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CATALYSTS AND PROCESS DEVELOPMENTS FOR TWO-STAGE LIQUEFACTION

Report No. 56

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EXECUTIVE SUMMARY

Research under way in this project centers upon developing and evaluating catalysts and process improvements for coal liquefaction in the two-stage, close-coupled catalytic process. The project is being carried out under contract to the United States Department of Energy.

As discussed in the previous quarterly report, promising results were obtained by liquefying Illinois No. 6 bituminous and Black Thunder subbituminous coals using oil-soluble catalysts Molyvan L and molybdenum octoate.

In this quarter, the liquefaction of Black Thunder coal was continued. Runs were made in catalytic/thermal (C/T) mode with supported AMOCAT™ 1C (NiMo) and AMOCAT™ 1B (Mo) catalysts. Although the initial performance in these runs was good (90% conversion with no resid production), both catalysts deactivated rapidly. Spent catalysts showed severe coke deposition as well as formation of a calcium-rich shell on the catalyst surface. Overall, C/T liquefaction is not a good process option for Black Thunder coal.

BACKGROUND

National energy policy requires that a reliable supply of reasonable-cost transportation fuels be available for both national and economic security. Unfortunately, the United States is importing about 50% of its petroleum supplies. Projections suggest an even greater reliance on imported oil in the future. An alternative to imported oil must be found if the United States is to achieve energy independence. Synthetic fuel from other fossil fuels is an alternative to imported oil. Coal is the most abundant of all fossil fuels, and the United States has one of the largest reserves of coal deposits in the world. The conversion of coal to transportation fuels would thus appear to be a necessary element of a serious synthetic fuels energy policy.

The conversion of coal to synthetic fuels through both direct and indirect routes was practiced commercially in Germany during World War II. Presently, South Africa produces transportation fuels through the indirect liquefaction of coal. Both technologies are uneconomic compared with producing transportation fuels from crude oil, but are or were practiced because of political considerations. Current assessments are that direct liquefaction offers the best chance for producing transportation fuels from coal at an economically viable cost.

There are no commercial direct coal liquefaction plants in operation anywhere in the world. However, several direct coal liquefaction technologies, such as the H-coal and the Exxon donor solvent processes, have been extensively studied. Currently, the most promising direct coal liquefaction route is the two-stage, close-coupled catalytic liquefaction process being studied at the Advanced Two-Stage Coal Liquefaction Facility at Wilsonville, Alabama. This facility serves as a research and demonstration plant to test new process options and catalysts for converting coal on a 6 tons of coal per day scale. Projected costs for direct coal liquefaction have dropped from over $90 per barrel in 1981 to about $36 per barrel today primarily because of improvements tested and demonstrated at Wilsonville. While the cost is still high, direct coal liquefaction by the two-stage process continues to show improving economics and viability as a commercial technology.

The two-stage, close-coupled liquefaction process is a hydrotreating process that is conducted in two reactors in series with minimal residence time between reactors. This has been designated as "close-coupling." The first reactor is the primary coal conversion reactor where coal, a recycled coal-derived solvent, and hydrogen react to produce a hydrogenated coal product slate. The first reactor may be operated as either a catalytic or a thermal (non-catalytic) stage. The second reactor is operated with catalyst and serves to upgrade the first stage products by removing oxygen, nitrogen, and sulfur heteroatoms, increasing the hydrogen content of the products, and further reducing the boiling point of the products. The process is optimized to produce a maximum distillate product slate, optimum hydrogen utilization, high coal conversion, and sufficient coal-derived solvent for recycle. While the reactions taking place in both reactors are similar, each reactor sees a different feedstock because of the ongoing conversion. It is thus probable that each reactor will need to be optimized to produce the best overall liquefaction process. Two obvious areas for improvement to the two-stage process are improved catalysts and improved process options to reduce hydrogen consumption.

In view of the aforementioned considerations, the Department of Energy funds research programs designed to improve the economics and viability of direct two-stage coal liquefaction processes. This Department of Energy-funded research program is directed toward finding better catalysts and alternative process options to improve the overall liquefaction process. Better catalysts would improve liquefaction performance by increasing coal conversion, distillate yields, and/or hydrogen utilization. Ideally, catalysts should have both good hydrogenation activity and good activity maintenance. Process improvements include the removal of heteroatoms from coal before liquefaction to reduce hydrogen requirements and beneficiation before liquefaction to remove unreactive ash and other components from the coal.

PROGRAM OBJECTIVES

The overall objectives of this program are to develop and evaluate new catalyst formulations and process improvements for use in two-stage, close-coupled coal liquefaction. Improved catalysts are needed that can lead to higher coal conversion, distillate yields, or better hydrogen utilization. First stage catalysts are optimized primarily for enhanced coal conversion, and second stage catalysts for coal-resid upgrading. Process improvements include pretreatment to lower the oxygen content and unreactive components in coal before liquefaction. The program requires close cooperation between Amoco Oil and other Department of Energy-sponsored research efforts, particularly the Wilsonville Advanced Two-Stage Coal Liquefaction Facility.

PROJECT DESCRIPTION

Our technical approach to this project includes developing first and second stage coal liquefaction catalysts and testing the effects of selected pretreatment procedures on subsequent liquefaction. The procedures to be evaluated are methods for removing oxygen from coals and the beneficiation of coals to reduce ash and unreactive components prior to their liquefaction. Partially removing oxygen from coals prior to liquefaction has the potential of reducing hydrogen requirements and prolonging catalyst life. Beneficiation and pretreatment to reduce ash and unreactive components increases reactor throughput, improves catalyst life, and reduces the losses of product carried along with the unconverted coal and mineral matter rejected from the process.

Our overall approach to catalyst development is to prepare new formulations and modify existing refinery catalysts as candidates for either first or second stage coal liquefaction catalysts. First stage catalysts need to be accessible to large coal fragment molecules and promote both further fragmentation and hydrogen transfer to radical fragments. Second stage catalysts need to promote aromatics saturation and heteroatom removal as well as reduce molecular weight to produce usable distillable liquids.

Coal liquefaction catalysts serve to promote both molecular weight reduction and hydrogen transfer to coal. We will screen candidate catalysts in fixed-bed reactors for upgrading a coal-resid and Panasol mixture. The fixed-bed unit allows accurate measurements of catalyst aging. Catalysts that show good second stage activity and activity maintenance for upgrading the coal-resid mixture will also be good candidates for further testing as first stage catalysts. Coals to be used as feedstocks are Illinois No. 6 (bituminous), Black Thunder (subbituminous), and Martin Lake (lignitic) coals. Resids are coal-derived liquids from the Wilsonville Coal Liquefaction Facility. Panasol (byproduct from naphtha reforming) is used as a solvent. Base case runs using a standard catalyst combination are being done to provide meaningful comparisons. Our approach allows accurate comparison of the relative effects of various pretreatments, coals, solvents, or catalysts on the liquefaction process.

Seven tasks have been identified to bring about the above objectives. Task 1 called for the completion of a detailed project management plan, which was issued. Task 2 is a general laboratory support task for the coal liquefaction tests. It provides the analytical support for characterizing most of the liquefaction samples and catalysts being tested. It includes microautoclave tests to more fully understand the results of larger scale experiments and qualification of solvents used or produced during liquefaction. Task 3.1 is directed toward determining the liquefaction behaviors of standard coal samples and comparing them with the beneficiated or pretreated samples. Pretreatment procedures emphasize methods for oxygen removal and beneficiation to remove ash and inert materials.

Catalyst development and testing are being done under the three parts of Task 3.2. Task 3.2.1 is used to test a series of candidate first stage catalysts and the best second stage catalysts as first stage catalysts with Illinois No. 6 bituminous and Black Thunder subbituminous coals in our continuous pilot plant unit. The objective is to identify a high-activity first stage catalyst.

In Task 3.2.2 up to 50 candidate catalysts were evaluated with an Illinois No. 6 coal-resid and Panasol mixture to identify the five best second stage catalysts. Novel and conventional catalysts have been prepared and tested. Several of Sandia's hydrous metal oxide catalysts have been tested, and two additional tests are planned under Task 3.2.3. Consultation to Sandia on preparing their catalyst in a suitable form is also being provided.

Other process developments are being evaluated under Task 3.3. Decarboxylation of low-rank coals by hydrothermal and caustic- or catalyst-aided treatments will be tested.

RESULTS AND DISCUSSION

During this fourteenth quarter of the contract, experimental work on Task 3.2.1 (first stage catalyst) continued by liquefying Black Thunder bituminous coal using supported AMOCAT™ catalysts. A run was also made with Sandia hydrous titanate catalysts under Task 3.2.3; however, results haven't been analyzed and will be reported in the next quarterly. Other detailed reports of the results and efforts for this quarter are given by task below.

Task 2: Laboratory Support and Microautoclave Runs

No activity limited to this task was undertaken; results of laboratory analyses are reported under the remaining tasks when applicable.

Task 3.1: Feedstock Effects

A run aimed at oxygen rejection/conversion with Black Thunder coal feed in a low pressure pretreatment stage is complete and will be discussed in the next quarterly report.

Task 3.2.1: First Stage Catalysts

The objective of experimentation in this subproject was to evaluate first stage catalysts for liquefaction of Illinois No. 6 coal and Black Thunder subbituminous coal using a two-stage, continuous-flow unit.

First Stage Catalyst Tests with Black Thunder Coal

The tests that were performed used a C/T configuration with AMOCAT™ 1C catalyst or AMOCAT™ 1B catalyst in the first stage without dispersed molybdenum catalyst. C/T liquefaction of Black Thunder coal was studied in Wilsonville Run 260, but the second stage temperature was too low because of furnace limits. Iron oxide and a sulfiding agent, TPS-20, or iron sulfate were added to the feed for most of these tests.

EXPERIMENTAL: FEEDSTOCK DESCRIPTIONS AND TEST CONDITIONS

Analyses of the Black Thunder subbituminous coal feed, obtained from the mine and ground at the University of North Dakota, are given in Table 1. Different batches of coal were used in the Black Thunder reference test (FCL-126) and first stage catalyst tests (FCL-135). Liquefaction solvent was a blend of V-1074 and V-203 liquids from Wilsonville Run 258. The analyses of the two batches (FSN-113 and FSN-136) are given in Table 2. The bench-scale liquefaction runs were made in AU-135L continuous, two-stage pilot plant with 1-liter stirred autoclave reactors. Feed slurries consisted of 33/67 mixtures of coal/liquefaction solvent. AMOCAT™ catalysts were presulfided with 8% hydrogen sulfide in hydrogen or with TPS-20 in liquefaction solvent before use. Product solubility was determined by millipore filtration, distillate yields were determined by modified D-86 and D-1160 distillation, and distilled fractions were analyzed for C, H, S, N, O, and aromatic carbon by ^{13}C NMR. Unconverted solids were analyzed for metals by ICP (inductively coupled plasma spectroscopy).

Improvements in laboratory procedures have reduced the time and cost of analyses of pilot plant products. Three distillations and three large-scale preparations of insolubles can be completed at the same time, which allows workup of three pilot plant samples in one day. One sample a day was analyzed at the beginning of this contract. Extra laboratory time is available for batch studies of liquefaction fundamentals, such as the one described at the end of this report. Use of tetrahydrofuran (THF), which is very expensive, has been cut in half by using toluene for preparation of large-scale insolubles for analysis of ash constituents. The modified procedure gives identical ash yields and acceptable speed and ease of analysis.

High-space velocity tests with Black Thunder coal and dispersed catalysts have been completed and will be discussed in the next quarterly report. A run evaluating a powdered Sandia hydrous titanate catalyst is complete and will be discussed in the next quarterly report.

<u>Black Thunder Coal: C/T with AMOCAT™ 1C Catalyst in Stage 1</u>

Process conditions and product analyses are given in Tables A-1, A-2, and A-3 of the Appendix. The first week of the run was very smooth; then a few small upsets occurred that may have affected the end of the run. The decline in performance was not immediate and did not seem reasonable in view of the minor nature of the process upsets. After the next run, which was very short, a disintegrated bearing in the first stage stirrer was discovered. It is likely that inadequate stirring could have partly been responsible for poor performance at the end of this run.

RESULTS

Catalyst activity was good for the initial part of the run and probably reflects true performance of this system. A period late in the run with low catalyst activity is also included for completeness. Yields are compared with those from the Black Thunder reference run (C/C) and a dispersed catalyst period ("slurry," Molyvan L/iron oxide/TPS-20) in Table 3. A complete set of yields is given in Table A-4 of the Appendix.

Initial results from the C/T run with AMOCAT™ 1C catalyst showed conversion of all of the resid from the coal and some of the resid in the slurry solvent (-32% resid yield). Excess conversion could be corrected by operation at shorter residence times or lower temperatures. Light hydrocarbon gas yields were quite high, 16%, but could be decreased with lower severity. Coal conversion was excellent at 94%.

Late in the run, after the catalyst had deactivated, the resid yield had increased, coal conversion had dropped to 87%, but hydrocarbon gas yields were still high (18%). Gas yields must be strongly influenced by the thermal second stage. The product analyses for both early and late periods in the run are in Table 4.

Product analyses are also compared with those from the Black Thunder reference run and the earlier dispersed Mo catalyst run reported in the last quarterly, in Table 5. The hydrogen and heteroatom content of the product was about the same as with the dispersed Mo catalyst, even for C/T periods where catalyst deactivation was severe. However, deoxygenation was better initially in the C/T run, and about the same as from the dispersed Mo at the end of the C/T run when the catalyst was deactivated. Supported catalyst in stage 1 (AMOCAT™ 1C in the C/C and C/T runs) appeared to increase deoxygenation but not have much effect on hydrogen addition, desulfurization, or denitrogenation. The supported catalyst in stage 2 (AMOCAT™ 1C in the C/C run) provided additional deoxygenation, hydrogen addition, desulfurization, and denitrogenation.

Overall, the results from the early part of the C/T run with AMOCAT™ 1C catalyst were outstanding and showed excess capacity for resid conversion. It may be possible to operate this type of process at much higher coal throughput. The severe catalyst deactivation after one week was disturbing. Deactivation may have been caused by poor stirring in the first stage, but could be a reproducible feature of this type of process.

Black Thunder Coal: C/T with AMOCAT™ 1B Catalyst in Stage 1

Molybdenum catalysts are more resistant to deactivation than nickel/molybdenum catalysts for certain heavy feedstocks. Molybdenum catalysts are initially less active for hydrogenation and heteroatom removal, but aged molybdenum catalysts often retain higher activity than the corresponding nickel/molybdenum catalysts. Results from the previous C/T run were very promising initially, but the catalyst deactivated severely after about a week. This run with AMOCAT™ 1B molybdenum catalyst was an attempt to maintain higher, more stable activity for a longer period of time. Instead, product from this run deteriorated sharply after three days. When the first stage reactor was opened, it was obvious that the stirrer bearing had disintegrated and the reactor contents were not stirred at all during this run. Process conditions and analyses are given in Tables A-5 through A-7 of the Appendix.

A second C/T run with AMOCAT™ 1B catalyst was completed early this quarter. We had hoped that AMOCAT™ 1B (Mo) catalyst would show more stable activity, although initial activity was expected to be low. Process conditions and analyses are given in Tables A-9 through A-11 of the Appendix. Initial results from two runs with AMOCAT™ 1B catalyst are given in Table 6, and both show poorer conversion to THF solubles and poorer resid conversion than with the NiMo catalyst. The performance of AMOCAT™ 1B improved somewhat when ferrous sulfate was used as the iron additive instead of iron oxide/sulfiding agent. Yields for all sample periods are reported in Tables A-8 and A-12 of the Appendix. Product elemental analyses from all three tests were comparable early in the run (Table 7).

The AMOCAT™ 1B catalyst did not show more stable activity as the catalyst aged, which is shown in Table 8. The first period was operated with a second stage temperature of 800°F. No supported catalyst was present in the second stage. The second stage temperature was decreased to 780°F in a successful attempt to reduce gas yields, and distillate yields also dropped, which was expected. As the run continued, resid yields increased from 12% to 27%. Product hydrogenation and heteroatom contents remained relatively constant throughout the run (Table 9). Analyses of spent catalyst from this run showed severe coke deposition as well as formation of a calcium-rich shell on the catalyst surface. AMOCAT™ 1B catalyst is not resistant to this important mode of fouling from liquefaction of Black Thunder coal.

Effect of Solvent Hydrogenation on Conversion

Coal conversion to THF soluble material was highest in C/T runs when the catalyst was fresh. Improved conversion could be from good hydrogenation of the slurry solvent by active catalyst. The role of solvent hydrogen content on conversion of Black Thunder Coal was tested in two batch experiments. Distillate fractions (650-935°) from Black Thunder coal with H/C ratios of 1.11 and 1.22 were used to liquefy Black Thunder coal at a 1:2 coal:solvent ratio. The hydrogen-rich solvent gave 78% conversion and 0.7% preasphaltenes (1 hr, 790°F, 2000 psig). The hydrogen-poor solvent gave only 67% conversion and 7.3% preasphaltenes, a ten-fold increase in preasphaltenes, at the same conditions. Good conversion of preasphaltenes appears to be the key to good conversion of Black Thunder coal to liquid, and good hydrogen donor strength is essential. Prehydrogenation of the slurry solvent may give higher conversion at high space velocity, which has been difficult to achieve with this feedstock.

Analyses of Spent Catalyst Samples

Initial performance of all the C/T runs with Black Thunder coal was excellent. No resid was produced, and the coal conversion was over 90%. To prolong the period of high activity, spent catalyst samples were studied to determine the cause of deactivation. Samples from Professor Haynes (University of Wyoming) showed a layer of calcium on the surface of the catalyst. Haynes noted that coke could be burnt off only after the catalyst pellets were broken, suggesting that the calcium layer hindered diffusion. Our spent AMOCAT™ 1B catalyst was easily decoked at 860°F without breaking the pellets, but they also showed a thinner, discontinuous Ca shell. Because Ca contributes to catalyst deactivation, SO_2-demineralization should improve the catalyst life.

Our AMOCAT™ 1B-1C catalysts contained more coke than the sample from Haynes (29% vs 11%, Table 10). Haynes' catalyst was used at 800°F compared with 780°F for our test. Our spent catalyst contained more coke at 780°F than at 790°F (Black Thunder reference run), possibly from poorer preasphaltene conversion at the lower temperature. Preasphaltene conversion can be improved by hydrogenation of the slurry solvent, as shown in the previous section of this report. Use of a prehydrogenated solvent may also extend catalyst life.

Task 3.2.2: Second Stage Catalysts

No activity in this task during the quarter.

Task 3.2.3: Hydrous Metal Oxide Catalysts

A powdered version of Sandia's hydrous metal titanate catalyst was tested and results are being analyzed.

CONCLUSIONS AND FUTURE WORK

Several tests were done with C/T mode using AMOCAT™ 1C and 1B in the first stage. The results indicated good performance initially with conversions over 90% and no resid; however, both catalysts deactivated rapidly and showed a layer of calcium on the surface of the spent catalyst. Overall, C/T liquefaction is not a good process for Black Thunder coal.

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TABLE 1

COAL ANALYSES

Coal	Black Thunder Subbituminous	
Batch Number	FCL-126	FCL-135
As Received, Wt% H_2O	23.60	23.31
Dry, Wt%		
C	69.60	69.05
H	5.01	5.23
N	1.07	.90
S	.46	.50
O (Difference)	17.14	17.64
Ash	6.72	6.68
Wet, Wt%		
Fe	.13	.18
Na	.03	.05
K	.03	.02
Ca	.88	.88
Mg	.11	.15
Al	.31	.42
Ti	.02	.03
Si	.33	.57

TABLE 2

SOLVENT ANALYSES

Wilsonville Run	258	
Wilsonville Coal	Black Thunder Subbituminous	
Batch Number	FSN-113	FSN-136
Elemental Analyses, Wt%		
C	89.54	89.25
H	8.28	8.26
N	.80	.75
S	.05	.05
O	1.33	1.69
Distillation, Wt%		
IBP-650°F	8.83	8.55
650-975°F	53.70	
975+°F	37.46	
650-935°F	50.34	49.92
935+°F	40.83	41.52
Solubility, Wt%		
THF Insolubles	.10	.22
Toluene Insolubles	1.31	.99
Hexane Insolubles	15.33	4.31

TABLE 3

C/T LIQUEFACTION OF BLACK THUNDER SUBBITUMINOUS COAL: DISTILLED PRODUCT YIELDS

Process	C/C Amocat 1C	C/T Amocat 1C	Slurry
Residence Time, Hours	3	3	3
Stage 1 °F	790	780	800
Stage 2 °F	740	800	800
Catalyst Age, Hours	190	110	
Molyvan L, ppm Mo Mo Octoate, ppm Mo Iron Oxide, Wt% TPS-20, Wt%	0 0 0 0	0 0 1.0 2.0	96 0 1.0 2.0
Yields, Wt% of MAF Coal			
C1-C3	12.3	(16)	15.1
C4-360°F	10	31	26
360-650°F	48	53	47
650-935°F	19	11	-8
935°F+	-18	-32	-4
C_4-935°F	77	94	65
Conversion	84.8	93.7	90.2
Hydrogen	-9.0	-8.3	-6.5

TABLE 4

C/T LIQUEFACTION OF BLACK THUNDER SUBBITUMINOUS COAL: YIELDS WITH AMOCAT™ 1C CATALYST IN STAGE 1

Process	C/T	C/T
Stage 1 Catalyst	AMOCAT™ 1C	AMOCAT™ 1C
Stage 1 °F	780	780
Stage 2 °F	800	800
Catalyst Age, Hours	110	280
Fe_2O_3, Wt% TPS-20, Wt%	1.0 2.0	1.0 2.0
Yields, Wt% of MAF Coal		
C1-C3	(16)	17.8
C4-360°F	31	21
360-650°F	53	23
650-935°F	11	12
935°F+	-32	0
C_4-935°F	94	57
Conversion	93.7	87.4
Hydrogen	-8.3	-6.2

TABLE 5

C/T LIQUEFACTION OF BLACK THUNDER SUBBITUMINOUS COAL: PRODUCT ANALYSES

Process	C/C Amocat 1C	C/T Amocat 1C	Slurry
Residence Time, Hrs.	3	3	3
Reactor 1 Temp., °F	790	780	800
Reactor 2 Temp., °F	740	800	800
Catalyst Age, Hrs.	190	200	
Molyvan L, ppm Mo Mo Octoate, ppm Mo Iron Oxide, Wt% TPS-20, Wt%	0 0 0 0	0 0 1.0 2.0	96 0 1.0 2.0
Wt% Analyses			
<u>360-650°F</u>			
Aromatic Carbon		43	44
H/C	1.51	1.45	1.45
N	.29	.52	.52
O	1.4	1.7	3.1
<u>650-935°F</u>			
Aromatic Carbon		58	58
H/C	1.29	1.17	1.16
S	.01	.01	.28
N	.42	.56	.61
O	.5	.9	1.2
<u>935°F+, Solids</u>			
H/C	.91	.79	.80
S	.14	.56	.38
N	.96	1.05	1.26

C = With AMOCAT™ 1C catalyst T = Without AMOCAT™ 1C catalyst

TABLE 6

C/T LIQUEFACTION OF BLACK THUNDER SUBBITUMINOUS COAL:
YIELDS WITH AMOCAT™ 1C AND AMOCAT™ 1B IN THE FIRST STAGE

Process	C/T	C/T	C/T
Stage 1 Catalyst	AMOCAT™ 1C	AMOCAT™ 1B	AMOCAT™ 1B
Stage 1 °F	780	780	780
Stage 2 °F	800	800	800
Catalyst Age, Hours	110	110	120
Fe_2O_3, Wt% TPS-20, Wt% $FeSO_4/H_2O$, Wt%	1.0 2.0	1.0 2.0	 2.7
Yields, Wt% of MAF Coal			
C1-C3	(16) est.	14	17
C4-360°F	31	13	10
360-650°F	53	36	47
650-935°F	11	9	8
935°F+	-32	3	-6
C_4-935°F	94	58	65
Conversion	93.7	90.3	89.4
Hydrogen	-8.3	-5.4	-6.6

TABLE 7

C/T LIQUEFACTION OF BLACK THUNDER SUBBITUMINOUS COAL:
ANALYSES WITH AMOCAT™ 1C AND AMOCAT™ 1B IN THE FIRST STAGE

Process	C/T	C/T	C/T
Stage 1 Catalyst	AMOCAT™ 1C	AMOCAT™ 1B	AMOCAT™ 1B
Stage 1 °F	780	780	780
Stage 2 °F	800	800	800
Catalyst Age, Hours	110	110	120
Fe_2O_3, Wt% TPS-20, Wt% $FeSO_4/H_2O$, Wt%	1.0 2.0	1.0 2.0	 2.7
360-650°F, Wt%			
Aromatic C	42.5	43.5	44.0
H/C	1.45	1.44	1.45
N	.52	.45	.47
O	1.7	4.0[1]	4.0
650-935°F			
Aromatic C	57.5	54.5	56.6
H/C	1.17	1.18	1.17
S	.01	.04	.03
N	.56	.49	.54
O	.9	1.2	1.1
935+°F, Solids			
H/C	.79	.81	.85
S	.56	.37	.88
N	1.05	1.20	1.31

[1]This fraction contained some dioxane that was used in the product workup.

TABLE 8

C/T LIQUEFACTION OF BLACK THUNDER SUBBITUMINOUS COAL: PRODUCT YIELDS SHOWING CATALYST DEACTIVATION

Process	C/T	C/T	C/T	C/T
Stage 1 Catalyst	Am. 1B	Am. 1B	Am. 1B	Am. 1B
Stage 1 °F	780	780	780	780
Stage 2 °F	800	780	780	780
Catalyst Age, Hours	120	190	260	320
$FeSO_4/H_2O$, Wt% $FeSO_4/CH_3OH$, Wt% $FeSO_4/H_2O/CH_3OH$, Wt%	2.7	2.7	 1.1	 2.7
Yields, Wt% of MAF Coal				
C1-C3	17.3	11.9	(11)	10.3
C4-360°F	10	8	6	4
360-650°F	47	29	31	26
650-935°F	8	15	7	3
935°F+	-6	12	17	27
C_4-935°F	65	52	44	33
Conversion	89.4	90.8	86.9	88.4
Hydrogen	-6.6	-5.6	-5.6	-4.8

TABLE 9

C/T LIQUEFACTION OF BLACK THUNDER SUBBITUMINOUS COAL: PRODUCT ANALYSES SHOWING CATALYST DEACTIVATION

Process	C/T	C/T	C/T	C/T
Stage 1 Catalyst	Am. 1B	Am. 1B	Am. 1B	Am. 1B
Stage 1 °F	780	780	780	780
Stage 2 °F	800	780	780	780
Catalyst Age, Hours	120	190	260	320
$FeSO_4/H_2O$, Wt%	2.7	2.7		
$FeSO_4/CH_3OH$, Wt%			1.1	
$FeSO_4/H_2O/CH_3OH$, Wt%				2.7
360-650°F, Wt%				
Aromatic C	44.0	44.0	42.5	43.6
H/C	1.45	1.45	1.44	1.45
N	.47	.46	.45	.45
O	4.0[1]	4.5	4.0	4.5
650-935°F				
Aromatic C	56.5	55.0	54.4	55.4
H/C	1.17	1.21	1.19	1.19
S	.03	.04	.04	.03
N	.54	.55	.50	.55
O	1.1	1.4	1.3	1.4
935+°F, Solids				
H/C	.85	.83	.84	.82
S	.88	.80	.27	.72
N	1.31	1.35	1.24	1.32

[1]This fraction contains some dioxane that was used in the product workup.

TABLE 10

SPENT FIRST STAGE CATALYSTS: BLACK THUNDER SUBBITUMINOUS COAL

Catalyst	Amocat 1C	Amocat 1C	Amocat 1B	Amocat 1C	Amocat 1B
Temperature, °F	790	780	780	Fresh	Fresh
Run	51-218	135-23	135-25		
Run Length, Days	24	17	17		
Elemental Analyses, Wt%					
Primary Metals					
Mo	5.30	7.50		9.10	10.30
Ni	1.19	1.63		2.22	.24
Al	21.80	34.60		35.40	--
Si	1.82	1.64		1.39	1.10
Deposits					
C	24.08	28.71	29.24		
H	1.65	1.58	1.61		
S	9.62?	4.88	4.00	.20	.23
Ti	.28	.03		.00	--
Fe	6.00	.79		.02	.02
Na	.41	.74		.08	.09
Mg	.17	.11		.00	.00
Ca	.34	.44		.00	.00
Pore Properties					
Vol. <1200 Å Diam., cc/g	.14	.17	.17	.59	.63
BET Surface Area, m^2/g	77	76	67	200	194

APPENDIX

TABLE A-1

BLACK THUNDER SUBBITUMINOUS COAL: C/T LIQUEFACTION WITH AMOCAT 1C CATALYST IN STAGE 1

	1.0% Fe2O3						0.4%	1.4%		1.0%	SOLVENT
	FSN-113		FSN-136								
SAMPLE PERIOD	2	3	4	5	6	9	11	12	13	16	18
HOURS ON STREAM	39.5	63.5	87.1	112	134	201	243	263	288	351	396
WT FRACTION COAL IN FEED	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.00
1ST-STAGE TEMPERATURE, F	780	780	780	780	780	780	780	780	780	780	780
SPACE TIME, MIN.	88.9	83.5	78.3	93.4	96.7	98.4	67.9	81.8	71.8	75.4	69.7
SEVERITY, STTUS.	16.8	15.8	14.8	17.7	18.3	18.6	12.9	15.5	13.6	14.3	13.2
COAL;CAT LHSV	0.79	0.84	0.89	0.75	0.72	0.71	1.03	0.85	0.97	0.93	0.00
SOLVENT;CAT LHSV	2.05	2.18	2.33	1.95	1.88	1.85	2.69	2.22	2.53	2.41	3.93
2ND-STAGE TEMPERATURE, F	800	800	800	800	800	800	800	800	800	600	800
SPACE TIME, MIN.	88.9	83.5	78.3	93.4	96.7	98.4	67.9	81.8	71.8	75.4	69.7
SEVERITY, STTUS.	29.9	28.1	26.4	31.5	32.6	33.2	22.9	27.6	24.2	0.03	23.5
COAL;CAT LHSV	.	.	.	.	.	.	.	.	.	.	.
SOLVENT;CAT LHSV	.	.	.	.	.	.	.	.	.	.	.
COMBINED SEVERITY, STTUS	46.8	43.9	41.2	49.2	50.9	51.8	35.8	43	37.8	14.3	36.7
SYSTEM PRESSURE, PSIG	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500
1000 SCFH H2 PER BBL FEED.	7.08	6.86	6.95	8.50	10	9.56	6.92	7.98	7.33	7.03	6.24

TABLE A-2

BLACK THUNDER SUBBITUMINOUS COAL: C/T LIQUEFACTION WITH AMOCAT 1C CATALYST IN STAGE 1

	1.0% Fe2O3						-0.4%	1.4%		-1.0%	SOLVENT
	FSN-113		FSN-136								
SAMPLE PERIOD	2	3	4	5	6	9	11	12	13	16	18
HOURS ON STREAM	39.5	63.5	87.08	112	133.9	201.3	242.6	263.2	288.4	350.8	396.4
PRODUCT GAS ANALYSES (VOL%)											
C1	1.820	1.450	1.610	1.610	1.610	2.140	1.790	1.690	1.690	1.690	1.220
C2	0.702	0.807	0.569	0.569	0.569	0.862	0.659	0.630	0.630	0.630	0.497
C3	0.409	0.480	0.327	0.327	0.327	0.611	0.421	0.423	0.423	0.423	0.381
C4	0.160	0.258	0.154	0.154	0.154	0.252	0.260	0.280	0.280	0.280	0.279
C5	0.059	0.103	0.073	0.073	0.073	0.081	0.118	0.130	0.130	0.130	0.122
C6	0.059	0.103	0.073	0.100	0.100	0.100	0.100	0.100	0.100	0.100	0.100
CO	0.183	0.278	0.215	0.215	0.215	0.263	0.230	0.168	0.168	0.168	0.065
CO2	0.132	0.301	0.355	0.355	0.355	0.328	0.592	0.411	0.411	0.411	0.132
H2S	.	.	.	.	.	.	.	.	.	.	.
FEED AND PRODUCT WATER (WT%)											
H2O IN FEED	.	.	.	.	.	.	.	.	.	.	.
FEED H2O LOSS G HR	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
H2O IN PRODUCT	4.900	9.600	8.800	9.700	5.500	7.400	7.900	8.900	8.900	5.600	0.200
SOLUBLE OR SLURRY CATALYSTS (WT%)											
SOL-SLURRY CAT	0.333	0.333	0.333	0.333	0.333	0.333	0.137	0.466	0.466	0.333	.
ASH IN CATALYST	100	100	100	100	100	100	100	100	100	100	.
S IN CATALYST	40	40	40	40	40	40	40	40	40	40	.
PRODUCT SLURRY SOLUBILITY, WT % INSOLUBLE											
THF	2.670	3.700	4.080	3.710	3.960	3.910	5.130	5.210	5.320	9.520	0.707
TOLUENE	3.600	4.740	5.380	4.930	5.640	5.910	9.870	9.810	9.300	13.92	2.130
HEXANE	7.390	8.160	10.6	9.200	9.780	10.54	15.39	15.64	16.39	19.52	11.96
ASH	2.200	1.940	2.130	2.020	2.140	2.010	1.780	1.800	1.760	2.040	.
PRODUCT DISTILLED FRACTIONS, WT%											
WT% NAPHTHA	13.84	14.49	14.9	14.46	14.17	13.05	11.2	11.39	12.49	4.370	0.000
WT% DISTILLATE	23.09	19.35	18.48	21.12	21.22	21.65	14.12	10.79	14.14	11.7	13.26
WT% VACUUM DISTILLATE	38.18	38.18	39.71	36.55	37.23	36.6	38.4	40.69	37.14	39.3	48.41
WT% RESID+UNCONV COAL	23.42	26.09	25.34	25.68	26.04	28.48	35.09	35.93	35.2	42.22	36.19

TABLE A-3

<u>BLACK THUNDER SUBBITUMINOUS COAL: C/T LIQUEFACTION WITH AMOCAT 1C CATALYST IN STAGE 1</u>

	------1.0% Fe2O3------						0.4%-	--1.4%--		1.0%-	SOLVENT
	FSN-113		---------------FSN-136---------------------								
SAMPLE PERIOD	2	3	4	5	6	9	11	12	13	16	18
DISTILLATE (360-650 F), WT%											
C	.	.	.	87.1	.	85.9	.	.	85.7	.	85.1
H	.	.	.	10.5	.	10.5	.	.	10.2	.	10.7
S	.	.	.	0.01	.	0.01	.	.	0.01	.	0.01
O	.	.	.	1.73	.	2.28	.	.	3.12	.	3.17
N	.	.	.	0.52	.	0.54	.	.	0.51	.	0.47
TOTAL	.	.	.	99.8	.	99.2	.	.	99.6	.	99.5
VACUUM DISTILLATE (650-RESID), WT%											
C	.	.	.	89.4	.	88.6	.	.	88.1	.	88.3
H	.	.	.	8.68	.	8.62	.	.	8.62	.	8.96
S	.	.	.	0.01	.	0.02	.	.	0.03	.	0.02
O	.	.	.	0.92	.	1.24	.	.	1.45	.	0.85
N	.	.	.	0.56	.	0.57	.	.	0.60	.	0.54
TOTAL	.	.	.	99.6	.	99.1	.	.	98.8	.	98.7
RESID + UNCONV COAL, WT%											
C	.	.	.	84.7	.	83.4	.	.	85.4	.	89.9
H	.	.	.	5.55	.	5.35	.	.	5.65	.	6.38
S	.	.	.	0.56	.	0.39	.	.	0.30	.	0.11
O (DIFFERENCE)	.	.	.	0.25	.	2.68	.	.	2.37	.	2.47
N	.	.	.	1.05	.	1.12	.	.	1.24	.	1.17
TOTAL	.	.	.	99.7	.	97.3	.	.	97.6	.	97.5
THF INSOLUBLES..........	.	.	.	.	.	.	.	.	.	.	.
RESID THF INSOLUBLES, WT%											
ASH	.	.	.	.	.	.	.	.	.	.	.
K	.	.	.	.	.	.	.	.	.	.	.
SI	.	.	.	.	.	.	.	.	.	.	.
AL	.	.	.	.	.	.	.	.	.	.	.
CA	.	.	.	.	.	.	.	.	.	.	.
FE	.	.	.	.	.	.	.	.	.	.	.
MG	.	.	.	.	.	.	.	.	.	.	.
MO	.	.	.	.	.	.	.	.	.	.	.
NA	.	.	.	.	.	.	.	.	.	.	.
NI	.	.	.	.	.	.	.	.	.	.	.
TI	.	.	.	.	.	.	.	.	.	.	.
V	.	.	.	.	.	.	.	.	.	.	.

TABLE A-4

BLACK THUNDER SUBBITUMINOUS COAL: C/T LIQUEFACTION WITH AMOCAT 1C CATALYST IN STAGE 1

	1.0% Fe2O3						0.4%	1.4%		1.0%	SOLVENT
	FSN-113		FSN-136								
SAMPLE PERIOD	2	3	4	5	6	9	11	12	13	16	18
HOURS ON STREAM	39.5	63.5	87.1	112	134	201	243	263	288	351	396
NET PRODUCT YIELDS (WT% MAF COAL OR EQUIVALENT)											
OILS	94.1	88.4	50.2	51.2	49.7	41.2	28.7	26.3	27.3	16.1	-17
ASPHALTENES	-25	-26	10.7	6.85	5.54	7.30	11.2	12.4	17.6	11	12.2
PREASPHALTENES	0.04	0.60	2.89	2.54	3.98	5.13	15.7	15.2	13.1	14	1.22
H2S	.	.	.	0.65	.	0.81	.	.	1.04	.	-.00
NH3	.	.	.	0.89	.	0.82	.	.	0.26	.	0.03
CO + CO2	2.68	4.98	5.20	6.36	7.51	7.21	7.76	6.30	5.78	5.54	0.78
H2O	.	.	.	17.7	.	12.5	.	.	12.5	.	0.00
C1 TO C3	16.7	16.2	13.8	16.8	19.9	28.8	16	17.8	16.4	15.7	5.26
C4 TO 360	38.6	27.4	29.5	26.2	40.2	30.3	20.6	19.3	22.9	4.19	3.06
360 TO 650	62.5	51.5	48.9	59.1	54.6	55.1	29.8	17.3	30.8	20.2	9.24
650 TO 935	3.48	9.69	17.3	4.46	-1.5	-5.6	6.88	15.9	4.40	8.75	-3.6
935 +	-35	-25	-31	-28	-33	-25	-.59	2.52	0.95	9.17	-12
C4 TO 650	101	78.9	78.4	85.4	94.8	85.4	50.4	36.6	53.7	24.4	12.3
C4 TO 935	105	88.6	95.7	89.8	93.3	79.8	57.3	52.5	58.1	33.1	8.69
UNCONVERTED COAL	1.50	6.55	6.94	5.88	6.02	6.30	12	12.2	13	26.9	0.93
ASH	-.37	-1.1	-.29	-.79	-.75	-1.2	-1	-2.3	-2.4	-1.1	.
HYDROGEN	.	.	.	-8.3	.	-9.4	.	.	-6.1	.	-1.1
PRODUCT MATERIAL BALANCES											
% OVERALL BALANCE	99.5	109	106	102	108	110	108	120	102	108	100
% ASH BALANCE	95.7	87.6	96.6	90.8	91.3	85.6	86.5	74.4	73.9	87.6	.
HETEROATOM REMOVAL (% OF HETEROATOMS IN FEED)											
SULFUR	.	.	.	49.9	.	62.3	.	.	67.2	.	-1.2
OXYGEN	.	.	.	.	.	.	.	.	.	.	.
NITROGEN	.	.	.	23.9	.	22	.	.	6.95	.	1.77

TABLE A-5

BLACK THUNDER COAL: C/T LIQUEFACTION WITH AMOCAT 1B CATALYST IN STAGE 1. PROCESS CONDITIONS

	Fe2O3/TPS-20				FeSO4/H2O		
SAMPLE PERIOD	2	3	5	6	7	8	9
HOURS ON STREAM	46.8	70.1	118	141	164	187	211
WT FRACTION COAL IN FEED .	0.33	0.33	0.33	0.33	0.33	0.33	0.33
1ST-STAGE TEMPERATURE, F .	780	780	780	780	780	780	780
SPACE TIME, MIN.	83	85.6	82.6	87.4	94.5	89.8	83.9
SEVERITY, STTUS.	15.7	16.2	15.6	16.6	17.9	17	15.9
COAL;CAT LHSV ..	0.84	0.82	0.84	0.80	0.73	0.77	0.83
SOLVENT;CAT LHSV	2.19	2.13	2.20	2.08	1.91	2.01	2.16
2ND-STAGE TEMPERATURE, F .	800	800	800	800	800	800	800
SPACE TIME, MIN.	83	85.6	82.6	87.4	94.5	89.8	83.9
SEVERITY, STTUS.	28	28.9	27.8	29.5	31.8	30.3	28.3
COAL;CAT LHSV ..	.	.	.	.	.	.	.
SOLVENT;CAT LHSV	.	.	.	.	.	.	.
COMBINED SEVERITY, STTUS .	43.7	45.1	43.5	46	49.7	47.3	44.1
SYSTEM PRESSURE, PSIG	2500	2500	2500	2500	2500	2500	2500
1000 SCFH H2 PER BBL FEED.	7.59	7.39	7.45	8.30	9.15	8.35	7.59

The first stage stirrer was not working for at least the last part of the run.

TABLE A-6

BLACK THUNDER COAL: C/T LIQUEFACTION WITH AMOCAT 1B CATALYST IN STAGE 1, PRODUCT ANALYSES

	Fe2O3/TPS-20				FeSO4/H2O		
SAMPLE PERIOD	2	3	5	6	7	8	9
HOURS ON STREAM	46.81	70.06	118.2	140.5	163.9	187.3	210.8
PRODUCT GAS ANALYSES (VOL%)							
C1	1.240	1.330	1.330	1.330	1.270	1.480	1.400
C2	0.523	0.543	0.543	0.505	0.528	0.593	0.576
C3	0.377	0.375	0.375	0.293	0.379	0.368	0.378
C4	0.288	0.242	0.242	0.134	0.264	0.211	0.233
C5	0.163	0.131	0.131	0.071	0.127	0.096	0.100
C6	0.163	0.131	0.131	0.071	0.127	0.096	0.100
CO	0.288	0.292	0.292	0.278	0.272	0.319	0.250
CO2	0.351	0.359	0.359	0.393	0.422	0.550	0.420
H2S	.	.	.	.	.	.	.
FEED AND PRODUCT WATER (WT%)							
H2O IN FEED	.	.	.	.	.	.	.
FEED H2O LOSS G HR	0.000	0.000	0.000	0.000	0.000	0.000	0.000
H2O IN PRODUCT	6.740	8.340	7.000	8.400	.	11.8	10.5
SOLUBLE OR SLURRY CATALYSTS (WT%)							
SOL-SLURRY CAT	0.333	0.333	0.333	0.333	1.000	1.000	1.000
ASH IN CATALYST	100	100	100	100	100	100	100
S IN CATALYST	40	40	40	40	40	40	40
PRODUCT SLURRY SOLUBILITY, WT % INSOLUBLE							
THF	5.140	3.580	5.800	4.960	5.420	5.230	5.070
TOLUENE	6.800	6.420	10.05	10.47	6.600	6.330	8.710
HEXANE	10.28	10.53	14.56	14.86	12.66	13.22	12.57
ASH	2.240	1.550	2.300	2.320	.	2.970	2.860
PRODUCT DISTILLED FRACTIONS, WT%							
WT% NAPHTHA	9.510	10.14	6.920	10.81	.	13.42	12.4
WT% DISTILLATE	15.31	17.3	16.29	13.63	.	16.17	14.68
WT% VACUUM DISTILLATE	40.73	38.14	37.6	37.6	.	35.51	34
WT% RESID+UNCONV COAL	33.24	32.65	37.5	37.5	.	33.85	38.86

TABLE A-7

BLACK THUNDER COAL: C/T LIQUEFACTION WITH AMOCAT 1B CATALYST IN STAGE 1,
ELEMENTAL ANALYSES

	Fe2O3/TPS-20				FeSO4/H2O		
SAMPLE PERIOD	2	3	5	6	7	8	9
DISTILLATE (360-650 F), WT%							
C	.	.	.	85	.	85.9	.
H	.	.	.	10.2	.	10.3	.
S	.	.	.	0.01	.	0.01	.
O	.	.	.	3.97	.	2.77	.
N	.	.	.	0.45	.	0.46	.
TOTAL	.	.	.	99.6	.	99.4	.
VACUUM DISTILLATE (650-RESID), WT%							
C	.	.	.	89.8	.	89.4	.
H	.	.	.	8.84	.	8.78	.
S	.	.	.	0.04	.	0.06	.
O	.	.	.	1.20	.	1.22	.
N	.	.	.	0.49	.	0.46	.
TOTAL	.	.	.	100	.	99.9	.
RESID + UNCONV COAL, WT%							
C	.	.	.	83.9	.	83.5	.
H	.	.	.	5.66	.	5.56	.
S	.	.	.	0.37	.	1.05	.
O (DIFFERENCE)	.	.	.	2.64	.	0.00	.
N	.	.	.	1.20	.	1.20	.
TOTAL	.	.	.	97.4	.	100	.
THF INSOLUBLES	.	.	.	.	.	.	.
RESID THF INSOLUBLES, WT%							
ASH	.	.	.	.	.	.	.
K	.	.	.	1640	.	1350	.
SI	.	.	.	69E3	.	57E3	.
AL	.	.	.	24E3	.	41E3	.
CA	.	.	.	52E3	.	0.00	.
FE	.	.	.	12E4	.	18E4	.
MG	.	.	.	21E3	.	16E3	.
MO	.	.	.	103	.	106	.
NA	.	.	.	6100	.	8500	.
NI	.	.	.	228	.	168	.
TI	.	.	.	5000	.	3670	.
V	.	.	.	301	.	203	.

TABLE A-8

BLACK THUNDER COAL: C/T LIQUEFACTION WITH AMOCAT 1B CATALYST IN STAGE 1, NET YIELDS

	Fe2O3/TPS-20				FeSO4/H2O		
SAMPLE PERIOD	2	3	5	6	7	8	9
HOURS ON STREAM	46.8	70.1	118	141	164	187	211
NET PRODUCT YIELDS (WT% MAF COAL OR EQUIVALENT)							
OILS	52.5	48.4	36	34.1	50.7	36.5	43
ASPHALTENES	3.38	5.98	7.34	7.36	14.6	17.6	5.62
PREASPHALTENES	4.02	8.58	13.8	19.1	2.54	2.19	12.2
H2S	.	.	.	0.66	.	0.90	.
NH3	.	.	.	0.43	.	0.61	.
CO + CO2	6.16	6.13	6.17	7.19	8.35	9.63	6.72
H2O	.	.	.	13.5	.	15.6	.
C1 TO C3	13.7	13.8	13.9	14.1	16.8	16.8	14.9
C4 TO 360	21.4	15.7	8.95	14.7	.	14.4	15.2
360 TO 650	33.1	41.5	37.2	28.7	.	39.9	33.9
650 TO 935	16.8	11.7	8.10	7.55	.	5.05	-5.2
935 +	-10	-4.9	4.06	10.7	.	-1.9	18.1
C4 TO 650	54.5	57.2	46.2	43.4	.	54.3	49.1
C4 TO 935	71.3	68.9	54.3	51	.	59.3	43.9
UNCONVERTED COAL	10.2	7.06	12.5	9.56	10.3	8.30	8.07
ASH	-.22	-2.7	0.07	0.39	.	0.37	-.10
HYDROGEN	.	.	.	-5.4	.	-6.5	.
PRODUCT MATERIAL BALANCES							
% OVERALL BALANCE	138	111	110	105	107	104	98
% ASH BALANCE	97.4	68.5	101	105	.	103	99.1
HETEROATOM REMOVAL (% OF HETEROATOMS IN FEED)							
SULFUR	.	.	.	50.7	.	36	.
OXYGEN	.	.	.	.	.	.	.
NITROGEN	.	.	.	11.6	.	16.2	.

TABLE A-9

BLACK THUNDER COAL: C/T LIQUEFACTION WITH AMOCAT 1B CATALYST IN STAGE 1, PROCESS CONDITIONS

	FeSO4/H2O 1% Fe/COAL						FeSO4/MeOH 0.4% Fe		FeSO4/H2O 1% Fe		SOLVENT CORRECTION		
SAMPLE PERIOD	2	4	5	6	8	9	11	12	14	15	16	18	19
HOURS ON STREAM	40	87	111	134	181	205	250	273	294	318	341	387	410
WT FRACTION COAL IN FEED .	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.33	0.00	0.00
1ST-STAGE TEMPERATURE, F .	780	780	780	780	780	780	780	780	780	780	780	780	780
SPACE TIME, MIN.	76.1	95.6	96.1	113	90.8	82.6	74.4	77.5	79.4	79	79.4	74.1	84.7
SEVERITY, STTUS.	14.4	18.1	18.2	21.3	17.2	15.6	14.1	14.7	15	15	15	14	16
COAL;CAT LHSV ..	0.91	0.72	0.72	0.61	0.76	0.83	0.94	0.90	0.87	0.87	0.87	0.00	0.00
SOLVENT;CAT LHSV	2.36	1.88	1.87	1.60	1.98	2.17	2.44	2.34	2.26	2.27	2.26	3.70	3.23
2ND-STAGE TEMPERATURE, F .	800	800	800	800	780	780	780	780	780	780	780	780	780
SPACE TIME, MIN.	76.1	95.6	96.1	113	90.8	82.6	74.4	77.5	79.4	79	79.4	74.1	84.7
SEVERITY, STTUS.	25.6	32.2	32.4	37.9	17.2	15.6	14.1	14.7	15	15	15	14	16
COAL;CAT LHSV ..	.	.	.	.	.	.	.	.	.	.	.	.	.
SOLVENT;CAT LHSV	.	.	.	.	.	.	.	.	.	.	.	.	.
COMBINED SEVERITY, STTUS .	40.1	50.3	50.6	59.3	34.4	31.3	28.2	29.4	30.1	29.9	30.1	28	32.1
SYSTEM PRESSURE, PSIG	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500
1000 SCFH H2 PER BBL FEED.	6.85	8.78	8.30	10.9	7.67	6.97	7.02	7.32	7.89	7.80	7.65	7.43	7.71

TABLE A-10

BLACK THUNDER COAL: C/T LIQUEFACTION WITH AMOCAT 1B CATALYST IN STAGE 1, PRODUCT ANALYSES

	FeSO4/H2O 1% Fe/COAL						FeSO4/MeOH 0.4% Fe		FeSO4/H2O 1% Fe		SOLVENT CORRECTION		
SAMPLE PERIOD	2	4	5	6	8	9	11	12	14	15	16	18	19
HOURS ON STREAM	40.05	86.97	110.5	133.8	181	204.5	250	22.6	294.4	318	341.4	387.2	410.2
PRODUCT GAS ANALYSES (VOL%)													
C1	1.250	1.470	1.470	1.090	1.170	1.060	1.060	1060	1.020	1.040	0.821	0.777	0.777
C2	0.489	0.604	0.604	0.450	0.480	0.445	0.445	0445	0.384	0.418	0.335	0.331	0.331
C3	0.313	0.402	0.402	0.318	0.325	0.327	0.327	0327	0.242	0.277	0.229	0.238	0.238
C4	0.172	0.167	0.167	0.173	0.180	0.198	0.198	0198	0.113	0.130	0.108	0.123	0.123
C5	0.076	0.052	0.052	0.054	0.075	0.083	0.083	0083	0.053	0.056	0.053	0.045	0.045
C6	0.076	0.052	0.052	0.054	0.075	0.083	0.083	0083	0.053	0.056	0.053	0.045	0.045
CO	0.163	0.174	0.174	0.093	0.135	0.106	0.106	0106	0.113	0.130	0.123	0.070	0.070
CO2	0.446	0.349	0.349	0.228	0.395	0.291	0.291	0291	0.428	0.470	0.365	0.182	0.182
H2S	.	.	.	.	.	.	.		.	.	.	.	.
FEED AND PRODUCT WATER (WT%)													
H2O IN FEED	.	.	.	.	.	.	.		.	.	.	.	.
FEED H2O LOSS G HR	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0000	0.000	0.000	0.000	0.000	0.000
H2O IN PRODUCT	9.320	11.43	11.25	11.76	10.54	9.210	7.030	6930	11.04	7.390	.	0.000	0.000
SOLUBLE OR SLURRY CATALYSTS (WT%)													
SOL-SLURRY CAT	1.670	1.670	1.670	1.670	1.670	1.670	0.670	0670	1.670	1.670	1.670	.	.
ASH IN CATALYST	29	29	29	29	29	29	29	29	29	29	29	.	.
S IN CATALYST	12	12	12	12	12	12	12	12	12	12	12	.	.
PRODUCT SLURRY SOLUBILITY, WT % INSOLUBLE													
THF	3.930	5.430	5.280	5.060	4.740	4.810	5.530	5840	5.160	5.790	5.060	1.070	0.940
TOLUENE	5.060	6.330	7.280	7.220	9.150	9.840	10.5	1.71	11.4	12.01	10.85	2.850	2.670
HEXANE	9.660	11.36	12.14	12.1	15.13	14.81	18.18	1.48	17.47	21.03	18.95	7.140	10.19
ASH	.	2.730	2.220	2.462	2.180	2.350	1.960	2010	2.280	2.390	2.400	.	.
PRODUCT DISTILLED FRACTIONS, WT%													
WT% NAPHTHA	13.14	13.15	11.88	13.22	11.31	8.950	5.380	5280	10.47	6.390	.	0.000	0.000
WT% DISTILLATE	14.57	17.9	17.32	18.24	13.65	13.37	13.49	1.64	12.5	13.4	.	12.03	16.3
WT% VACUUM DISTILLATE	39.05	34.67	35.83	34.24	37.81	37.23	36.78	3.86	33.04	37.36	.	47.59	47.73
WT% RESID+UNCONV COAL	30.5	31.72	32.74	32.54	35.3	38.76	42.13	3.67	42.54	41.58	.	39.08	34.59

TABLE A-11

BLACK THUNDER COAL: C/T LIQUEFACTION WITH AMOCAT 1B CATALYST IN STAGE 1.
ELEMENTAL ANALYSES

	FeSO4/H2O 1% Fe/COAL						FeSO4/MeOH 0.4% Fe		FeSO4/H2O 1% Fe		SOLVENT CORRECTION		
SAMPLE PERIOD	2	4	5	6	8	9	11	12	14	15	16	18	19
DISTILLATE (360-650 F), WT%													
C	.	.	85.2	.	.	85.4	.	84.8	.	85.9	.	.	85.8
H	.	.	10.3	.	.	10.3	.	10.2	.	10.4	.	.	10.4
S	.	.	0.01	.	.	0.01	.	0.01	.	0.01	.	.	0.01
O	.	.	4.00	.	.	4.46	.	4.03	.	4.50	.	.	3.16
N	.	.	0.47	.	.	0.46	.	0.45	.	0.45	.	.	0.44
TOTAL	.	.	99.9	.	.	101	.	99.5	.	101	.	.	99.8
VACUUM DISTILLATE (650-RESID), WT%													
C	.	.	90.3	.	.	89.7	.	89.9	.	90.1	.	.	90.3
H	.	.	8.86	.	.	9.05	.	8.95	.	8.94	.	.	9.17
S	.	.	0.03	.	.	0.04	.	0.04	.	0.04	.	.	0.03
O	.	.	1.11	.	.	1.36	.	1.30	.	1.35	.	.	0.94
N	.	.	0.54	.	.	0.55	.	0.50	.	0.55	.	.	0.52
TOTAL	.	.	101	.	.	101	.	101	.	101	.	.	101
RESID + UNCONV COAL, WT%													
C	.	.	80.7	.	.	84.4	.	85.8	.	85.2	.	.	90
H	.	.	5.74	.	.	5.90	.	6.03	.	5.84	.	.	6.50
S	.	.	0.88	.	.	0.80	.	0.27	.	0.72	.	.	0.20
O (DIFFERENCE)	.	.	4.55	.	.	1.52	.	1.61	.	1.14	.	.	2.03
N	.	.	1.31	.	.	1.35	.	1.24	.	1.32	.	.	1.29
TOTAL	.	.	95.5	.	.	98.5	.	98.4	.	98.9	.	.	98
THF INSOLUBLES	.	.	.	.	.	.	.	.	.	.	.	.	.
RESID THF INSOLUBLES, WT%													
ASH	.	.	.	.	.	.	.	.	.	.	.	.	.
K	.	8500	8500	.	.	4700	.	3270	.	2320	.	.	.
SI	.	59E3	59E3	.	.	79E3	.	11E4	.	81E3	.	.	.
AL	.	30E3	30E3	.	.	39E3	.	35E3	.	45E3	.	.	.
CA	.	71E3	71E3	.	.	99E3	.	92E3	.	11E4	.	.	.
FE	.	86E3	86E3	.	.	15E4	.	10E4	.	14E4	.	.	.
MG	.	11E3	11E3	.	.	12E3	.	16E3	.	8100	.	.	.
MO	.	70	70	.	.	58	.	61	.	108	.	.	.
NA	.	4100	4100	.	.	5900	.	7400	.	5800	.	.	.
NI	.	129	129	.	.	170	.	227	.	204	.	.	.
TI	.	3090	3090	.	.	4400	.	5500	.	4700	.	.	.
V	.	169	169	.	.	163	.	206	.	185	.	.	.

TABLE A-12

BLACK THUNDER COAL: C/T LIQUEFACTION WITH AMOCAT 1B CATALYST IN STAGE 1, NET YIELDS

	FeSO4/H2O 1% Fe/COAL						FeSO4/MeOH 0.4% Fe		FeSO4/H2O 1% Fe		SOLVENT CORRECTION		
SAMPLE PERIOD	2	4	5	6	8	9	11	12	14	15	16	18	19
HOURS ON STREAM	40	87	111	134	181	205	250	273	29	318	341	387	410
NET PRODUCT YIELDS (WT% MAF COAL OR EQUIVALENT)													
OILS	54.8	44.2	43.8	41.8	31.9	36.6	24.6	26.7	24.	11.5	20.4	-8.9	-15
ASPHALTENES	8.42	10.4	9.84	9.79	14	9.85	19.1	15.7	14.	24.5	22.9	1.50	7.74
PREASPHALTENES	2.26	1.41	5.83	6.41	15.2	17.5	16.4	16	22.	21.3	21	1.92	1.82
H2S	.	.	0.32	.	.	0.24	.	0.58	.	0.33	.	.	-.07
NH3	.	.	0.26	.	.	-.06	.	0.30	.	-.05	.	.	0.00
CO + CO2	5.81	6.22	5.89	4.84	5.69	3.85	3.85	4.01	6.0	6.64	5.23	1.22	1.27
H2O	.	.	12.6	.	.	18.6	.	17.7	.	17.8	.	.	0.11
C1 TO C3	11.6	18.2	17.3	17.2	12.8	10.9	10.9	11.4	10.	11.3	8.91	4.02	4.17
C4 TO 360	20.2	12.3	7.73	12.9	9.03	6.01	6.00	6.25	4.1	4.48	.	1.74	1.81
360 TO 650	33.1	47.1	45.3	48.5	30	28.4	26.6	34.4	2	26.8	.	6.34	14.5
650 TO 935	24.2	8.36	12.7	3.58	17.6	12.8	6.64	7.63	-1.	6.79	.	-4.5	-4.4
935 +	-11	-11	-5.2	-5.8	5.76	17.8	22.1	11.2	34.	20.5	.	-8.2	-17
C4 TO 650	53.3	59.4	53	61.4	39	34.4	32.6	40.7	30.	31.3	.	8.08	16.4
C4 TO 935	77.5	67.8	65.7	64.9	56.6	47.2	39.2	48.3	28.	38	.	3.56	11.9
UNCONVERTED COAL	6.32	10.1	11.6	9.68	9.48	8.99	12.7	13.6	10.	12.2	10	1.64	1.38
ASH	.	1.63	-.33	0.57	-.59	-.01	-.64	-.48	-.1	-.16	0.43	.	.
HYDROGEN	.	.	-6.6	.	.	-5.6	.	-5.6	.	-4.8	.	.	-1
PRODUCT MATERIAL BALANCES													
% OVERALL BALANCE	104	101	98.3	117	103	89.5	102	101	10	93.6	92.6	107	92.9
% ASH BALANCE	.	118	96.4	106	93.6	99.9	91.9	93.9	98.	98.3	105	.	.
HETEROATOM REMOVAL (% OF HETEROATOMS IN FEED)													
SULFUR	.	.	19.9	.	.	15.2	.	54.6	.	20.7	.	.	-70
OXYGEN	.	.	.	.	.	.	.	.	.	.	.	.	.
NITROGEN	.	.	6.99	.	.	-1.7	.	8.01	.	-1.3	.	.	0.21