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ADVANCED RESEARCH FOR THE CHARACTERIZATION OF
HYDROGEN DONOR SOLVENTS IN TWO-STAGE LIQUEFACTION

Quarterly Report for the Period April 1—June 30, 1984

By
John A. Hamshar
John D. Potts

Work Performed Under Contract No. AC22-83PC60047

Cities Service Research and Development Company
Tulsa, Oklahoma

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Prepared for the United States Department of Energy
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INTRODUCTION

The purpose of this research program is to develop an improved method for producing a highly effective solvent for the Integrated Two-Stage Liquefaction (ITSL) process and to study the characteristics of the solvent at a variety of process conditions. The ITSL process for conversion of coal to distillate liquids combines Short Contact Time (SCT) hydroliquefaction and expanded bed hydrogenation and hydrocracking of the coal extract, either before or after deashing.

The proposed improvement in the ITSL process is based on a modification of the expanded bed hydrotreating stage. The function of this process step is to hydrotreat and hydrocrack the heavy SCT extract to distillate product, to remove sulfur and nitrogen, and to rehydrogenate the recycle hydrogen donor solvent. These operations are usually performed in a series of two or three catalytic expanded bed reactors using a common catalyst and similar process conditions.

One of the primary reasons for the success of the ITSL process has been the separation of the dissolution and hydrotreating stages so that the process operating conditions for each stage may be optimized. It should be possible to further improve the ITSL process by also separating the hydrogenation and hydrocracking operations in the second stage of the process.

The investigative program to determine the efficiency of separating the hydrogenation and hydrocracking steps will be comprised of the following main areas:

1. Study of solvent hydrogenation and hydrocracking in the absence of coal or extract;
2. Evaluation and optimization of the reaction conditions, catalysts and solvents for the coal dissolution, extract hydrogenation and hydrocracking steps of ITSL;

3. Evaluation of aging characteristics of superior hydrogenation and hydrocracking catalysts.

An extensive amount of research will be required to determine the effects of temperature, pressure, space velocity, catalyst, etc. in the above areas. Performance of these studies in an ITSL pilot plant would be expensive and time consuming. Cities Service Research & Development Company will perform these studies in small scale batch and continuous units specifically designed for this purpose.

SUMMARY

Solvent-only reactions at SCT conditions in hydrogen (Subtask 2.2) were explored for each of the four blended solvents, ranging from 6.1 to 8.6 weight percent hydrogen content (nominally 6.1, 7.7, 7.4, 8.6). Generally, no loss of solvent to gas was measured, but small amounts of retrograde reactions (oil to preasphaltenes, asphaltenes, insolubles) were observed at both low and high severity. Insolubles were produced in solvent A (6.1% H) and consumed in solvent D (8.6% H). Procedures were developed to correct SCT results for the solvent-only effects observed. The trends shown in reports to date, without this correction, are valid because the corrections are small.

Ten hydrotreating and hydrocracking catalysts have been selected and obtained from a variety of catalyst manufacturers. A matrix of eleven runs was made using the base catalyst and solvent B (7.7% H) (Subtask 2.3), to determine ranges of operating conditions appropriate for further catalyst testing.

A series of 72 run conditions were completed at SCT conditions for coal extraction (Subtask 3.1) in the microautoclave, studying the effects of temperature, residence time, pressure, solvent makeup, and atmosphere. At hydrogen-deficient conditions, coal conversions were observed to decline at the highest reaction severities (ie. 850-900°F), apparently due to condensation (coking) reactions. Preasphaltene and asphaltene yields are maximized at intermediate severities, and oil yields increase with severity over the range of conditions tested. Coal conversions in a nitrogen atmosphere were generally slightly less than in hydrogen, for all solvents, and at both mild and severe conditions. Coal conversions maximize at a solvent hydrogen content of approximately 7.5 percent. At this level of hydrogen content, solvent B (7.7% H), made by mild hydrogenation of KC-oil, out-performs solvent C (7.4% H), made by blending hydrogen-rich and hydrogen-deficient components. The performance of solvent C was, however, significantly better than that of either component by itself.

WORK ACCOMPLISHED

The effort this quarter concentrated on Subtask 2.2 - Solvent Reactions at SCT Conditions, Subtask 2.3 - Solvent Hydrocracking with Catalyst, and Subtask 3.1 - Short Contact Time (SCT) Dissolver with Hydrogenated Solvents. Subtask 2.2 experiments were completed on all four solvents using a hydrogen atmosphere. Subtask 2.3 was started with an 11 run matrix on the base catalyst (Shell 324 Ni/Mo 1/32-inch extrudate). Subtask 3.1 was completed with both hydrogen and nitrogen atmospheres for SCT reactions.

Analyses and blend ratios of the four solvents prepared in Subtask 2.1 are given in Table 1 for reference. These solvents, covering the hydrogen-content range of 6.1 to 8.6 weight percent, are the basis for the solvent studies performed in the remainder of this program. The solvents were re-analysed periodically along with reaction products from a series of runs using the solvent. Therefore the solvent analyses used in run calculations may differ from those reported in Table 1, within the ± 1 percent range of variation on OAPI analyses. The solvent analysis used in any particular series of runs is reported along with the yields and conversions. The average Illinois No. 6 coal analysis is reported in Table 2.

Calculation procedures for yields, conversions, and other run parameters are summarized in Appendix A.

Subtask 2.2 - Solvent Reactions at SCT Conditions

Operating conditions, yields and conversions for the eleven runs using solvent C are reported in Table 3 together with similar data for runs with solvents A, B, and D. Abbreviated 2-run matrices for solvents A, B and D were completed using a hydrogen atmosphere at 2500 psig. Analytical data for these runs are presented in Table 4. Solvent-only reactions in hydrogen proceeded to a minor extent only. Within the experimental accuracy, no gas yield was detected, and no loss of solvent

weight was observed. However, small changes in the OAPI (oil, asphaltenes, preasphaltenes, insolubles) composition of the solvents were observed. Figures 1 through 4 show the OAPI conversions observed for each solvent, at both mild (700°F, 2500 psig H₂, 10 min.) and at severe (850°F, 2500 psig H₂, 10 min.) operating conditions. Conversion is defined as the disappearance of the component, as a percentage of the total charge solvent. The 2-3 percent negative conversion of preasphaltenes in Figure 1 indicate that preasphaltenes are being produced for three of the four solvents at both mild and severe conditions. Asphaltene conversions are nearly zero, except for solvent C (Figure 2). For this solvent, asphaltenes are produced, rather than preasphaltenes as with the other three solvents. Figure 3 shows that part of the oil fraction is consumed in order to produce asphaltenes and preasphaltenes, apparently by polymerization. Figure 4 shows a tendency to produce insolubles (coke) from the low-hydrogen solvent A at severe conditions, and conversely to convert insolubles in the high-hydrogen solvent D.

The solvent-only reaction data were used to generate corrections to the net coal conversion observed for these solvents in SCT reactions. These corrected conversions will be reported subsequently, along with nitrogen-atmosphere data for Subtasks 2.2 and 3.1.

Subtask 2.3 - Solvent Hydrocracking with Catalysts

A series of ten hydrotreating/hydrocracking catalysts have been selected for study and samples obtained, as shown in Table 5. These catalysts are all either currently commercial, or are planned to be commercial within the next few years. Nickel-molybdenum, cobalt-molybdenum, and nickel-tungsten extruded and beaded catalysts are included together with a bimodal-support catalyst, incorporating coke-resistant macropores.

A matrix of eleven runs was made on solvent B using the base catalyst, Shell 324 Ni/Mo (modified), in order to determine the range of operating conditions to be studied for other catalysts and solvents. The operating conditions for these runs are presented in Table 6.

Subtask 3.1 - SCT Dissolver with Hydrogenated Solvents

SCT runs were made in a nitrogen atmosphere, using all four solvents: eleven-run matrices were run on solvents B and C, and seven-run matrices on solvents A and D. The operating conditions for these runs are reported in Tables 7 through 10. Each run was performed in duplicate, in order to generate sufficient quantities of SCT extract for subsequent hydrocracking tests.

Analytical data, including ultimates and OAPI's, for the extract products are given in Table 11 for solvent A in hydrogen, Table 12 for solvent B in hydrogen, Table 13 for solvent C in both hydrogen and nitrogen, and Table 14 for solvent D in both hydrogen and nitrogen. Calculated coal conversions for all of these runs are reported in Tables 15 through 18. The conversions are preliminary, pending minor adjustments due to additional analytical data. In addition, the coal conversions will be corrected for solvent-only reactions. These data and gas yields, hydrogen consumptions, and the fate of heteroatoms will be reported subsequently, as will data on solvents A and B in a nitrogen atmosphere.

Coal conversion trends for solvent A and hydrogen are shown in Figure 5. The conversion increases with severity (temperature and/or time) up to a maximum of about 80 percent, then declines at still higher severity. This is apparently due to the deposition of coke from the reaction mixture, which is relatively hydrogen-deficient (solvent A is not hydrogenated). No hydrogen-pressure effect is seen. The conversion trends for OAPI cuts are similar to those of solvent D, discussed below, and are included in the tables.

Coal conversions for SCT reactions using solvent D and hydrogen are given in Figures 6 through 9. Total coal conversion, Figure 6, increases with severity, leveling off as 100 percent conversion is approached. Residence time has a greater effect at low temperatures. A small pressure effect is apparent, with the increase from 500 to 2500 psig causing an increase of about 2 percent on conversion at 850°F. The abbreviated experimental matrix does not provide information on the pressure effect at

low temperature. Coal conversion to preasphaltenes (Figure 7) follows the classical pattern of an intermediate product in a kinetically-controlled reaction: preasphaltenes are produced as coal converts, then react toward asphaltenes and oil as severity increases. Thus, preasphaltenes reach a peak level at an intermediate severity, declining at higher severity. Preasphaltenes reach the highest peak at the shortest residence time, indicating that the rate of coal conversion increases more rapidly with temperature than the rate of preasphaltene decomposition. Because of the complexity of these trends, no pressure effect could be resolved. Conversion to asphaltenes (Figure 8) also increases with severity to a maximum, then declines, as asphaltenes break down in favor of oil. Residence time has a positive effect at low severities, but has little effect at high severity. Pressure exerts a positive effect of about 4 percent on the conversion. Conversion to oil (Figure 9) increases with increasing severity (temperature, residence time, and pressure). Negative conversions to oil are observed at low severities, as the solvent experiences retrograde conversion in donating hydrogen to the coal.

Coal conversions in a nitrogen atmosphere are shown for solvent D in Figures 10 through 13. Total conversion quantities and trends are similar to those observed in a hydrogen atmosphere, except at high severities, where conversions decline in nitrogen, indicating condensation (coke forming) reactions. No pressure effect was observed in nitrogen runs for any solvent nor for any OAPI component. The conversion to preasphaltenes (Figure 11) follows trends similar to those observed in hydrogen (Figure 7), except that conversion to preasphaltenes drops off at high severities. The nitrogen data were not sufficient to resolve the detail shown for the hydrogen data, so a single lumped trend line is shown. Conversion to asphaltenes (Figure 12) agrees closely with trends shown for the hydrogen runs. Coal conversion to oil (Figure 13) follows a similar trend to the hydrogen runs, but oil yields are about 10 percentage points higher with nitrogen than with hydrogen at all severities.

Coal conversions for solvent C in nitrogen are shown in Figures 14 through 17. Trend shapes are similar to those observed for solvent D, but coal conversion peaks about 10 percentage points lower (80%) due to

the reduced hydrogen-donating capacity of solvent C relative to solvent D. The conversion to preasphaltenes (Figure 15) does not fall off as sharply at high severities as observed with solvent D. Both asphaltenes and oil (Figures 16 and 17) are lower for solvent C, and the oil yield tails off at high severities with solvent C, indicative of a hydrogen donor deficiency in the nitrogen atmosphere.

Total coal conversion at SCT extraction conditions in hydrogen and nitrogen is compared in Figure 18 for solvents C and D. In general, a small suppression of conversion by nitrogen is observed, both at high and low severities.

The coal conversion in hydrogen is shown as a function of the hydrogen content of the solvent in Figure 19, for both high severity and low severity operation. At either severity, conversion peaks at a hydrogen content of about 7.5 percent in the solvent. Solvents B and C have approximately the same hydrogen content, but solvent B performs better. Solvent C comprises essentially a blend of solvents A and D, which are lean and overly saturated with hydrogen, respectively, and therefore not good hydrogen donors. Solvent B is essentially a mildly hydrogenated KC-oil, and is neither too lean, nor overly saturated. Over-saturation produces poor donor solvent capabilities because fully saturated molecules do not generally release hydrogen as readily as partially-saturated molecules. These effects will be explored more thoroughly with proton NMR data, when available.

WORK FORECAST

The projected work for the third quarter 1984 will include the completion of Subtasks 2.2 and 2.3. A series of ten hydrotreating and hydrocracking catalysts have been selected and samples obtained, allowing Subtasks 2.3, 3.2 and 3.3 to proceed. Subtask 3.2, Coal Extract and Solvent Hydrogenation, will be started with selection and blending of feed samples. The microautoclave work on this subtask will begin. Samples will be submitted for proton NMR analysis for the entire program to date.

PROGRESS SUMMARY FOR JUNE

Microautoclave Runs: 18 single runs
18 duplicate runs

Analyses Requested:

OAPI: 54

CHNS: 36

Gas Analysis: 35

Crude Distillation by GC: 12

Analyses Received: 0

PROGRESS SUMMARY FOR OCT. 1983 TO JUNE 1984

Microautoclave Runs: 60 single runs
83 duplicate runs

Continuous Catalytic Reactor: 1 five-day run

Analyses Received:

OAPI: 173

CHNS: 109

CHN: 7

Gas Analysis: 84

Proximate: 2

TABLE 1

TYPICAL ANALYTICAL DATA FOR SOLVENTS A, B, C AND D

	<u>Solvent A</u>	<u>Solvent B</u>	<u>Solvent C</u>	<u>Solvent D</u>
Elemental Analysis				
% Carbon	90.1	90.0	86.7	88.0
% Hydrogen	6.1	7.7	7.4	8.6
% Nitrogen	0.89	0.62	0.48	0.37
% Sulfur	0.61	0.25	0.53	0.15
H/C Ratio	0.81	1.02	1.02	1.17
OAPI Analysis				
% Oil	92.20	94.28	92.74	94.60
% Asphaltenes	2.84	2.37	2.00	2.19
% Preasphaltenes	3.14	2.64	3.03	2.74
% Insolubles ¹	1.82	0.72	2.23	0.47
Distillation Data				
% IBP-500°F	2.8	5.6	7.8	11.4
% 500-650°F	25.9	14.3	39.7	57.4
% 650-850°F	52.7	55.3	38.6	22.3
%850°F+	18.6	24.8	13.9	8.9

Solvent A is an 85/15 weight percent blend of raw KC-Oil and Illinois #6 SCT extract.

Sovlent B is an 85/15 weight percent blend of Lummus hydrogenated KC-Oil and Illinois #6 SCT extract.

Solvent C is a 42.5/40/17.5 weight percent blend of raw KC-Oil, Xytel hydrogenated KC-Oil and Illinois #6 SCT extract.

Solvent D is an 80/20 weight percent blend of Xytel hydrogenated KC-Oil and Illinois #6 SCT extract.

¹ Ash + Organic Insolubles

TABLE 2

ANALYSIS OF ILLINOIS NO. 6 COAL

Proximate Analysis, Wt%

Moisture	3.0
Volatile Matter, dry	39.9
Fixed Carbon, dry	49.2
Ash, dry	10.9

Elemental Analysis, Wt.%

C	64.1
H	5.3
N	1.2
S	3.6
O (Diff)	14.9

TABLE 3

CALCULATED RESULTS FROM OAPI ANALYSES OF LIQUID PRODUCTS

SUBTASK 2.2 - SCT DISSOLVER WITH SOLVENTS ALONE

REACTION ATMOSPHERE IS HYDROGEN

RUN #	RUN DATE	SOL-VENT	TEMP F	PRESS PSIG	TIME MIN	LIQUID YIELD	HC-GAS YIELD	-----CONVERSIONS *-----			
								PAS	AS	OIL	INSOL
MRS2.2-1A	2/7/84	C	700	500	10	98.6	0.0	2.0	0.8	-3.0	-0.4
MRS2.2-2A	2/7/84	C	700	2500	10	101.0	0.0	2.2	-1.2	1.3	-0.5
MRS2.2-3A	2/6/84	C	850	500	10	99.5	0.0	1.0	-0.2	-0.1	0.6
MRS2.2-4A	2/6/84	C	850	2500	10	97.9	0.0	0.0	-1.7	0.8	0.0
MRS2.2-5A	2/6/84	C	900	2000	8	98.3	0.3	0.5	-3.1	2.1	0.5
MRS2.2-6A	2/6/84	C	775	1500	6	97.8	0.0	1.8	0.8	-3.6	0.7
MRS2.2-7A	2/7/84	C	650	1000	4	97.1	0.0	2.0	-0.2	-3.4	0.3
MRS2.2-8A	2/7/84	C	700	500	2	97.2	0.0	1.8	0.4	-3.8	0.1
MRS2.2-9A	2/7/84	C	700	2500	2	98.3	0.0	0.8	1.8	-3.0	-0.5
MRS2.2-10A	2/6/84	C	850	500	2	98.1	0.0	1.8	-1.2	-1.3	0.8
MRS2.2-11A	2/6/84	C	850	2500	2	98.8	0.0	1.6	-1.7	0.1	0.5
MRS2.2-12	6/15/84	A	850	2500	10	99.6	0.8	-1.5	0.2	2.5	-1.2
MRS2.2-13	6/15/84	B	850	2500	10	98.9	0.2	-1.7	-0.9	2.6	-0.3
MRS2.2-14	6/15/84	D	850	2500	10	98.7	0.0	-2.9	-0.4	2.1	1.2
MRS2.2-15	6/15/84	A	700	2500	10	98.6	0.1	-1.2	0.2	1.3	0.0
MRS2.2-16	6/15/84	B	700	2500	10	100.6	0.0	-0.6	-0.4	2.7	-1.2
MRS2.2-17	6/15/84	D	700	2500	10	99.7	0.0	-3.2	0.6	2.3	0.7

* NET CONVERSION OF FEED SOLVENT TO SPECIFIED COMPONENT, WT %.

PAS=PREASPHALTENES; AS=ASPHALTENES; INSOL=INSOLUBLE MATTER (IOM+ASH).

THE YIELDS TABULATED ABOVE ARE PRELIMINARY, PENDING MINOR ADJUSTMENTS DUE TO ADDITIONAL ANALYTICAL DATA.

FEED TO MICROAUTOCLAVE: 10 GRAMS SOLVENT.

SOLVENT ANALYSES, WT%,

SOLVENT	C	H	N	S	OIL	AS	PAS	INSOL
A	91.2	6.1	0.9	0.9	91.3	3.8	3.2	1.6
B	91.7	7.6	0.6	0.3	92.7	3.7	2.4	1.2
C	87.4	7.5	0.5	0.3	94.3	3.1	1.4	1.2
D	90.3	8.3	0.3	0.1	91.9	3.8	4.4	0.0

TABLE 4

ANALYTICAL DATA FOR SCT PRODUCTS

SUBTASK 2.2 - SCT DISSOLVER WITH SOLVENTS ONLY

RUN #	----- ULTIMATE, WT % DRY -----				----- OAPI, WT. % DRY *-----			
	C	H	N	S	OIL	AS	PAS	INSOL
MRS2.2-12	89.3	5.6	.74	0.94	93.81	4.07	1.74	0.38
MRS2.2-13	91.0	7.1	.61	0.25	95.53	2.82	0.77	0.88
MRS2.2-14	90.3	8.4	.59	0.23	93.93	3.32	1.49	1.25
MRS2.2-15	90.4	5.8	.77	0.90	92.31	4.01	2.03	1.66
MRS2.2-16	91.0	7.2	.84	0.34	94.87	3.29	1.83	0.00
MRS2.2-17	90.0	8.4	.49	0.18	93.83	4.31	1.19	0.67

* PAS=PREASPHALTENES; AS=ASPHALTENES; INSOL=INSOLUBLE MATTER (IOM+ASH).

TABLE 5

HYDROTREATING/HYDROCRACKING CATALYSTS

Subtask 2.3 - Solvent Hydrocracking with Catalysts

1.	Shell 324 Modified	- 1/32" Extrudate	- Ni Mo	- Base Catalyst
2.	Shell 317	- 1/32" Extrudate	- Ni Mo	- Hi HC Activity
3.	Amer. Cyan. 1442B	- 1/32" Extrudate	- Co Mo	
4.	Harshaw 4301 E	- 1/12" Extrudate	- Ni W	- Hi HC Activity
5.	Procatalyse HR-941	- 2/4 mm Bead	- Ni Mo	
6.	Amer. Cyan.	- Beaded	- Co Mo	
7.	Katalco NM-506	- 1/16" Extrudate	- Ni Mo	- Resid HT Catalyst
8.	Davison Minisphere-32		- Ni Mo	
9.	Katalco NM136	- 1/32" Extrudate	- Ni Mo	- Resid HT Support
10.	Procatalyse HR901	- 2/4 mm Bead	- Co Mo	

TABLE 6

MICROAUTOCLAVE CONDITIONS

SUBTASK 2.3 - HYDROCRACKING OF SOLVENT B

CATALYST IS PRESULFIDED SHELL MODIFIED 324, 1/32" EXTRUDATES

RUN NO.	RUN DATE	REACTOR TEMPERATURE, F.	REACTOR PRESSURE, PSIG.	REACTION TIME, MIN.
-----	----	-----	-----	-----
MRS2.3-1	6/19/84	700	1000	60
MRS2.3-2	6/19/84	700	1000	120
MRS2.3-3	6/20/84	700	2000	60
MRS2.3-4	6/21/84	700	2000	120
MRS2.3-5A	6/25/84	850	1000	60
MRS2.3-5B	6/25/84	850	1000	60
MRS2.3-6	6/25/84	850	1000	120
MRS2.3-7	6/22/84	850	2000	60
MRS2.3-8	6/22/84	850	2000	120
MRS2.3-9	6/21/84	775	1500	90
MRS2.3-10	6/21/84	650	2500	30
MRS2.3-11	6/22/84	900	2500	30

FEED TO MICROAUTOCLAVE: 10 GRAMS SOLVENT B. SOLVENT B IS A 85/15 WEIGHT PERCENT BLEND OF LUMMUS-HYDROGENATED KC OIL AND SCT EXTRACT FROM ILLINOIS NO. 6 COAL.

TABLE 7

M I C R O A U T O C L A V E   C O N D I T I O N S

SUBTASK 3.1 - SCT DISSOLVER WITH SOLVENT A & ILLINOIS #6 COAL

NITROGEN ATMOSPHERE

RUN NO.	RUN DATE	REACTOR TEMPERATURE, F.	REACTOR PRESSURE, PSIG.	REACTION TIME, MIN
-----	----	-----	-----	-----
MRS3.1-55A/B	6/28/84	700	500	2
MRS3.1-56A/B	6/28/84	700	500	10
MRS3.1-57A/B	6/27/84	775	1500	6
MRS3.1-58A/B	6/18/84	850	500	2
MRS3.1-59A/B	6/18/84	850	2500	2
MRS3.1-60A/B	6/18/84	850	2500	10
MRS3.1-61A/B	6/27/84	900	2000	8

FEED TO MICROAUTOCLAVE: 10 GRAMS SOLVENT A AND 5.5 GRAMS MOISTURE-FREE ILLINOIS #6 COAL. SOLVENT A IS A 85/15 WEIGHT PERCENT BLEND OF RAW KC-OIL AND SCT EXTRACT FROM ILLINOIS #6 COAL.

TABLE 8

M I C R O A U T O C L A V E   C O N D I T I O N S

SUBTASK 3.1 - SCT DISSOLVER WITH SOLVENT B & ILLINOIS # 6 COAL

NITROGEN ATMOSPHERE

RUN NO.	RUN DATE	REACTOR TEMPERATURE, F.	REACTOR PRESSURE, PSIG.	REACTION TIME, MIN.
-----	----	-----	-----	-----
MRS3.1-62A/B	6/28/84	700	500	2
MRS3.1-63A/B	6/28/84	700	2500	2
MRS3.1-64A/B	6/29/84	700	500	10
MRS3.1-65A/B	6/29/84	700	2500	10
MRS3.1-66A/B	6/27/84	775	1500	6
MRS3.1-67A/B	6/26/84	850	500	2
MRS3.1-68A/B	6/26/84	850	2500	2
MRS3.1-69A/B	6/18/84	850	500	10
MRS3.1-70A/B	6/26/84	850	2500	10
MRS3.1-71A/B	6/27/84	900	2000	8
MRS3.1-72A/B	6/29/84	650	1000	4

FEED TO MICROAUTOCLAVE: 10 GRAMS SOLVENT B AND 5.5 GRAMS MOISTURE-FREE ILLINOIS #6 COAL. SOLVENT B IS A 85/15 WEIGHT PERCENT BLEND OF LUMMUS HYDROGENATED KC-OIL AND SCT EXTRACT FROM ILLINOIS #6 COAL.

TABLE 9

M I C R O A U T O C L A V E   C O N D I T I O N S

SUBTASK 3.1 - SCT DISSOLVER WITH SOLVENT C & ILLINOIS # 6 COAL

NITROGEN ATMOSPHERE

RUN NO.	RUN DATE	REACTOR TEMPERATURE, F.	REACTOR PRESSURE, PSIG.	REACTION TIME, MIN.
-----	----	-----	-----	-----
MRS3.1-37A/B	4/25/84	700	500	2
MRS3.1-38A/B	4/25/84	700	2500	2
MRS3.1-39A/B	4/25/84	700	500	10
MRS3.1-40A/B	4/25/84	700	2500	10
MRS3.1-41A/B	4/30/84	775	1500	6
MRS3.1-42A/B	4/27/84	850	500	2
MRS3.1-43A/B	4/27/84	850	2500	2
MRS3.1-44A/B	4/27/84	850	500	10
MRS3.1-45A/B	4/27/84	850	2500	10
MRS3.1-46A/B	4/30/84	900	2000	8
MRS3.1-47A/B	4/30/84	650	1000	4

FEED TO MICROAUTOCLAVE: 10 GRAMS SOLVENT C AND 5.5 GRAMS MOISTURE-FREE ILLINOIS #6 COAL. SOLVENT C IS A 42.5/40/17.5 WEIGHT PERCENT BLEND OF RAW KC-OIL, XYTEL HYDROGENATED KC-OIL AND SCT EXTRACT FROM ILLINOIS #6 COAL.

TABLE 10

M I C R O A U T O C L A V E   C O N D I T I O N S

SUBTASK 3.1 - SCT DISSOLVER WITH SOLVENT D & ILLINOIS # 6 COAL

NITROGEN ATMOSPHERE

RUN NO.	RUN DATE	REACTOR TEMPERATURE, F.	REACTOR PRESSURE, PSIG.	REACTION TIME, MIN.
-----	----	-----	-----	-----
MRS3.1-48A/B	5/4/84	700	500	2
MRS3.1-49A/B	5/4/84	700	500	10
MRS3.1-50A/B	5/4/84	775	1500	6
MRS3.1-51A/B	5/3/84	850	500	2
MRS3.1-52A/B	5/3/84	850	2500	2
MRS3.1-53A/B	5/3/84	850	2500	10
MRS3.1-54A/B	5/3/84	900	2000	8

FEED TO MICROAUTOCLAVE: 10 GRAMS SOLVENT D AND 5.5 GRAMS MOISTURE-FREE ILLINOIS #6 COAL. SOLVENT D IS A 80/20 WEIGHT PERCENT BLEND OF XYTEL HYDROGENATED KC-OIL AND SCT EXTRACT FROM ILLINOIS #6 COAL.

TABLE 11

ANALYTICAL DATA FOR SCT PRODUCTS

SUBTASK 3.1 - SCT DISSOLVER WITH ILLINOIS #6 COAL

DATA FOR SOLVENT A & HYDROGEN

RUN #	----- ULTIMATE, WT % DRY -----				----- OAPI, WT. % DRY *-----			
	C	H	N	S	OIL	AS	PAS	INSOL
MRS3.1-23A	83.53	5.58	.99	1.15	54.4	3.99	6.74	34.87
MRS3.1-23B	83.53	5.58	.99	1.15	55.38	4.02	7	33.6
MRS3.1-23C	82.29	5.64	.97	1.53	56.43	3.59	7.87	32.11
MRS3.1-23D	82.29	5.64	.97	1.53	56.56	4.60	7.78	31.06
MRS3.1-24A	84.27	5.56	.97	1.24	55.63	4.67	8.77	30.93
MRS3.1-24B	84.27	5.56	.97	1.24	55.47	5.27	9.07	30.19
MRS3.1-24C	81.52	5.42	.94	1.51	55.60	5.86	13.08	25.46
MRS3.1-24D	81.52	5.42	.94	1.51	56.94	3.85	12.19	27.02
MRS3.1-25A	85	5.74	1.03	1.92	59.84	5.54	12.81	21.81
MRS3.1-25B	85	5.74	1.03	1.92	57.54	7.67	12.34	22.45
MRS3.1-25C	83.41	5.66	1.15	1.77	60.57	5.77	19.17	14.49
MRS3.1-25D	83.41	5.66	1.15	1.77	59.23	6.39	20.47	13.91
MRS3.1-26A	85.08	5.42	1.05	2.13	57.71	6.9	18.59	16.8
MRS3.1-26B	85.08	5.42	1.05	2.13	58.4	6.22	18.49	16.89
MRS3.1-27A	84.42	5.76	1.13	2.53	62.82	6.79	13.69	16.7
MRS3.1-27B	84.42	5.76	1.13	2.53	62.88	6.08	14.76	16.28
MRS3.1-28A	82.61	5.47	.96	1.52	65.39	6.23	14.08	14.3
MRS3.1-28B	82.61	5.47	.96	1.52	66.09	6.85	13.96	13.1
MRS3.1-28C	83.65	5.61	1.05	1.58	65.73	8.08	14.97	11.22
MRS3.1-28D	83.65	5.61	1.05	1.58	66.52	7.69	14.45	11.34
MRS3.1-29A	83.22	5.75	1.07	1.08	65.62	5.74	12.12	16.52
MRS3.1-29B	83.22	5.75	1.07	1.08	64.96	6.63	12.28	16.13

* PAS=PREASPHALTENES; AS=ASPHALTENES; INSOL=INSOLUBLE MATTER (IOM+ASH).

TABLE 12

ANALYTICAL DATA FOR SCT PRODUCTS

SUBTASK 3.1 - SCT DISSOLVER WITH ILLINOIS #6 COAL

DATA FOR SOLVENT B & HYDROGEN

RUN #	----- ULTIMATE, WT % DRY -----				----- OAPI, WT. % DRY *-----			
	C	H	N	S	OIL	AS	PAS	INSOL
MRS3.1-1A	82.3	6.6	.88	1.35	60.43	6.71	13.11	19.75
MRS3.1-1B	82.3	6.6	.88	1.35	60.21	5.6	15.57	18.62
MRS3.1-2A	82.6	6.6	.91	1.35	60.70	6.20	13.74	19.36
MRS3.1-2B	82.6	6.6	.91	1.35	59.77	5.87	15.52	18.84
MRS3.1-3A	84.2	6.6	.87	1.06	74.52	9.87	10.24	5.37
MRS3.1-3B	84.2	6.6	.87	1.06	75.7	9.53	9.49	5.28
MRS3.1-4A	81.9	6.7	.86	.91	75.9	9.27	9.44	5.39
MRS3.1-4B	81.9	6.7	.86	.91	73.6	10.11	9.98	6.31
MRS3.1-5A	80.1	6.5	.92	1	76.53	9.55	6.68	7.24
MRS3.1-5B	80.1	6.5	.92	1	76.92	8.87	7.71	6.5
MRS3.1-6A	83.8	6.8	.9	1.51	63.12	7.21	17.24	12.43
MRS3.1-6B	83.8	6.8	.9	1.51	63.7	7.45	17.28	11.57
MRS3.1-7A	82.7	6.6	.91	1.44	60.17	6.77	7.27	25.79
MRS3.1-7B	82.7	6.6	.91	1.44	59.38	6.43	8.26	25.93
MRS3.1-8A	82.9	6.7	.89	1.53	59.37	6.59	8.67	25.37
MRS3.1-8B	82.9	6.7	.89	1.53	59.53	5.19	9.38	25.9
MRS3.1-9A	80.9	6.5	.9	1.55	60.08	4.33	10.72	24.87
MRS3.1-9B	80.9	6.5	.9	1.55	59.07	6.51	9.06	25.36
MRS3.1-10A	85.2	6.6	.92	1.41	66.14	8.82	16.06	8.98
MRS3.1-10B	85.2	6.6	.92	1.41	66.78	8.5	18.94	5.78
MRS3.1-11A	82.8	6.8	.9	1.24	67.44	9.97	14.31	8.28
MRS3.1-11B	82.8	6.8	.9	1.24	67.84	8.74	14.58	8.84

* PAS=PREASPHALTENES; AS=ASPHALTENES; INSOL=INSOLUBLE MATTER (IOM+ASH).

TABLE 13

ANALYTICAL DATA FOR SCT PRODUCTS

SUBTASK 3.1 - SCT DISSOLVER WITH ILLINOIS #6 COAL

DATA FOR SOLVENT C

RUN #	ULTIMATE, WT % DRY				OAPI, WT. % DRY *			
	C	H	N	S	OIL	AS	PAS	INSOL
MRS3.1-12A	80.5	6.3	.87	1.69	57.57	6.03	11.72	24.68
MRS3.1-12B	80.5	6.3	.87	1.69	58.23	5.17	11.28	25.32
MRS3.1-13A	77.13	6.1	.85	1.92	55.8	7.1	9.32	27.78
MRS3.1-13B	77.13	6.1	.85	1.92	56.4	6.33	10.95	26.32
MRS3.1-14A	82.9	6	.91	1.39	68.77	8.96	12.04	10.23
MRS3.1-14B	82.9	6	.91	1.39	67.71	9.25	11.85	11.19
MRS3.1-15A	83.3	6.1	.95	1.29	65.96	9.25	16.59	8.2
MRS3.1-15B	83.3	6.1	.95	1.29	72.45	10.65	12.56	4.34
MRS3.1-15C	81.03	6.62	.87	.68	72.94	12.80	6.76	7.50
MRS3.1-15D	81.03	6.62	.87	.68	72.94	12.97	7.77	6.32
MRS3.1-16A	79.8	6.26	.85	.83	73.1	8.2	13.5	5.2
MRS3.1-16B	79.8	6.26	.85	.83	72.79	8.04	12.43	6.74
MRS3.1-17A	81.7	6.53	.83	1.08	61.92	6.67	25.26	6.15
MRS3.1-17B	81.7	6.53	.83	1.08	60.72	6.99	25.20	7.09
MRS3.1-17C	81.65	6.59	.93	.85	63.78	7.94	14.65	13.63
MRS3.1-17D	81.65	6.59	.93	.85	63.16	11.52	11.17	14.15
MRS3.1-18A	80.9	6.41	.85	1.03	57.67	3.95	8.58	29.8
MRS3.1-18B	80.9	6.41	.85	1.03	59.44	2.96	7.06	30.54
MRS3.1-19A	82.35	6.6	.89	1.6	59.77	3.8	9.63	26.8
MRS3.1-19B	82.35	6.6	.89	1.6	59.65	4.5	9.35	26.5
MRS3.1-20A	80.53	6.5	.86	1.47	59.71	3.87	9.8	26.62
MRS3.1-20B	80.53	6.5	.86	1.47	60.62	3.74	9.85	25.79
MRS3.1-21A	81.24	6.48	.8	1.63	66.36	8.6	14.5	10.54
MRS3.1-21B	81.24	6.48	.8	1.63	65.47	8.68	14.87	10.98
MRS3.1-22A	80.8	6.38	.87	1.42	68.31	8.99	15.45	7.25
MRS3.1-22B	80.8	6.38	.87	1.42	67.17	12.01	13.04	7.78
MRS3.1-37A	81.76	6.31	.82	1.38	57.64	4.68	10.61	27.07
MRS3.1-37B	81.76	6.31	.82	1.38	55.78	4.23	11.48	28.51
MRS3.1-38A	81.56	6.23	.82	1.41	57.07	3.71	11.21	28.01
MRS3.1-38B	81.56	6.23	.82	1.41	56.08	4.44	10.74	28.74
MRS3.1-39A	81.55	6.26	.79	1.47	57.21	5.11	14	23.68
MRS3.1-39B	81.55	6.26	.79	1.47	58.95	4.55	12.79	23.71
MRS3.1-40A	82.14	6.55	.79	1.7	59.51	6.49	14.25	19.75
MRS3.1-40B	82.14	6.55	.79	1.7	59.93	5.29	14.22	20.56
MRS3.1-41A	81.58	6.09	.75	1.55	61.72	7.86	17.89	12.53
MRS3.1-41B	81.58	6.09	.75	1.55	62.26	7.97	17.64	12.13
MRS3.1-42A	83.21	6.22	.8	1.29	63.27	7.41	15.94	13.38
MRS3.1-42B	83.21	6.22	.8	1.29	62.57	9.3	15.77	12.36
MRS3.1-43A	82.56	6.13	.79	1.39	64.19	7.93	16.23	11.65
MRS3.1-43B	82.56	6.13	.79	1.39	64.75	7.90	16.07	11.28
MRS3.1-44A	82.42	5.69	.8	1.13	66.7	7.77	12.53	13
MRS3.1-44B	82.42	5.69	.8	1.13	66.28	7.76	12.31	13.63
MRS3.1-45A	86.01	5.94	.84	1.13	66.91	7.26	11.54	14.29
MRS3.1-45B	86.01	5.94	.84	1.13	64.81	11.58	9.06	14.55
MRS3.1-46A	83.92	5.35	.83	1.26	64.17	6.04	12.47	17.32
MRS3.1-46B	83.92	5.35	.83	1.26	62.62	7.19	15.9	14.29
MRS3.1-47A	81.92	6.05	.78	1.44	55.90	4.83	9.54	29.73
MRS3.1-47B	81.92	6.05	.78	1.44	57.38	4.48	8.69	29.45

NOTE: RUNS 12-22 USE HYDROGEN ATMOSPHERE; RUNS 37-47 USE NITROGEN ATMOSPHERE.

* PAS=PREASPHALTENES; AS=ASPHALTENES; INSOL=INSOLUBLE MATTER (IOM+ASH).

TABLE 14

ANALYTICAL DATA FOR SCT PRODUCTS

SUBTASK 3.1 - SCT DISSOLVER WITH ILLINOIS #6 COAL

DATA FOR SOLVENT D

RUN #	----- ULTIMATE, WT % DRY -----				----- OAPI, WT. % DRY *-----			
	C	H	N	S	OIL	AS	PAS	INSOL
MRS3.1-30A	81.86	7.38	.73	.99	59.57	3.09	8.53	28.81
MRS3.1-30B	81.86	7.38	.73	.99	58.57	3.11	9.04	29.28
MRS3.1-31A	82.23	7.5	.69	1.02	59.44	5.08	13.49	21.99
MRS3.1-31B	82.23	7.5	.69	1.02	63.07	4.56	14.14	18.23
MRS3.1-32A	81.44	7.32	.69	.78	63.32	6.27	16.45	13.96
MRS3.1-32B	81.44	7.32	.69	.78	60.70	5.45	17.56	14.29
MRS3.1-33A	82.76	7.47	.7	.56	66.64	8.25	16.46	8.65
MRS3.1-33B	82.76	7.47	.7	.56	64.84	8.05	18.55	8.56
MRS3.1-34A	82.3	7.42	.71	.91	67.06	9.33	20.42	3.19
MRS3.1-34B	82.3	7.42	.71	.91	67.91	8.72	17.35	6.02
MRS3.1-35A	82.89	7.37	.67	1.02	70.62	8.69	12.83	7.86
MRS3.1-35B	82.89	7.37	.67	1.02	71.44	10.5	10.66	7.4
MRS3.1-36A	84.65	7.19	.6	.63	74.78	7.81	10.58	6.83
MRS3.1-36B	84.65	7.19	.6	.63	73.74	8.19	12.4	5.67
MRS3.1-48A	80.85	7.07	.76	1.15	59.44	3.94	10.9	25.72
MRS3.1-48B	80.85	7.07	.76	1.15	59.86	4.44	11.64	24.06
MRS3.1-49A	81.79	7.26	.74	1.13	59.65	5.93	13.53	20.89
MRS3.1-49B	81.79	7.26	.74	1.13	60.9	5.34	13.83	19.93
MRS3.1-50A	80.74	6.99	.74	.95	62.57	8.07	18.32	11.04
MRS3.1-50B	80.74	6.99	.74	.95	62.08	8.20	17.83	11.89
MRS3.1-51A	82.2	6.72	.6	.95	69.38	9.62	12.78	8.22
MRS3.1-51B	82.2	6.72	.6	.95	67.72	9.65	14.65	7.98
MRS3.1-52A	82.39	6.72	.63	.83	67.25	10.17	14.15	8.43
MRS3.1-52B	82.39	6.72	.63	.83	68.09	10.42	13.73	7.76
MRS3.1-53A	81.58	6.72	.66	.82	71.41	10.4	9.61	8.58
MRS3.1-53B	81.58	6.72	.66	.82	71.37	9.72	10.77	8.14
MRS3.1-54A	84.1	6.59	.66	.66	72.31	9.59	8.36	9.74
MRS3.1-54B	84.1	6.59	.66	.66	73.11	8.39	8.96	9.54

NOTE: RUNS 30-36 USE HYDROGEN ATMOSPHERE; RUNS 48-54 USE NITROGEN ATMOSPHERE.

* PAS=PREASPHALTENES; AS=ASPHALTENES; INSOL=INSOLUBLE MATTER (IOM+ASH).

TABLE 15

CALCULATED RESULTS FROM OAPI ANALYSES OF LIQUID PRODUCTS

SUBTASK 3.1 - SCT DISSOLVER WITH ILLINOIS #6 COAL

DATA FOR SOLVENT A

RUN #	ATMOS- PHERE	TEMP F	PRESS PSIG	TIME MIN	---COAL CONVERSIONS, MAF BASIS---			
					TOTAL	PAS	AS	OIL
MRS3.1-23A	H2	700	500	2	9.5	14.2	6.4	-21.2
MRS3.1-23B	H2	700	500	2	13.4	15.0	6.5	-18.2
MRS3.1-23C	H2	700	500	2	16.3	18.0	5.4	-12.5
MRS3.1-23D	H2	700	500	2	19.7	17.7	8.5	-12.0
MRS3.1-24A	H2	700	500	10	22.3	20.2	8.4	-18.9
MRS3.1-24B	H2	700	500	10	24.6	21.1	10.2	-19.4
MRS3.1-24C	H2	700	500	10	36.3	34.5	12.6	-13.3
MRS3.1-24D	H2	700	500	10	31.4	31.7	6.3	-9.1
MRS3.1-25A	H2	775	1500	6	49.8	-60.1	13.6	81.0
MRS3.1-25B	H2	775	1500	6	47.9	-61.5	20.0	74.1
MRS3.1-25C	H2	775	1500	6	71.6	52.2	11.9	-2.1
MRS3.1-25D	H2	775	1500	6	73.4	56.2	13.8	-6.2
MRS3.1-26A	H2	850	500	2	65.6	49.3	14.9	-14.3
MRS3.1-26B	H2	850	500	2	65.3	49.0	12.9	-12.1
MRS3.1-27A	H2	850	2500	2	65.5	35.0	14.7	2.5
MRS3.1-27B	H2	850	2500	2	66.7	38.2	12.6	2.7
MRS3.1-28A	H2	850	2500	10	71.8	37.1	13.5	14.6
MRS3.1-28B	H2	850	2500	10	75.5	36.7	15.4	16.7
MRS3.1-28C	H2	850	2500	10	81.7	39.2	18.8	13.2
MRS3.1-28D	H2	850	2500	10	81.3	37.7	17.7	15.5
MRS3.1-29A	H2	900	2000	8	65.3	30.7	11.8	13.8
MRS3.1-29B	H2	900	2000	8	66.5	31.2	14.5	11.8

THE YIELDS TABULATED ABOVE ARE PRELIMINARY, PENDING MINOR ADJUSTMENTS DUE TO ADDITIONAL ANALYTICAL DATA.

FEED TO MICROAUTOCLAVE: 10 GRAMS SOLVENT A AND 5.5 GRAMS MOISTURE-FREE ILLINOIS #6 COAL. SOLVENT A IS A 85/15 WEIGHT PERCENT BLEND OF RAW KC-OIL AND SCT EXTRACT FROM ILLINOIS #6 COAL.

PAS = PREASPHALTENES; AS = ASPHALTENES

SOLVENT ANALYSES, WT%,

RUNS	C	H	N	S	OIL	AS	PAS	INSOL
23-29	90.1	6.1	0.9	0.6	92.2	2.8	3.1	1.8

TABLE 16

CALCULATED RESULTS FROM OAPI ANALYSES OF LIQUID PRODUCTS

SUBTASK 3.1 - SCT DISSOLVER WITH ILLINOIS #6 COAL

DATA FOR SOLVENT B

RUN #	ATMOS- PHERE	TEMP F	PRESS PSIG	TIME MIN	---COAL CONVERSIONS, MAF BASIS---			
					TOTAL	PAS	AS	OIL
MRS3.1-1A	H2	700	500	10	57.6	32.4	16.2	-4.9
MRS3.1-1B	H2	700	500	10	61.0	39.8	12.9	-5.6
MRS3.1-2A	H2	700	2500	10	59.0	34.2	14.6	-4.8
MRS3.1-2B	H2	700	2500	10	60.6	39.5	13.6	-7.6
MRS3.1-3A	H2	850	500	10	101.3	23.1	25.1	32.5
MRS3.1-3B	H2	850	500	10	101.6	20.9	24.1	35.9
MRS3.1-4A	H2	850	2500	10	101.0	21.2	23.7	40.0
MRS3.1-4B	H2	850	2500	10	98.1	23.0	26.5	34.7
MRS3.1-5A	H2	900	2000	8	95.2	13.2	24.9	45.0
MRS3.1-5B	H2	900	2000	8	97.0	16.8	23.5	51.1
MRS3.1-6A	H2	775	1500	6	80.4	44.0	17.3	-0.2
MRS3.1-6B	H2	775	1500	6	82.9	44.1	18.0	1.5
MRS3.1-7A	H2	650	1000	4	39.8	14.7	16.3	-6.6
MRS3.1-7B	H2	650	1000	4	39.4	17.7	15.3	-8.9
MRS3.1-8A	H2	700	500	2	41.3	18.9	15.7	-9.4
MRS3.1-8B	H2	700	500	2	39.7	21.0	11.5	-8.9
MRS3.1-9A	H2	700	2500	2	40.9	25.8	9.3	-2.8
MRS3.1-9B	H2	700	2500	2	39.4	20.7	16.0	-5.9
MRS3.1-10A	H2	850	500	2	91.0	39.7	21.7	5.5
MRS3.1-10B	H2	850	500	2	100.3	48.1	20.7	7.4
MRS3.1-11A	H2	850	2500	2	92.4	35.8	25.9	15.0
MRS3.1-11B	H2	850	2500	2	90.7	36.6	22.2	16.2

THE YIELDS TABULATED ABOVE ARE PRELIMINARY, PENDING MINOR ADJUSTMENTS DUE TO ADDITIONAL ANALYTICAL DATA.

FEED TO MICROAUTOCLAVE: 10 GRAMS SOLVENT B AND 5.5 GRAMS MOISTURE-FREE ILLINOIS #6 COAL. SOLVENT B IS A 85/15 WEIGHT PERCENT BLEND OF LUMMUS HYDROGENATED KC-OIL AND SCT EXTRACT FROM ILLINOIS #6 COAL.

PAS = PREASPHALTENES; AS = ASPHALTENES

SOLVENT ANALYSES, WT%,

RUNS	C	H	N	S	OIL	AS	PAS	INSOL
1-11	86.7	7.4	0.5	0.5	92.1	2.0	3.5	2.4

TABLE 17

CALCULATED RESULTS FROM OAPI ANALYSES OF LIQUID PRODUCTS

SUBTASK 3.1 - SCT DISSOLVER WITH ILLINOIS #6 COAL

DATA FOR SOLVENT C

RUN #	ATMOS- PHERE	TEMP F	PRESS PSIG	TIME MIN	---COAL CONVERSIONS, MAF BASIS---		
					TOTAL	PAS	AS
MRS3.1-12A	H2	700	500	10	36.2	31.4	14.1
MRS3.1-12B	H2	700	500	10	33.6	30.3	11.6
MRS3.1-13A	H2	700	2500	10	25.1	24.4	17.9
MRS3.1-13B	H2	700	2500	10	29.5	29.7	15.4
MRS3.1-14A	H2	850	500	10	83.0	30.8	22.1
MRS3.1-14B	H2	850	500	10	79.3	31.0	23.6
MRS3.1-15A	H2	850	2500	10	88.6	45.4	23.5
MRS3.1-15B	H2	850	2500	10	100.4	33.0	27.8
MRS3.1-15C	H2	850	2500	10	90.1	15.9	35.4
MRS3.1-15D	H2	850	2500	10	93.9	18.9	35.7
MRS3.1-16A	H2	900	2000	8	97.2	37.3	21.1
MRS3.1-16B	H2	900	2000	8	93.0	32.8	19.9
MRS3.1-17A	H2	775	1500	6	94.5	73.4	16.0
MRS3.1-17B	H2	775	1500	6	91.6	73.2	17.0
MRS3.1-17C	H2	775	1500	6	71.1	40.3	20.0
MRS3.1-17D	H2	775	1500	6	69.5	29.5	31.1
MRS3.1-18A	H2	650	1000	4	19.8	21.6	7.6
MRS3.1-18B	H2	650	1000	4	17.5	16.9	4.5
MRS3.1-19A	H2	700	500	2	30.8	24.4	7.0
MRS3.1-19B	H2	700	500	2	31.7	23.6	9.1
MRS3.1-20A	H2	700	2500	2	29.5	25.6	7.4
MRS3.1-20B	H2	700	2500	2	32.1	25.8	7.0
MRS3.1-21A	H2	850	500	2	80.6	40.1	22.2
MRS3.1-21B	H2	850	500	2	79.2	41.2	22.4
MRS3.1-22A	H2	850	2500	2	90.9	43.1	23.4
MRS3.1-22B	H2	850	2500	2	89.1	35.7	33.1
MRS3.1-37A	N2	700	500	2	31.0	27.4	6.8
MRS3.1-37B	N2	700	500	2	26.8	30.0	5.4
MRS3.1-38A	N2	700	2500	2	27.9	29.3	3.8
MRS3.1-38B	N2	700	2500	2	25.6	27.9	6.1
MRS3.1-39A	N2	700	500	10	41.3	38.0	8.2
MRS3.1-39B	N2	700	500	10	41.2	34.2	6.4
MRS3.1-40A	N2	700	2500	10	53.9	38.5	12.3
MRS3.1-40B	N2	700	2500	10	51.4	38.4	8.6
MRS3.1-41A	N2	775	1500	6	76.0	50.0	16.7
MRS3.1-41B	N2	775	1500	6	77.2	49.3	17.0
MRS3.1-42A	N2	850	500	2	74.1	43.0	14.9
MRS3.1-42B	N2	850	500	2	77.3	42.5	20.6
MRS3.1-43A	N2	850	2500	2	79.1	44.3	16.6
MRS3.1-43B	N2	850	2500	2	80.2	43.9	16.6
MRS3.1-44A	N2	850	500	10	74.9	33.0	16.2
MRS3.1-44B	N2	850	500	10	72.9	32.3	16.2
MRS3.1-45A	N2	850	2500	10	72.8	28.5	13.7
MRS3.1-45B	N2	850	2500	10	72.0	21.2	26.4
MRS3.1-46A	N2	900	2000	8	62.6	32.2	10.5
MRS3.1-46B	N2	900	2000	8	71.8	42.4	14.0
MRS3.1-47A	N2	650	1000	4	22.9	24.0	7.2
MRS3.1-47B	N2	650	1000	4	23.7	21.4	6.2

THE YIELDS TABULATED ABOVE ARE PRELIMINARY, PENDING MINOR ADJUSTMENTS DUE TO ADDITIONAL ANALYTICAL DATA.

FEED TO MICROAUTOCLAVE: 10 GRAMS SOLVENT C AND 5.5 GRAMS MOISTURE-FREE ILLINOIS #6 COAL. SOLVENT C IS A 42.5/40/17.5 WEIGHT PERCENT BLEND OF RAW KC-OIL, XYTEL HYDROGENATED KC-OIL AND SCT EXTRACT FROM ILLINOIS #6 COAL.

PAS = PREASPHALTENES; AS = ASPHALTENES

SOLVENT ANALYSES, WT%,

RUNS	C	H	N	S	OIL	AS	PAS	INSOL
12-22	90.0	7.7	0.6	0.2	94.3	2.4	2.6	0.7
37-47	89.3	7.5	0.8	0.4	92.2	3.8	2.7	1.3

TABLE 18

CALCULATED RESULTS FROM OAPI ANALYSES OF LIQUID PRODUCTS

SUBTASK 3.1 - SCT DISSOLVER WITH ILLINOIS #6 COAL

DATA FOR SOLVENT D

RUN #	ATMOS- PHERE	TEMP F	PRESS PSIG	TIME MIN	---COAL CONVERSIONS, MAF BASIS---			
					TOTAL	PAS	AS	OIL
MRS3.1-30A	H2	700	500	2	25.8	18.8	5.8	-7.8
MRS3.1-30B	H2	700	500	2	24.5	20.4	5.9	-10.8
MRS3.1-31A	H2	700	500	10	47.0	33.9	11.8	-9.1
MRS3.1-31B	H2	700	500	10	58.6	35.8	10.2	2.1
MRS3.1-32A	H2	775	1500	6	71.2	43.3	15.6	4.8
MRS3.1-32B	H2	775	1500	6	64.0	46.7	13.1	-3.3
MRS3.1-33A	H2	850	500	2	87.9	42.5	21.3	11.7
MRS3.1-33B	H2	850	500	2	88.2	48.9	20.7	6.2
MRS3.1-34A	H2	850	2500	2	104.4	54.9	24.8	14.1
MRS3.1-34B	H2	850	2500	2	95.8	45.5	22.9	16.7
MRS3.1-35A	H2	850	2500	10	90.4	31.5	22.6	23.4
MRS3.1-35B	H2	850	2500	10	91.7	24.9	28.1	25.9
MRS3.1-36A	H2	900	2000	8	94.1	23.6	19.2	28.4
MRS3.1-36B	H2	900	2000	8	97.4	29.4	20.6	28.3
MRS3.1-48A	N2	700	500	2	31.0	25.6	4.8	1.1
MRS3.1-48B	N2	700	500	2	36.2	27.9	6.4	2.4
MRS3.1-49A	N2	700	500	10	47.0	33.4	10.9	-0.4
MRS3.1-49B	N2	700	500	10	50.0	34.3	9.0	3.5
MRS3.1-50A	N2	775	1500	6	77.3	49.1	17.9	11.3
MRS3.1-50B	N2	775	1500	6	74.6	47.5	18.3	9.7
MRS3.1-51A	N2	850	500	2	86.7	30.8	22.2	28.9
MRS3.1-51B	N2	850	500	2	87.4	36.6	22.3	23.8
MRS3.1-52A	N2	850	2500	2	86.1	35.0	23.9	21.8
MRS3.1-52B	N2	850	2500	2	88.2	33.7	24.7	24.4
MRS3.1-53A	N2	850	2500	10	85.6	21.0	24.6	35.0
MRS3.1-53B	N2	850	2500	10	86.8	24.7	22.6	35.5
MRS3.1-54A	N2	900	2000	8	82.6	16.5	21.5	32.9
MRS3.1-54B	N2	900	2000	8	83.4	18.2	17.7	34.3

THE YIELDS TABULATED ABOVE ARE PRELIMINARY, PENDING MINOR ADJUSTMENTS DUE TO ADDITIONAL ANALYTICAL DATA.

FEED TO MICROAUTOCLAVE: 10 GRAMS SOLVENT D AND 5.5 GRAMS MOISTURE-FREE ILLINOIS #6 COAL. SOLVENT D IS A 80/20 WEIGHT PERCENT BLEND OF XYTEL HYDROGENATED KC-OIL AND SCT EXTRACT FROM ILLINOIS #6 COAL.

PAS = PREASPHALTENES; AS = ASPHALTENES.

SOLVENT ANALYSES, WT%,

RUNS	C	H	N	S	OIL	AS	PAS	INSOL
30-36	88.0	8.6	0.4	0.2	93.6	1.8	3.6	1.0
48-54	90.3	8.3	0.3	0.1	91.9	3.8	4.4	0.0

Figure 1

SOLVENT-ONLY REACTIONS IN HYDROGEN

Subtask 2.2

Figure 2

SOLVENT-ONLY REACTIONS IN HYDROGEN

Subtask 2.2

Figure 3
SOLVENT-ONLY REACTIONS IN HYDROGEN
Subtask 2.2

Figure 4

SOLVENT-ONLY REACTIONS IN HYDROGEN

Subtask 2.2

Figure 5

NET COAL CONVERSION VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT A & HYDROGEN

LEGEND: P

* * * 500

△ △ △ 1500

□ □ □ 2000

2500

SOLVENT A IS AN 85/15 WEIGHT PERCENT BLEND OF
RAW KC OIL AND ILLINOIS #8 SCT EXTRACT

Figure 6.

NET COAL CONVERSION VS. TEMPERATURE

SCT REACTION OF ILLINOIS #6 COAL IN SOLVENT D & HYDROGEN

LEGEND: P * * * 500 Δ Δ Δ 1500 □ □ □ 2000 # # # 2500

SOLVENT D IS AN 80/20 WEIGHT PERCENT BLEND OF
XYTEL HYDROGENATED KC-OIL AND ILLINOIS #6 SCT EXTRACT.

Figure 7

NET PREASPHALTENES VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT D + H₂

LEGEND: P * * * 500 Δ Δ Δ 1500 □ □ □ 2000 # # # 2500

SOLVENT D IS AN 80/20 WEIGHT PERCENT BLEND OF
XYTEL HYDROGENATED KC-OIL AND ILLINOIS #8 SCT EXTRACT.

PROC GPLOT.

Figure 8

NET ASPHALTENES VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT D + H₂

LEGEND: P * * * 500 Δ Δ Δ 1500 □ □ □ 2000 # # # 2500

SOLVENT D IS AN 80/20 WEIGHT PERCENT BLEND OF
XYTEL HYDROGENATED KC-OIL AND ILLINOIS #8 SCT EXTRACT.

PROC G PLOT

Figure 9

NET OIL VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT D + H₂

LEGEND: P * * * 500 Δ Δ Δ 1500 □ □ □ 2000 # # # 2500

SOLVENT D IS AN 80/20 WEIGHT PERCENT BLEND OF
XYTEL HYDROGENATED KC-OIL AND ILLINOIS #8 SCT EXTRACT. PROC GPLOT

Figure 10

NET COAL CONVERSION VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT D & NITROGEN

LEGEND: P   * * * 500   Δ Δ Δ 1500   □ □ □ 2000   # # # 2500

SOLVENT D IS AN 80/20 WEIGHT PERCENT BLEND OF
XYTEL HYDROGENATED KC-OIL AND ILLINOIS #8 SCT EXTRACT.

Figure 11

NET PREASPHALTENES VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT D & NITROGEN

LEGEND: P * * * 500 Δ Δ Δ 1500 □ □ □ 2000 # # # 2500

SOLVENT D IS AN 80/20 WEIGHT PERCENT BLEND OF
XYTEL HYDROGENATED KC-OIL AND ILLINOIS #8 SCT EXTRACT.

Figure 12

NET ASPHALTENES VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT D & NITROGEN

LEGEND: P   * * * 500   Δ Δ Δ 1500   □ □ □ 2000   # # # 2500

SOLVENT D IS AN 80/20 WEIGHT PERCENT BLEND OF
XYTEL HYDROGENATED KC-OIL AND ILLINOIS #8 SCT EXTRACT.

Figure 13

NET OIL VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT D & NITROGEN

LEGEND: P   * * * 500   Δ Δ Δ 1500   □ □ □ 2000   # # # 2500

SOLVENT D IS AN 80/20 WEIGHT PERCENT BLEND OF
XYTEL HYDROGENATED KC-OIL AND ILLINOIS #8 SCT EXTRACT.

Figure 14

NET COAL CONVERSION VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT C & NITROGEN

LEGEND: P

* * * 500

Δ Δ Δ 1000

□ □ □ 1500

2000

+ + + 2500

SOLVENT C IS A 42.5/40/17.5 WEIGHT PERCENT BLEND OF
RAW KC-OIL, XYTEL HYDROGENATED KC-OIL AND ILLINOIS #8
SCT EXTRACT.

Figure 15.

NET PREASPHALTENES VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT C & N2

LEGEND: P   * * * 500   Δ Δ Δ 1000   □ □ □ 1500   # # # 2000   + + + 2500

SOLVENT C IS A 42.5/40/17.5 WEIGHT PERCENT BLEND OF
RAW KC-OIL, XYTEL HYDROGENATED KC-OIL AND ILLINOIS #8
SCT EXTRACT.

Figure 16

NET ASPHALTENES VS. TEMPERATURE

SCT REACTION OF ILLINOIS #8 COAL IN SOLVENT C & N₂

LEGEND: P   * * * 500   Δ Δ Δ 1000   □ □ □ 1500   # # # 2000   + + + 2500

SOLVENT C IS A 42.5/40/17.5 WEIGHT PERCENT BLEND OF
RAW KC-OIL, XYTEL HYDROGENATED KC-OIL AND ILLINOIS #8
SCT EXTRACT.

Figure 17

NET OIL VS. TEMPERATURE

SCT REACTION OF ILLINOIS #6 COAL IN SOLVENT C & N2

Figure 18

COAL CONVERSION IN SOLVENTS C & D

Subtask 3.1 - REACTIONS IN H₂ & N₂

NET COAL CONVERSION, % MAF

Figure 19

COAL CONVERSION VS H-CONTENT OF SOLVENT H₂ ATMOSPHERE

700 F; 2 MIN
500 PSIG

++++

850 F; 10 MIN
2500 PSIG

****

FIGURE A

Appendix A
Calculation Methods for Yields and Conversions

SCT Coal Conversion Runs

The results are reported as conversions, on the basis of MAF coal fed to the reactor. The product and feed weights are calculated and reported by an elemental-material-balance program. They are not reported herein, due to the extensive nature of the material balance reports for the hundreds of microautoclave runs in this program.

(1) MAF Coal Conversion =

$$\left[\frac{(\text{g. dry coal fed}) + (\text{g. insols in solvent}) - (\text{g. insols in product oil})}{(\text{g. MAF coal fed})} \right] \times 100\%$$

(2) MAF Coal Conversion to PAS =

$$\left[\frac{(\text{g. PAS in TLP}) - (\text{g. PAS in solvent fed})}{(\text{g. MAF coal fed})} \right] \times 100\%$$

(3) MAF Coal Conversion to AS =

$$\left[\frac{(\text{g. AS in TLP}) - (\text{g. AS in solvent fed})}{(\text{g. MAF coal fed})} \right] \times 100\%$$

(4) MAF Coal Conversion to Oil =

$$\left[\frac{(\text{g. Oil in TLP}) - (\text{g. Oil in solvent fed})}{(\text{g. MAF coal fed})} \right] \times 100\%$$

Equation (1) is based on the assumption that ASH in = ASH out. The ash content of the TLP is not measured.

Nomenclature:

PAS = preasphaltenes (THF soluble/toluene insoluble)

AS = asphaltenes (Toluene soluble/heptane insoluble)

Oil = Oil (Heptane soluble)

TLP = Total Liquid Product from reactor, including solids

Adjusted Net Coal Conversions

The coal conversions discussed above are subject to bias due to thermally-induced changes in the solvent. Solvent-only yield data at SCT conditions may be used to correct for apparent yields caused by solvent-only reactions. These reactions are observed to be minor in extent.

Adjusted Net MAF Coal Conversion =

$$\left[\frac{(\text{g. dry coal fed}) - (\text{g. insols in TLP}) + [(\text{g. solvent})(1-X_{sg})(\text{Insol}_{rs})]}{(\text{g. MAF coal})} \right] \times 100\%$$

Adjusted Net Coal Conversion to PAS =

$$\left[\frac{(\text{g. PAS in TLP}) - [(\text{g. solvent})(1-X_{sg})(\text{PAS}_{rs})]}{(\text{g. MAF coal})} \right] \times 100\%$$

Adjusted Net Coal Conversion to AS =

$$\left[\frac{(\text{g. AS in TLP}) - [(\text{g. solvent})(1-X_{sg})(\text{AS}_{rs})]}{(\text{g. MAF coal})} \right] \times 100\%$$

Adjusted Net Coal Conversion to Oil =

$$\left[\frac{(\text{g. Oil in TLP}) - [(\text{g. solvent})(1-X_{sg})(\text{Oil}_{rs})]}{(\text{g. MAF coal})} \right] \times 100\%$$

X_{sg} = Wt. fraction conversion of solvent to gas in solvent-only reactions.

Insol_{rs} = Wt. fraction insolubles in reacted solvent from solvent-only reactions.

PAS_{rs} = Wt. fraction PAS in reacted solvent from solvent-only reactions.

AS_{rs} = Wt. fraction AS in reacted solvent from solvent-only reactions.

Oil_{rs} = Wt. fraction Oil in reacted solvent from solvent-only reactions.

DOE/PC/60047-T4

ADVANCED RESEARCH FOR THE CHARACTERIZATION OF HYDROGEN DONOR
SOLVENTS IN TWO-STAGE LIQUEFACTION

DOE