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DIRECT LIQUEFACTION OF LOW-RANK COAL

Quarterly Technical Progress Report

for the period January 1 - March 31, 1995

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QUARTERLY TECHNICAL PROGRESS REPORT
for the period January 1 - March 31, 1995

EXECUTIVE SUMMARY

A multistep direct liquefaction process specifically aimed at low-rank coals (LRCs) has been developed at the Energy & Environmental Research Center (EERC). The process consists of a preconversion treatment to prepare the coal for solubilization, solubilization of the coal in the solvent, and polishing using a phenolic solvent or solvent blend to complete solubilization of the remaining material. The product of these three steps can then be upgraded during a traditional hydrogenation step.

This project will address two research questions necessary for the further development and scaleup of this process: 1) determination of the recyclability of the solvent used during solubilization and 2) determination of the minimum severity required for effective hydrotreatment of the liquid product. The project will be performed as two tasks, the first consisting of ten recycle tests and the second consisting of twelve hydrotreatment tests performed at various conditions.

Several activities were performed during this quarter.

- The composite solvent was redistilled to verify that the light material could be quantitatively recovered from the heavier vehicle solvent fraction.

- Additional heavy fraction was prepared from coal-derived hydrogenated anthracene oil (HAO61) for use as the vehicle solvent during the Task 1 tests.

- A simulated product slurry was prepared and distilled to verify that the water, cresylic acid (POH), and HAO61 light fraction could be separated from the coal and/or coal-derived liquids (CDLs)/HAO61 heavy fraction following the polishing step.

- The high-pressure/temperature (HPT) equipment was prepared for the Task 1 operations. The existing gas-sampling system was modified, and the gas collection procedures for the new system were verified using both wet- and dry test meters. The scale used to weigh back the quench vessel was refurbished. A leak in the quench was repaired. The autoclave system in Bay 2 was pressure-tested. The pressure transducers, thermocouples, gas meters, CO and H_2 volumes, and the Fluke Hydra data bucket were calibrated. The Bay 2 autoclave system was shaken down.

- Task 1 operations, analytical work, and data reduction were initiated during this quarter. The first experimental matrix test was attempted. Unfortunately, the gas supplier had mistakenly charged methane into the CO feed gas cylinder, and the test had to be repeated. Appropriate cleanup to remove carbonized material from the system, additional analytical effort to determine the cause of the run failure, and

preparation of additional blended solvent set the Task 1 schedule back about two weeks. Test 1 was successfully completed on the second try. In addition, Test 2 was completed and the pretreatment and solubilization steps of Test 3 were performed.

The preliminary liquid balances for each step (i.e., pretreatment/solubilization step and polishing step) agreed well from one test to another, indicating that the tests are being performed and the products recovered in a consistent and predictable manner. It can also be concluded that the reaction chemistry for the individual steps is essentially the same for each of the tests since roughly the same relative quantity of liquid was produced. The pretreatment/solubilization liquid balances were significantly different from those of the polishing step, indicating that there was a difference in reaction chemistry between the pretreatment/solubilization and polishing steps.

Preliminary calculations of the carbon, hydrogen, and oxygen contents of the recycle solvent were compared. Although it is very early to look for trends in the composition of the recycle solvent, it appears that the solvent is becoming more carbon-rich and hydrogen- and oxygen-poor with each pass through the system.

1.0 INTRODUCTION

Direct liquefaction research at the Energy & Environmental Research Center (EERC) has, for a number of years, concentrated on developing a direct liquefaction process specifically for low-rank coals (LRCs) through the use of hydrogen-donating solvents and solvents similar to coal-derived liquids, the water/gas shift reaction, and lower-severity reaction conditions. The underlying assumption of all of the research was that advantage could be taken of the reactivity and specific qualities of LRCs to produce a tetrahydrofuran (THF)-soluble material that might be easier to upgrade than the soluble residuum produced during direct liquefaction of high-rank coals. A multistep approach was taken to produce the THF-soluble material, consisting of 1) preconversion treatment to prepare the coal for solubilization, 2) solubilization of the coal in the solvent, and 3) polishing to complete solubilization of the remaining material. The product of these three steps can then be upgraded during a traditional hydrogenation step.

To provide a preliminary comparison between the EERC process and existing direct liquefaction processes, product slurry produced during solubilization (Step 2) and polishing (Step 3) steps (i.e., without the Step 1 pretreatment) was catalytically hydrotreated to equilibrium based upon hydrogen uptake. The hydrotreatment was performed in this manner to define the practical upper limit of the product's hydrotreatability. The results were positive, and further tests were performed incorporating the pretreatment step (Step 1). Steps 1 through 3 (pretreatment, solubilization, and polishing) were performed in an integrated fashion. The products were catalytically hydrotreated to demonstrate the maximum hydrotreatability of the solubilized slurry and to provide products that could be compared to the products of existing processes.

The results of the EERC's research indicated that additional studies to more fully develop this process were justified. Two areas were targeted for further research: 1) determination of the recyclability of the solvent used during solubilization and 2) determination of the minimum severity required for hydrotreatment of the liquid product. This project addresses these two areas.

2.0 GOALS AND OBJECTIVES

The project goals are to determine the recyclability of the solvent used during solubilization and to determine the minimum hydrotreatment severity required to upgrade the liquid product of the multistep EERC process.

The project will be performed as two tasks. The first task will consist of ten recycle tests. Lignite will be solubilized via the pretreatment, solubilization, and polishing steps. The product of these three steps will be combined with a vehicle solvent and the resulting stream distilled to remove water, solubilization solvent, and oxygenated light, coal-derived liquids. The overheads will be further distilled to separate the water and light oil streams. The light oil will then be recycled for use as the solubilization solvent in the next test sequence. The analyses of the products of these tests will be used to characterize the recycle solvent stream and to calculate mass and material balances.

The second task will consist of a series of twelve hydrotreatment tests at various conditions. The tests will be performed according to a statistically designed experimental matrix to enable the identification and evaluation of the most effective low-severity hydrotreatment conditions. Analyses of the products of these tests will be used to characterize the hydrotreated product and to calculate mass and material balances.

3.0 ACCOMPLISHMENTS

3.1 Work Planned for This Quarter

Work originally scheduled for the quarter included performance of the Task 2 hydrotreatment severity tests. Because of scheduling changes made during the first quarter of the project, the Task 1 testing was rescheduled for performance during this quarter. Task 2 has been rescheduled for the April through June 1995 quarter.

3.2 Work Performed During This Quarter

Four different activities were performed during this quarter: 1) the distillation of the blended solvent was repeated; 2) additional HAO61 solvent was fractionated for use during the Task 1 tests; 3) the HPT equipment was prepared, calibrated, and shaken down prior to the initiation of the Task 1 tests; and 4) Task 1 operations began. A summary of tests performed during this quarter is presented in Table 1.

3.2.1 Redistillation of Composite Solvent

The September through December 1994 quarterly technical progress report mentioned that the distillation of the composite solvent would be repeated at an overheads temperature closer to 479 K (206°C) to be certain that the light material (cresylic acid [POH] and the light fraction of coal-derived, hydrogenated anthracene oil [HAO61]) could be quantitatively recovered from the HAO61 heavy fraction. The distillation was repeated as Run N598. During N598, the solvent cuts from Run N595 were combined and distilled at an overheads temperature of approximately 476 K (203°C). The temperature and pressure profiles of this distillation are shown in Figures 1 and 2. Table 2 compares the recovery results of this distillation with the results of the previous distillations of the same material. As the table shows, the quantity of light fraction collected was nearly identical to the quantity present in the composite solvent. The results of this distillation verified that the composite solvent can be reproducibly and quantitatively fractionated if the overheads temperature is kept close to 479 K (206°C).

3.2.2 Preparation of Additional HAO61 Heavy Fraction

Additional HAO61 solvent heavy fraction will be required to complete all ten of the Task 1 solvent recyclability tests. To provide the needed solvent fraction, HAO61 solvent was fractionated during Runs N601 and N604. The fractionation was performed in two steps: separating the nondistillable material from the distillable material and then fractionating the distillable material into light-, middle-, and heavy-oil fractions. The separation of distillable

TABLE 1

Summary of Tests Performed from January through March 1995

Run No.	Description	Mass Balance, %
N596	Autoclave Bay 2 calibration	NA[a]
N597	Bay 2 shakedown	100.74
N598	Redistillation of composite solvent	99.43
N599	Task 1, Test 1 pretreatment and solubilization steps[b]	98.83
N600	Task 1, Test 1 polishing step[b]	98.61
N601	Distillation of HAO61	98.94
N602	Repeat of Task 1, Test 1 pretreatment and solubilization steps	95.97
N603	Repeat of Task 1, Test 1 polishing step	98.28
N604	Fractionation of N601 overheads	99.54
N605	Distillation of simulated feed slurry	103.52
N606	Task 1, Test 1 distillation of polished product	97.86
N607	Task 1, Test 2 pretreatment and solubilization steps	98.62
N608	Verification of gas charge and recovery procedures	NA
N609	Task 1, Test 2 polishing step	98.74
N610	Task 1, Test 2 distillation of polished product	–[c]
N611	Task 1, Test 3 pretreatment and solubilization steps	91.56

[a] Not applicable.

[b] Runs in which CH_4 was inadvertently used as the reducing gas.

[c] Data not available yet.

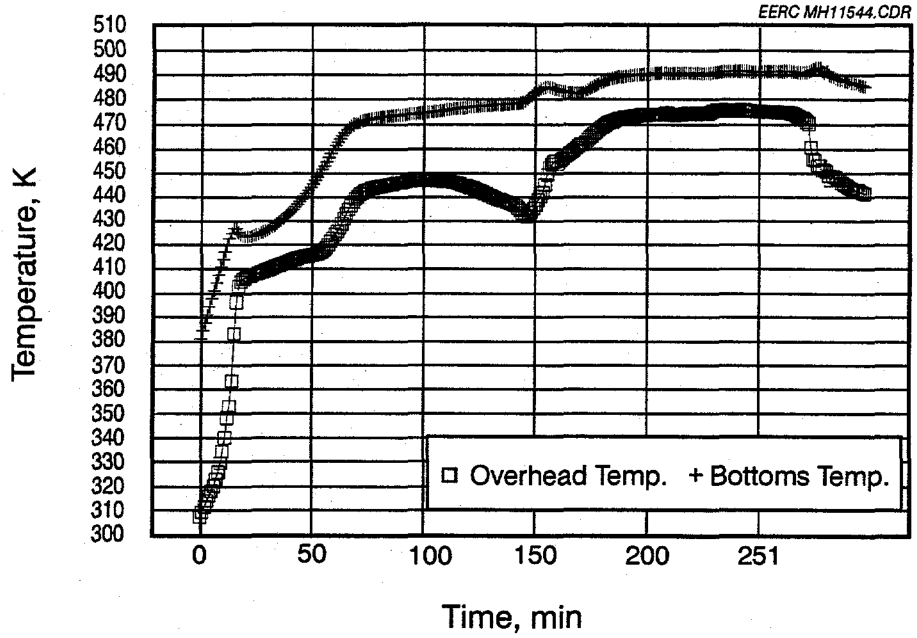

Figure 1. Temperature profile for redistillation of composite solvent.

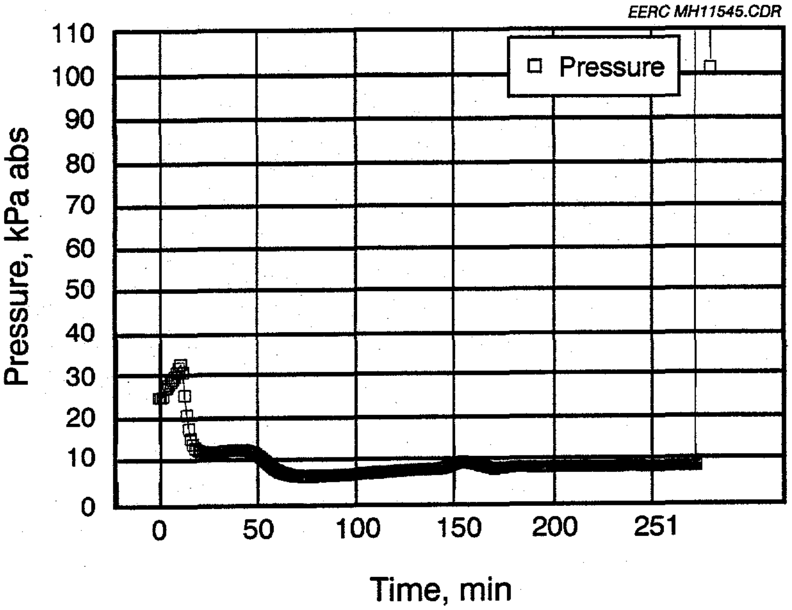

Figure 2. Pressure profile for redistillation of composite solvent.

TABLE 2

Summary of Composite Solvent Distillation

Run No.	HAO61 Heavy Fr., kg	HAO61 Light Fr., kg	POH, kg	Total Fed, kg	Distilled Heavy Fr., kg	Distilled Light Fr., kg	Total Out, kg	Distillation Losses, kg	% of Distillate	% Recovery
N594[a]	0.400	0.272	0.400	1.056	0.616	0.452	1.070	0.002	0.2	101.3
N595[a]				1.069	0.594	0.470	1.066	0.002	0.2	99.7
N598				1.048	0.402	0.638	1.042	0.006	0.6	99.4

[a] Test performed during September - December 1994 quarter; shown here for comparative purposes.

from nondistillable material was performed at an overheads temperature of about 558 K (285°C) and a pressure of about 15.24 kPa (2.21 psig). The distillable material made up 90.52 wt% of the total as-received HAO61 stream. The overheads from this separation were fractionated into light, middle, and heavy oils by vacuum distillation at about 13.55 kPa (1.96 psig) of pressure. The target pressure of 8.47 kPa could not be maintained. The distillation was therefore performed at conditions as close as possible to those of Run N593, the original HAO61 fractionation test. Vitron® hoses and heads were installed on the distillation unit to enable the target pressure to be attained during future distillations. The light-oil fraction made up 36.01 wt% of the total stream while the middle-oil fraction made up 13.16 wt%. Samples of each fraction were analyzed for comparison to the products of N593. Table 3 summarizes the HAO61 fractionation, and Table 4 compares the analyses of the N604 and N593 tests. As Table 4 shows, the N604 products were virtually identical to those of N593.

3.2.3 Preparation of HPT Equipment for Task 1

Several different activities were performed to prepare the HPT equipment for the Task 1 tests.

- The existing gas-sampling system was modified to permit collection of the product gas in a single vessel.
- The gas collection procedures for the new system were verified using both wet- and dry test meters.
- The scale used to weigh back the quench vessel was refurbished.
- A leak in the quench was repaired.
- The autoclave system in Bay 2 was pressure tested.
- The pressure transducers, thermocouples, gas meters, Fluke Hydra data bucket, and CO and H_2 volumes were calibrated.
- The autoclave system in Bay 2 was shaken down. The gas balance, liquid balance, and overall mass balance for the shakedown test were 103.88%, 99.09%, and 100.74%, respectively.

3.2.4 Distillation of a Simulated Product Slurry

A simulated product slurry was prepared and distilled to verify that the water, POH, and HAO61 light fraction could be separated from the coal and/or coal-derived liquids (CDLs)/HAO61 heavy fraction following the polishing step. The slurry contained 0.175 kg of POH, 0.176 kg of HAO61 light fraction, 0.351 kg of HAO61 heavy fraction, and 0.295 kg of Freedom lignite. Following water removal, the distillation was performed at 7.79 kPa (1.13 psig), 478 K (204.8°C) overheads temperature, and a 496 K (222.7°C) pot temperature. A 103.52% material balance was achieved. Analyses of the water and light-oil fractions have

TABLE 3

Summary of HAO61 Fractionation

N601	Separation of distillable and nondistillable material
	Performed at 15.24 kPa (2.21 psig) and 558 K (285°C)
	Results:
	Nondistillable material = 9.48 wt% of material fed
	Distillable material = 90.52 wt% of material fed (target = 90 wt%)
	Mass balance = 98.94%
N602	Fraction of distillable material from N601
	Performed at 13.55 kPa (1.96 psig)
	Results:
	Heavy fraction = 50.83 wt% of material fed
	Middle fraction = 13.16 wt% of material fed (target = 13.04 wt%)
	Light fraction = 36.01 wt% of material fed (target = 35.8 wt%)
	Mass balance = 99.54%

not been completed, but the material balance information and the easewith which the distillation was performed indicate that the light fractions can be quantitatively separated from the product stream.

3.2.5 Task 1 Operations

3.2.5.1 Test 1

Test 1 was attempted as Runs N599 and N600. The N599 feed consisted of 0.35 kg of Freedom lignite slurried in 0.7 kg of a 50:50 mixture of POH and HAO61 light fraction. The feed gas was supposed to be CO with H_2S used as the reaction promoter. The pretreatment was carried out for 30 minutes at 1.03×10^4 kPa (1500 psig) and 422 K (149°C). Following pretreatment, the temperature was increased and the solubilization step performed. Solubilization took place at 2.84×10^4 kPa (4125 psig) and 646 K (373°) for 60 minutes. The unit was quenched and the product recovered. The autoclave was filled with solids, an occurrence that had never happened during previous research using this particular process. Freedom lignite had never been used in the process before; it was decided to continue the sequence, performing the polishing step. Analytical tests were performed and operating conditions evaluated in an effort to determine why the solubilization step had not worked well. Run N600 was performed as the polishing step for the product slurry of Run N599.

TABLE 4

Comparison of N593 and N604 Distillation Products

Analysis	N593 Light Fr.	N604 Light Fr.	N593 Heavy Fr.	N604 Heavy Fr.
Carbon, wt%	90.59	90.35	91.86	91.58
Hydrogen, wt%	8.45	8.47	6.74	6.86
Nitrogen, wt%	0.56	0.70	0.63	0.72
Sulfur, wt%	0.02	0.00	0.16	0.19
Water, wt%	0.05	0.04	0.04	0.02
Distillation[a]				
IBP	313.8	313.8	410.4	402.6
5%	349.9	352.6	415.4	414.9
10%	354.9	356.0	421.0	420.4
20%	363.2	363.2	427.6	425.4
30%	372.1	371.5	432.6	430.4
40%	377.6	377.6	437.6	434.9
50%	384.3	386.0	443.2	441.0
60%	391.5	393.8	449.9	447.6
70%	399.9	404.3	458.2	454.3
80%	410.4	414.3	469.3	464.3
90%	426.5	431.5	479.9	478.8
95%	442.6	448.8	491.5	493.2
Max. Temp., K	444.3	449.9	494.3	496.0
% Residuum	1.70	1.40	1.65	1.83
% Distillable	96.39	97.11	97.34	97.06
% Loss	1.90	1.50	1.01	1.10

[a] Distillation data are in K unless otherwise noted.

Approximately 0.176 kg of a 50:50 mixture of POH and HAO61 light fraction were placed in the autoclave. The unit was charged to 6.21 × 10^3 kPa (900 psig) of H_2 and heated to 733 K (460°C). The N599 product slurry was charged to the system, held at run conditions for 20 minutes, and quenched.

Analysis of the N599 product gas revealed an extremely high concentration of methane. The newly delivered CO feed gas tank was sampled and found to be filled with methane. Further analyses of the products of either N599 or N600 were not performed. Preliminary data reduction was performed to check operator performance. The system was thoroughly cleaned and prepared for a second attempt at the data point. Approximately two weeks of operating time was lost to the gas supplier's mistake.

A second attempt at Test 1 was made during Run N602. For this run, 0.35 kg of Freedom lignite was slurried with 0.7 kg of a 50:50 mixture of POH and HAO61 light fraction and charged to the autoclave with CO and H_2S, which served as the reaction promoter. Pretreatment was performed at 425 K (152°C) and 9.67 × 10^3 kPa (1402 psig) for 35 minutes. The conditions were then increased to 650 K (377°C) and 2.40 × 10^4 kPa (3478 psig) for 60 minutes for the solubilization step. The unit was then quenched and the product recovered and sampled. The overall material balance for the test was 95.97%.

The N602 product slurry was polished during Run N603. The product slurry was charged to a preheated autoclave containing 0.179 kg of the startup solvent and H_2. The unit was operated for 20 minutes at reaction conditions of 714 K (441°C) and 3.18 × 10^4 kPa (4605 psig). The reaction was quenched and the product collected and sampled. An overall material balance of 98.29% was achieved for the run.

The polished product from N603 was distilled during N606 to remove the light fractions for use as Test 2 solvent feed. The N603 product slurry was combined with 0.79 kg of HAO61 heavy fraction (which would serve as the vehicle solvent for the product going into the hydrotreatment step). The water fraction was easily separated from the product slurry. A light oil fraction was removed at 8.23 kPa (1.19 psig), an overhead temperature of 471 K (198.2°C), and a pot temperature of 494 K (221°C). The overall material balance for the Test 1 distillation was 97.86%. After samples were taken for analysis, the light oil fraction was used as the feed solvent for Test 2.

3.2.5.2 Test 2

Approximately 0.325 kg of Freedom lignite was slurried with 0.65 kg of the light oil fraction from the Run N606 distillation. The autoclave was charged with slurry, CO, and H_2S. Run N607 consisted of a 30-minute pretreatment performed at 9.63 × 10^3 kPa (1397 psig) and 426 K (153°C), followed by solubilization at 2.43 × 10^4 kPa (3520 psig) and 650 K (377°C). The unit was quenched and the product recovered. An overall material balance of 98.62% was achieved for this run.

The product slurry from Run N607 was polished during Run N609. The slurry was hot-charged to an autoclave containing 0.162 kg of recycle solvent and H_2. The polishing step was performed at an average temperature of 713 K (440°C) and 3.13 × 10^4 kPa (4540 psig) for 20 minutes. The reaction was quenched and product recovered. A material balance of 98.74% was achieved for the polishing step.

The N609 product slurry was combined with HAO61 heavy fraction and distilled during Run N610. Water was removed and the distillation performed at endpoint conditions of 7.55 kPa (1.10 psig) and 492 K (219°C) to separate the light oil fraction from the heavier fraction. The light oil fraction was recycled for use as the feed solvent for Test 3.

3.2.5.3 Test 3

Run 611 was performed at the end of the quarter as the pretreatment/solubilization steps of Test 3. A material balance of 91.56% was achieved for this run. Details of this run will be presented and discussed in the next quarterly technical report.

3.2.5.4 Discussion

Table 5 presents the liquid balances for the pretreatment and solubilization steps for Tests 1-3 and the polishing step of Tests 1 and 2. As the table shows, the pretreatment/solubilization liquid balances agree well from one test to the next. The same can be said for the polishing step liquid balances for Tests 1 and 2. The fact that the balances are very similar indicates that the tests are being performed and the products recovered in a very consistent and predictable manner. It can also be concluded that the reaction chemistry for the individual steps (i.e., pretreatment/solubilization step and polishing step) is essentially the same for each of the tests since roughly the same relative quantity of liquid was produced. The pretreatment/solubilization liquid balances are significantly different from those of the polishing step, indicating that there is a difference in reaction chemistry between the pretreatment/solubilization and polishing steps.

Some of the analytical results needed for the calculation of detailed product slates for each of the tests are not yet available. Therefore, preliminary calculations were performed using the data that were available. Table 6 presents the preliminary calculations of the carbon, hydrogen, and oxygen contents of the feed solvent and the product of the polishing step for both Test 1 and Test 2. The values are presented on a nitrogen-, sulfur-, and moisture-free basis. Although it is very early to look for trends in the composition of the recycle solvent, it appears that the solvent is becoming more carbon-rich, hydrogen-poor, and oxygen-poor with each pass through the system. These trends are as would be expected.

3.3 Conclusions

- The light-oil fraction can be quantitatively recovered from the heavier vehicle solvent fraction.
- Water, cresylic acid, and HAO61 light fraction can be separated from the heavier fractions of the polishing step product slurry.

TABLE 5

Comparison of Liquid Balances for Task 1, Tests 1 through 3

Test Number	Pretreatment/ Solubilization, %	Polishing, %
1	91.25	97.30
2	91.69	97.31
3	91.56	NA[a]

[a]Test has not yet been completed.

TABLE 6

Preliminary C, H, and O Contents of Recycle Solvent[a]

Test Number	Carbon, wt%	Hydrogen, wt%	Oxygen, wt%
Feed Solvent	84.53	8.01	7.46
N606	86.03	7.93	6.04
N610	87.23	7.87	4.90

[a]Values are on a nitrogen-, sulfur-, and moisture-free basis.

- The liquid balances obtained on the product slurries from the Task 1 tests performed to date indicate that the tests are being performed in a consistent and predictable manner. The same balances also indicate that the reaction chemistry of the individual steps within a given test sequence are significantly different from each other, but that the chemistry of the same step is very similar from test to test.
- Although it is early in the task operations to look for trends in solvent recyclability, it appears that the expected trends are being followed: the solvent is becoming more carbon-rich and hydrogen- and oxygen-poor.

4.0 FUTURE OBJECTIVES

The Task 1 and Task 2 tests will be performed, the products analyzed, and the data reduced and interpreted. Quality assurance checks will be performed as outlined in the project quality assurance plan. Preparation of the final technical report will begin during the next quarter.