 g of filter cake. Overall material balance was 98.4%. The products are being held in reserve for Task 4 tests.

Product work-up for all one-liter autoclave tests includes gas chromatography of gaseous products. Hydrogen content previously was determined by difference. A direct hydrogen gas analysis is now being used.

Samples of light distillate oil obtained from the post-run flash of products from the one-liter autoclave were analyzed by simulated distillation to provide information for the engineering and economic evaluation. The data are given in Table 12.

Second-Stage Catalytic Upgrading

The upgrading properties of the Run 23-LA series first-stage product were compared with those of a conventional two-stage liquefaction resid this quarter. Three microautoclave tests were made with Run 23d-LA distilled filtrate, (Runs 23d-1, 50 MA, 23d-2, 50 MA, and 23d-3, 50 MA (Table 3)) all at the same conditions (440 °C, 60 min, 1% Mo). Average 566°C⁺ resid conversion for the three microautoclave tests was ca. 45 wt %. This is 10-17 wt % higher than resid conversions obtained in microautoclave tests with Wilsonville Run 258A deashed resid.²

Gas yield in Run 23aoc-1, 300 CSTR, (made last quarter¹) was unusually high (17 wt % MAF feed). A microautoclave test was made with Run 23d-LA distilled filtrate at 450 °C, 60 min, and 1% Mo to examine the possibility that temperatures in the 300 mL CSTR had been monitored inaccurately and the run temperature was higher than 440 °C. Resid conversion in this microautoclave test (Run 23d-4, 50 MA) was 62 wt % (ca. 17 wt % higher than the average of Runs 23d-1, -2, -3, 50 MA). Gas yield also was higher than in the lower temperature tests, but only by ca. 4 wt % (to 7.7 wt % MAF feed). Calculations made using the ideal gas law and conditions in the reactor and those made using the collected volume of product gas were compared to evaluate if the gas yield determined for the 300 mL test was inaccurately calculated. Calculations made both ways were within one standard deviation and can not explain the high gas make in Run 23aoc-1, 300 CSTR. It is possible that the microautoclave tests may accurately represent trends, but not the magnitude of differences seen in the 300 mL reactor. Reaction severity will be further examined in the 300 mL CSTR in Task 4.

Two second-stage hydrotreating tests were completed in the 300 mL CSTR (Tables 4 and 5). Run 23dfg-1, 300 CSTR, was the second test made with first-stage products of the Run 23-LA series. The feed for this run contained a higher distillate content (ca. 20 wt %) than the first test, Run 23oac-1, 300 CSTR (ca. 6 wt %, see above), but in all other respects was a duplicate of the first run (Table 6). However, resid conversion for Run 23dfg-1, 300 mL CSTR was 65 wt %, 8% (abs.) higher than that which was obtained for Run 23oac-1, 300 mL CSTR. The hydrogen uptake and the hydrogen-to-carbon ratio of the 566 °C⁺ fraction also were higher. Each of these results suggest that there may have been more hydro conversion activity in this run than in the previous one. This may, in part be related to less severe distillation of the feedstock.

A baseline test was made with products of the Wilsonville pilot plant to compare with the first-stage products of the Run 23-LA series. Run 263J-1, 300 CSTR, was made with the distillation resid of interstage products from Wilsonville Run 263J. This resid contains undissolved coal,

fresh resid, and recycled process solvent. Simdist analysis of the feed found it to contain 28.7% 566 °C⁻, with an IBP of about 425 °C. Resid conversion was 44%, ca. 20 wt % less than that obtained with the first-stage filtrates from Run 23-LA series tests. Light gas yield was over 18 wt % (MAF feed basis), comparable to that obtained with the first-stage filtrates. THF extraction of Run 263J-1, 300 CSTR, feed gave 21.9 wt % IOM. The dry reactor-scrape product contained 64.9% IOM. This constitutes an increase in mass of IOM of 2.25:1. This result and the high gas yields may indicate that regressive polymerization reactions may have occurred and that the reaction conditions may have been too severe. This will be confirmed in work under Task 4.

In order to assess the quality of the material remaining in the 300 mL reactor at the end of the run, the reactor products recovered without THF ("dry reactor scrape") from four of the 300 mL CSTR tests (DARo-1, 300 CSTR, 23oac-1, 300 CSTR, 23dfg-1, 300 CSTR, and 263J-1, 300 CSTR) were distilled to a 566 °C cut point. The distillate and resid samples were submitted for proximate and ultimate analysis. The results are summarized in Tables 7-10. The H/C ratio of the 566 °C⁺ fraction of the feeds to these CSTR runs will be determined. The relatively low H/C ratio for all 566 °C⁺ products raises a number of issues which will be investigated in Task 4. The effects of test-condition severity on this value will be investigated. The behavior of these materials on recycle and how these low values affect ultimate conversion to 566 °C⁻ product also will be examined.

Task 4 - Integrated Flow Sheet Testing

First-Stage Tests, Filtration and Distillation of First-Stage Products

Microautoclave tests were made to examine the effect on coal conversion of lower KOH catalyst loading and the effect on conversion of reduced solvent to coal ratio. A one-liter autoclave test was made at catalyst loading lower than that used in the Run 23-LA series of tests.

Five microautoclave tests were made to examine the effect on coal conversion of lower KOH catalyst loading. Tests were made at 375°C for 60 min with solvent/dry coal ratio = 1.8, and catalyst loadings of 111, 83, 65, 55, and 45 mmole/kg dry coal. Coal conversions were 89.9, 90.6, 90.3, 87.9, and 88.2 wt %, respectively, assuming the KOH reports to the insolubles. (There is little difference in these conversions even if all the KOH reports to the solubles, because small amounts of catalyst were used, 0.025 - 0.010 g.). The results indicate that the first-stage catalyst loading can be reduced from 222 mmole/kg dry coal used in the one-liter autoclave series (Runs 23x-LA) without reducing coal conversion. The lower usage rate would result in a cost reduction of \$1.00 to \$1.50 per barrel of product gasoline.

Microautoclave tests also were made at reduced solvent/dry coal ratios at 375 °C for 60 min with 83 mmole KOH/kg dry coal. (Table 11). The solvent/dry coal ratios used were 1.5, 1.25, and 1. This is considerably lower than 1.9 which is the ratio used in the one-liter autoclave series 23x-LA. Coal conversions for these tests are all within 2 wt % (abs.) of 90 wt %. An additional test (Run 247, Table 11), made at a solvent/dry coal ratio = 1, but at a reduced KOH loading (50 mmole/kg dry coal), gave 90 wt % coal conversion. These encouraging results merit further investigation of reduced catalyst loading and solvent to dry coal ratios.

One-liter autoclave test Run 24-LA was made at the same conditions as the Run 23-LA series, except with half KOH loading (111 mmole KOH/kg dry coal vs. 222 mmole KOH/kg dry coal). The test proceeded as planned. A material balance for the run was 100.6 %. The recovered products are being analyzed. Mass and elemental balances for Run 24-LA were completed. The coal conversion was 81.7 wt %, considerably lower than conversion (88.8 wt %) obtained in the corresponding microautoclave test (Run 242) under the same conditions. A repeat of Run 24-LA is planned.

A one-liter autoclave test, Run 25-LA, was made at reduced catalyst loading and reduced solvent to dry coal ratio. The sample was filtered on-line. Products are being analyzed, results will be reported next quarter.

Samples of light distillate oil obtained from the first-stage one-liter autoclave were analyzed by simulated distillation to provide information for the engineering and economic evaluation (Table 12). The distillates of products from the 23-LA series of first-stage tests were composited and will be used for first-stage solvent recycle tests.

Plans were made to divide the filtrate of Run 23h-LA into three parts and distill each part to an different end point. The three end points will give samples with 25%, 50% and 75% residue for Task 4 microautoclave tests

Second-Stage Catalytic Upgrading

Run DARo-2, 300 CSTR, was completed in the continuous hydrotreating system. The Mo loading and temperature were the same as DARo-1, 300 CSTR.¹ However, the run time was shortened from 60 min to 30 min to determine the impact on gas yield and resid conversion. The 566 °C⁺ resid conversion appears to be high (ca. 67-69%). The overall material balance for the run will

be determined upon completion of the duplicate gas analyses, but is expected to be in the mid-90% range. Elemental analyses and duplicate gas analyses are being obtained.

Dry reactor scrape samples from Runs 23oac-1, 300 CSTR, and 23dfg-1, 300 CSTR, are being analyzed for Mo content. These materials will be used in second-stage recycle tests.

Task 5 - Engineering and Economic Study

One-Liter Run 23-LA Test Series

Elemental analysis data for Run 23c-LA were used to prepare an elemental balance for the first-stage reactor. However, a large filter cake-rate prorated factor (1.99) was needed to close the poor, as-measured ash balance. This makes the results somewhat suspect. Material and elemental balance data also were obtained for Runs 23d-LA, 23f-LA and 23g-LA. In these tests, the as-measured ash and carbon balances were good. This enabled the calculation of the elemental balances to be made with low prorating factors. The elemental balance also was completed for Run 23g-LA, based on analytical data for the filtration products.

In order to complete the elemental balances for all first-stage tests, an adjustment to the measured gas rate was necessary. The adjustment was based on a comparison of the measured excess CO₂ yield for each of the runs (through 23g-LA) vs. the extent of coal deoxygenation as indicated by the liquid and solid product oxygen contents. The excess CO₂ yield is the total CO₂ yield less the CO₂ formed from the CO shift reaction. The comparison revealed that the excess CO₂ yield was much larger than would be possible from the approximately 60% coal deoxygenation achieved in Runs 23d-LA, 23f-LA, and 23g-LA. A procedure was developed to correct this inconsistency. As a result of the procedure, the measured gas rates were lowered for all the 23-LA series tests. The measured gas composition, which was believed to be accurate, was not changed (Confidential Appendix 2, Tables CA1 to CA3). Based on the above, elemental and material balances were prepared for Runs 23d-LA, 23f-LA, and 23g-LA (Confidential Appendix 2, Tables CA4 to CA6).

Net yields by boiling range cut also were determined for Runs 23d-LA, 23f-LA, and 23g-LA using simulated distillation data (Confidential Appendix 2, Tables CA7 to CA9).

Second-Stage Catalytic Upgrading

Data from Runs 23oac-1,300 CSTR, and 23dfg-1,300 CSTR, were compiled. A brief review of these results indicates that there is a significant effect of DMDS and Molyvan L catalyst decomposition on gas yields. Together, the Molyvan L and DMDS are present as 12% on feed in Run 23-LA series tests. In order to determine the net yields on distilled, filtered first-stage product resid feed, a procedure was developed to estimate yields from the decomposition of the DMDS and Molyvan L contained in the reactor feed (Confidential Appendix 2, Tables CA10 and CA11). Net yields for Runs 23oac-1, 300 CSTR, and 23dfg-1, 300 CSTR, were calculated using these estimates (Confidential Appendix 2, Tables CA12 and CA13). Net yields were determined by boiling range for Runs 23oac-1, 300 CSTR, and 23dfg-1, 300 CSTR, based on simulated distillation data (Confidential Appendix 2, Tables CA14 and CA15).

An elemental balance for Run 263J-1, 300 CSTR, (see above) was completed (Confidential Appendix 2, Table CA16).

Task 6 - Reporting

All reporting is on schedule. Bi-monthly conference calls were held as scheduled with UK/CAER and LDP. Reports documenting the calls were issued. The technical review meeting for this quarter was canceled to provide participants more time to assemble contributions to the Task 3 Topical Report. A Task 4 Work Plan was drafted and was approved for implementation.

FORECAST AND PLANS

The Task 3 Topical report will be completed. First-stage tests, designed to decrease process costs by reducing catalyst loading, solvent/dry coal ratio, and system pressure, will be continued under Task 4.

First-stage results from Runs 23d-LA, 23f-LA, and 23g-LA will be compared and evaluated. Second-stage results from Runs 23oac-1, 300 CSTR, and 23dfg-1, 300 CSTR, also will be evaluated. These data will help provide the basis for the Integrated Liquefaction System (ILS) Balance for the Novel Concept Case. Once the ILS balance is completed, work on the support system balances (e.g., upgrading of second-stage product distillates to finished products) will begin.

Net yields by boiling range will be determined for second-stage Run 263J-1, 300 CSTR, and the product distribution will be compared to the results from Runs 23oac-1, 300 CSTR, and 23dfg-1, 300 CSTR. In addition, the resid conversion rate constants for these runs will be calculated.

It is not possible to determine the exact amount of gas yield from Molyvan L. Therefore, it was recommended that in Task 4, an order of magnitude lower loading of Molyvan L or a molybdenum catalyst with much less or no hydrocarbon content should be tested. Tests using lower catalyst loading will be carried out next quarter.

REFERENCES

1. Brandes, S. D.; Winschel, R. A.; Derbyshire, F. J.; Kimber, G. M.; Anderson, R. K.; Carter, S. D.; Rantell, T.; Vego, A.; Jacques, D.; Peluso, M. "Exploratory Research on Novel Coal Liquefaction Concept, Quarterly Report for April 1 through June 30, 1997", DOE/PC 95050-34, August 1997.
2. Brandes, S. D.; Winschel, R. A.; Derbyshire, F. J.; Kimber, G. M.; Anderson, R. K.; Carter, S. D.; Rantell, T.; Vego, A.; Peluso, M. "Exploratory Research on Novel Coal Liquefaction Concept, Task 2 - Evaluation of Process Steps, Topical Report", DOE/PC 95050-22, May 1997.

TABLE 1
DISTILLATION OF FILTRATES

Run No.	Sample No.	Pot Temp. (°C)	Vapor Temp. (°C)	Vapor Pressure (torr)	Equivalent Atmospheric Cut Point		Yields (%)		Residue Softening Point (°C)	Mass Balance (%)
					(°C)	(°F)	Distillate	Residue		
23aoc-LA	23aoc-F	355	275	5.4	454	849	73.3	25.3	290	98.6
23d-LA	23d-LA	340	240	5	416	780	78.5	29.5	165	98.1
23f-LA	23f-LA	292	243	4	426	799	72	27.9	165	99.9
23g-LA	23g-LA	349	255	4	440	824	74.4	25.4	255	99.9

TABLE 2
SUMMARY OF FILTRATION TESTS

Run No.	NCF No.	Filter Membrane	Aperture, Retention (µm)	Temp (°C)	Viscosity (MPA s)	Solids in Feed (%)	Pressure (kPa)	Cake Resistivity (m/kg x 10 ¹⁰)	Total Flow After 30 min (kg / m ²)
23- LA	69	GFA	1	300	0.4	5.3	210	33	208
23a- LA	CONSOL	GFA	1	250	n/m	n/m	210	n/m	n/m
23c- LA	CONSOL	GFA	1	250	n/m	n/m	210	n/m	n/m
23d- LA	CONSOL	GFA	1	250	0.8	n/m	105	8	276
23f- LA	CONSOL	GFA	1	250	n/m	n/m	210	n/m	n/m
23g- LA	71	GFA	1	250	0.8	4.6	210	34	225

n/m = not measured

TABLE 3

**SECOND-STAGE MICROAUTOCLAVE HYDROTREATING TESTS
OF FIRST-STAGE RUN 23D-LA FILTRATE (a)**

Run No.	23d-1, 50 MA [R7-202-1]	23d-2, 50 MA [R7-202-2]	23d-3, 50 MA [R7-210-1]	Average $\pm$ Standard Deviation
Catalyst precursor	Molyvan L	Molyvan L	Molyvan L	-
Reaction time, min	60	60	60	-
Reaction Temperature, °C	440	440	440	
Mo, ppmw feed	10,028	10,082	10,043	10,051 $\pm$ 28
Products, wt % feed				
C ₁ -C ₃ gas (b)	3.2	4.5	5.0	4.2 $\pm$ 0.9
C ₄ + vapor	1.4	2.2	2.3	1.9 $\pm$ 0.5
CO + CO ₂	0.5	0.4	0.5	0.5 $\pm$ 0.1
566 °C ⁻	51.8	51.8	53.5	52.4 $\pm$ 1.0
566 °C ⁺	43.1	41.1	38.7	41.0 $\pm$ 2.3
Total	100	100	100	100
Derived values				
Resid conversion, wt % 566 °C ⁺	42.6	45.2	48.5	45.4 $\pm$ 3.0
H ₂ consumed, mg/ g MAF feed	46	37	41	41 $\pm$ 4
C ₁ -C ₃ gas, wt% MAF feed (c)	3.0	4.0	4.4	3.8 $\pm$ 0.7
SIMDIS adjustments to distillation resid weight, g	-0.064	-	-	-

- (a) 2 g samples of 23d-LA distilled filtrate used in each test. The filtrate contained 1.36 wt% ash.
- (b) Calculated from composition, pressure and temperature of the mounted reactor, using the ideal gas correlation with a compressibility factor of 1.0
- (c) Calculated from composition and volume of recovered reactor gas (some gas may be lost transferring the reactor and filling the sample piston)

TABLE 4

SUMMARY OF 300 mL CSTR RUNS

Run No.	Feedstock	From Feed Coal	Digest solvent	Digest Temp and Time	Catalyst	Hydride Ion Agent	Feed, g	Moly-van L, g	DMDS Feed, g	Reaction Temp at Time
23dfg-1, 300 CSTR [R7-223-1]	23 La filtrate resid blend	Black Thunder	Reilly AO	375 °C for 60 min	KOH	E	93	11.6	7.1	440 °C for 60 min
263J-1, 300 CSTR [R7-239-1]	Wilsonville Run 263J interstage product	Black Thunder	NA	NA	NA	NA	100	12.3	7.4	440 °C for 60 min

NA = Not Available

TABLE 5

HYDROTREATING RESULTS, 300 mL CSTR RUNS

Run No.	Feedstock	566 °C ⁺ in Feedstock, wt % MAF Feed	566 °C ⁺ Resid Conv., wt % MAF Feed	C ₁ -C ₃ Gas Yield, wt % MAF Feed	C ₁ -C ₃ Gas Yield per g Resid Converted	H ₂ Uptake by Diff., mg/g MAF Feed (a)	H ₂ Uptake per g Resid Converted	H ₂ Balance, (Uptake Basis), % (b)	Fixed Carbon Ratio, Out/In(c)
23dfg-1, 300 CSTR [R7-223-1]	23 La filtrate resid blend	80.9	65.0	15.2	28.9	33	63	98	0.7
263J-1, 300 CSTR [R7-239-1]	Wilsonville Run 263J interstage product	66.5	43.9	18.4	63.0	27	92	101	1.0

(a) Includes the hydrogen associated with the decomposition of DMDS.

(b) Excludes hydrogen gas calculated to be merely passing through the reactor.

(c) Fixed carbon in product recovered from the reactor (excludes distillate) divided by fixed carbon in the feed.

TABLE 6**300 mL CSTR TESTS OF FIRST-STAGE FILTRATES (a)**

	23oac-1, 300 CSTR	23dfg-1, 300 CSTR
566 °C ⁻ fraction of feed, wt %	6	24.6
566 °C ⁺ resid conversion, wt %	57	65
H/C wt ratio of dry resid 566 °C fraction	0.039	0.042
C ₁ -C ₃ gas yield, wt % MAF feed	17.3	15.2
H ₂ uptake, mg/g MAF feed	29	33
Fixed carbon recovered in resid, out/in	0.82	0.67

(a) Conditions: 440 °C for 60 minutes, with 1% Mo (Molyvan L)

TABLE 7**ELEMENTAL ANALYSES OF DARo-1, 300 CSTR, "REACTOR DRY SCRAPE" PRODUCT**

Sample	C, wt %	H, wt %	N, wt %	S, wt %	O, wt % diff.	ash, wt %	Total, wt %	H/C Ratio
whole reactor dry scrape	85.27	7.00	1.40	1.12	3.14	2.07	100.00	0.082
566 °C ⁻ distillate fraction	91.53	7.15	1.01	0.03	0.11	0.17	100.00	0.078
566 °C ⁻ distillate fraction, normalized to 100% recovery	82.51	8.81	1.57	0.43	6.65	0.03	100.00	0.107
566 °C ⁺ residual fraction	89.44	4.27	1.14	2.16	-2.17	5.16	100.00	0.048

TABLE 8**ELEMENTAL ANALYSES OF 23oac-1, 300 CSTR, "REACTOR DRY SCRAPE" PRODUCT**

Sample	C, wt %	H, wt %	N, wt %	S, wt %	O, wt % diff	Ash, wt %	Total, wt %	H/C Ratio
whole reactor dry scrape	84.52	6.27	1.51	1.15	0.03	6.52	100.00	0.074
566 °C ⁻ distillate fraction	82.96	8.34	1.46	0.04	7.20	0.00	100.00	0.101
566 °C ⁻ distillate fraction, normalized to 100% recovery	88.07	10.44	1.67	0.29	2.56	-3.03	100.00	0.119
566 °C ⁺ residual fraction	81.91	3.20	1.39	1.78	(1.83)	13.55	100.00	0.039

TABLE 9**ELEMENTAL ANALYSES OF 23dfg-1, 300 CSTR, "REACTOR DRY SCRAPE" PRODUCT**

Sample	C, wt %	H, wt %	N, wt %	S, wt %	O, wt % diff	Ash, wt %	Total, wt %	H/C Ratio
whole reactor dry scrape	82.18	6.84	1.665	1.075	2.52	5.72	100.00	0.083
566 °C ⁻ distillate fraction	88.98	7.08	1.35	0.04	2.51	0.04	100.00	0.080
566 °C ⁻ distillate fraction, normalized to 100% recovery	83.08	9.73	2.03	0.12	7.24	-2.19	100.00	0.117
566 °C ⁺ residual fraction	81.11	3.44	1.24	2.20	-3.02	15.03	100.00	0.042

TABLE 10**ELEMENTAL ANALYSES OF 263J-1, 300 CSTR, "REACTOR DRY SCRAPE" PRODUCT**

Sample	C, wt %	H, wt %	N, wt %	S, wt %	O, wt % diff	Ash, wt %	Total, wt %	H/C Ratio
whole reactor dry scrape	68.47	4.56	0.60	2.72	0.36	23.30	100.00	0.067
566 °C- distillate fraction	86.10	8.28	1.03	0.03	4.56	0.00	100.00	0.096
566 °C- distillate fraction, normalized to 100% recovery	91.78	10.91	-0.02	1.52	0.17	-4.36	100.00	0.119
566 °C+ residual fraction	59.76	2.19	0.83	3.16	0.43	33.63	100.00	0.037

TABLE 11**FIRST-STAGE MICROAUTOCLAVE TESTS AT REDUCED SOLVENT/COAL RATIOS**

Black Thunder Mine Coal
375 °C, 60 min

Run No.	Solvent (a) /dry coal	Catalyst (b), mmole/kg dry coal	Coal Conversion, wt%
242	1.5	83	88.8
245	1.25	83	92.1
246	1.0	83	88.4
247	1.0	50	89.4

(a) solvent: Reilly Industries anthracene oil
(b) catalyst: KOH

TABLE 12

SIMULATED DISTILLATION OF FIRST-STAGE LIGHT DISTILLATE OIL

Sample No.		23c-LA	23d-LA	23f-LA	23g-LA	23h-LA
Found Recovery (%)		87.5	91.7	96.8	98.5	97.5
b pt (°C)	b pt (°F)	amount in fraction (%)				
<150	<302	3.8	2.5	2.6	2.7	2.6
150-200	302-392	8.0	8.5	9.1	9.0	8.8
200-250	392-482	30.9	33.3	32.1	32.9	31.7
250-300	482-572	10.9	11.9	14.2	14.4	13.5
300-350	572-662	20.4	20.7	21.8	22.0	22.6
350-400	662-752	10.5	11.7	12.4	12.9	12.9
400-450	752-842	2.4	2.7	3.2	3.0	3.5
450-500	842-932	0.6	0.4	0.9	1.2	1.0
500-550	932-1022	0.0	0.0	0.5	0.4	0.9
550-600	1022-1112	0.0	0.0	0.0	0.0	0.0

bpt (°C)	bpt (°F)	amount bpt <T° (%)				
<150	<302	3.8	2.5	2.6	2.7	2.6
<200	<392	11.8	11.0	11.7	11.7	11.4
<250	<482	42.7	44.3	43.8	44.6	43.1
<300	<572	53.6	56.2	58.0	59.0	56.6
<350	<662	74.0	76.9	79.8	81.0	79.2
<400	<752	84.5	88.6	92.2	93.9	92.1
<450	<842	86.9	91.3	95.4	96.9	95.6
<500	<932	87.5	91.7	96.3	98.1	96.6
<550	<1022	87.5	91.7	96.8	98.5	97.5
<600	<1112	87.5	91.7	96.8	98.5	97.5