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ADVANCED DIRECT LIQUEFACTION CONCEPTS for PETC GENERIC UNITS Phase II

**Quarterly Technical Progress Report
for Period April through June 1996**

by

**University of Kentucky
Center for Applied Energy Research**

CONSOL Inc.

Hydrocarbon Technologies, Inc.

LDP Associates

MASTER

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SUMMARY

TASK 2.1 LABORATORY SUPPORT (UK/CAER)

Catalyst Development

A catalyst screening test (CST) was developed to evaluate the activity of various catalyst precursors for their liquefaction activity in a solvent comprising the solids-free components of a recycle solvent generated at Wilsonville, namely a ROSE SR V-130 deashed resid from period A and V-1074 heavy distillate from period B. Since the deashed resid has an elemental composition very nearly the same as in the solids-free fraction of the recycle solvent, the reactivity of these two resids may be nearly the same. In this test, since the relative amounts of distillate, deashed resid and dry coal are nearly the same as in Run 263J, the overall composition should approximate the feed stream used in the Wilsonville pilot plant except for the absence of the solids component. Removing the solids from the reaction mixture should simplify the interpretation of the results since normally a considerable amount of recycled catalyst is contained in this fraction.

An indication of an upper limit of resid conversion that might be expected in this test was obtained in a run in which crushed AKZO AO-60 was added to BT coal. The catalyst was added at a level of 10 wt% Mo+Ni on a dry coal basis. After 30 min at 440 °C, the 524 °C+ (975 °F+) resid conversion was 39.7% and coal conversion (THF) was 95.8%. Thus far, this is the highest 30 minute resid conversion that has been observed in this test.

The performance of the catalysts used in ALC-1 Condition #1, i.e. Molyvan A and HTI's proprietary FeOOH/SO₄ catalyst, was determined in the CST in order to have a comparison in our micro reactors. The activity of this catalyst was among the highest we observed for any dispersed catalyst in this test, i.e. 30.3% resid conversion. It was distinctly better than either the Fe or Mo components alone. It was also very sensitive to the presence of H₂S, since neither the Fe or Mo precursors performed well in the absence of H₂S.

Since in the microautoclaves there is no purging during the run, the water partial pressure due to the water in the coal as well as the water associated with the FeOOH catalyst could be affecting the activity of the Fe catalyst. To test this, the HTI FeOOH/SO₄ catalyst was dried before running in the micro reactors. One sample was prepared by calcining at 490°C for 3 h and a second was prepared by drying at 95°C and 250 torr overnight. In both cases the dried materials were humidified before use to assure that adequate water was present to hydrate the surface. The activities of both dried precursors were higher than the starting material indicating that water in these micro reactors will affect activities of the Fe catalysts. Also, a few runs were made to evaluate the use of elemental sulfur as sulfiding agent. Resid conversion when elemental sulfur was used was always lower than when H₂S was used.

In a series of runs, Mo, Ni/Mo and Fe/Mo impregnated coals were pretreated for 30 minute at temperatures from 275 °C to 440 °C and then reacted for 30 minutes at 440 °C. The Mo-only impregnated coals gave better resid conversion than the Fe/Mo impregnated coal. Overall, however,

Ni/Mo gives the best results with enhanced coal dissolution.

The effect of varying the Ni in Ni/Mo impregnated coals was investigated during this reporting period. Ni at levels of 100 and 500 ppm on coals with 500 ppm Mo were run without pretreatment. The higher Ni concentration gave approximately 29% resid conversion versus 27% for the run at 100 ppm. This suggests that the higher Ni concentration can have a beneficial effect. These two Ni concentrations were also evaluated in runs involving 30 min pretreatments at 300 °C. In these runs the 100 ppm Ni impregnated coal have a higher resid conversion (32%) than the 500 ppm coal (28%). With the 30 min pretreatment, a slight *decrease* in conversion is seen, but hydrogen consumption was found to be the same. In total, the data collected to date suggests that most of the improvements in conversion from added Ni can be accrued at low concentrations

The activity of phosphomolybdic acid impregnated onto coal was quite good giving up to 70% resid conversion (maf coal basis). This was significantly higher than the conversions observed when the solid acid was merely added to the reaction mixture. The experiments also indicate that the amount of water used in the impregnation affected the amount of resid conversion with larger amounts of water favoring higher conversion.

Liquefaction catalysis using ammonium tetrathiomolybdate (ATTM) as a precursor was also evaluated during this reporting period. Both as-received and dry coals impregnated with ATTM gave nearly the same 524 °C+ (975 °F+) resid conversions of 50.9 and 55.6%, respectively, in the presence of H₂S. The corresponding coal conversions were 85 and 89% in the presence of H₂S. Impregnating a coal with 100 ppm Mo from a 1% aqueous solution increases the moisture in the coal feed by less than 3 w%. Runs were also made in the absence of H₂S to determine whether H₂S had an effect on product distribution. Surprisingly, the yield of IOM products were considerably higher in the absence of H₂S, indicating a strong positive dependence on the presence of H₂S.

Coal Cleaning by Column Flotation

Column flotation is an advanced flotation technique which utilizes the conventional froth flotation principles. A low-ash Wyodak Black Thunder coal (80% passing 325 mesh) containing 5.12 wt% ash responded favorably to froth flotation technique. High recovery of combustibles were obtained at a low or moderate ash rejection, suggesting that a majority of ash may be present as 'inherent' ash. Both conventional and column flotation provided a 3.4% ash clean coal at 75% combustible recovery at a ash rejection of about 65%. Experiments were performed on minus 1/4 in crushed coal. An average particle size after 10 minutes grind was about 11 µm. Froth flotation tests were conducted using a laboratory Denver flotation machine giving a product having an ash content as low as 3.9% and combustible recovery of 52.5%. This was obtained using 2 lb/t of fuel oil and MIBC. From a release analysis it was determined that 3.7% ash clean coal could be obtained at 80% combustible recovery. A 3% ash clean coal could be obtained at only 30% combustible recovery.

Column flotation studies were conducted using a 2-in. I.D., 20-ft. high column. As expected, both ash content and combustible recovery increased with increasing air flow rate. The lowest ash content obtained at 3.5 L/min air flow could be due to experimental error. The ash content of clean coal

remained mostly constant at ~4.5% as wash water rate was increased, however, combustible recovery decreased from 70% to 30%.

The level of ash removal was comparable to the results reported previously with oil agglomeration, especially low pH oil agglomeration which selectively dropped the calcium content in the feed material. However, because there was no selective partitioning of the components in the ash from this column floated coal, there was little incentive to examine this material in a continuous run.

TASK 2.2 LABORATORY SUPPORT (CONSOL)

All CONSOL'S support activities for Run ALC-1 were completed on schedule. This included characterization of materials produced in Sandia's and HTI's hydrotreating and dewaxing work conducted in advance of Run ALC-1; on-site monitoring of Run ALC-1; characterization of the coal-agglomerates produced by CONSOL for Run ALC-1 and analysis the rejected solids and waters to generate elemental and material balances of the agglomeration process; review of HTI's preliminary results from Run ALC-1; and organization of the post-run meeting to discuss Runs ALC-1 and ALC-2.

Activities planned for next quarter include: generate elemental and material balances for the agglomerate production runs; analyze the Run ALC-1 separator overhead water samples to determine if there is unreported carbon, nitrogen and sulfur that may improve the elemental balances; characterize the wax produced in Run ALC-1; evaluate the solvent quality of the Run ALC-1 dewaxed and hydrotreated recycle oils; participate in planning Run ALC-2. The process oil samples collected during Run ALC-1 are being characterized by CONSOL under DOE Contract DE-AC22-94P93054.

TASK 3 CONTINUOUS OPERATIONS/PARAMETRIC STUDIES

TASK 3.3 (HYDROCARBON TECHNOLOGIES, INC.)

The first run ALC-1 was started in mid-April and ran for 28 days being completed on 13 May. Five conditions were run including a reference case, three with oil-agglomerated coal and one to evaluate solvent modification by dewaxing and hydrotreating. In the reference case, Wyodak coal from the Black Thunder mine was processed in an all-slurry mode with a catalyst comprising 1 wt% Fe, as a HTI's proprietary FeOOH/SO₄, and 100 ppm Mo, as Molyvan A. It was run in a recycle mode at a recycle solids/dry coal ratio of 0.20/1 and a coal concentration in slurry of 38%. In Conditions #2-#4, oil agglomerated coal was run at the same solids/dry coal ratio and a slightly lower space velocity. Exceptionally high coal conversions to THF solubles were observed in these conditions. In Condition #5, heavy distillate solvent was dewaxed and then hydrotreated to produce a more hydrogen rich solvent. A draft report for the run was prepared and circulated on 5 June to DOE and all the participants. A number of changes were suggested and a final ALC-1 report will be issued at a later date to be included in a subsequent quarterly report.

TASK 4 CONCEPTUAL PROCESS DESIGN

TASK 4.4 PRELIMINARY TECHNICAL ASSESSMENT (LDP ASSOCIATES)

Most of the activity for this reporting period was in support of ALC-1. Prior to the run a recommendation was made to conduct the pre-ALC-1 dewaxed oil hydrotreating at HTI at the lowest possible severity to minimize cracking of the 343 °C+ (650 °F+) material. This suggestion was based upon an analysis of the results from Sandia. Daily meetings were attended during the course of the run and the operations and results were monitored throughout the run period. Other program participants were kept up to date on activities during those periods when their representatives were not on-site. An elementally balanced material balance procedure was developed to calculate yields, hydrogen consumption, distillate boiling range yields and to estimate distillate product properties for the liquefaction section for the five run conditions. An estimated material balance was also prepared for the distillate solvent dewaxing and hydrotreating condition. A preliminary analysis of the run was provided at the ALC-1 Review Meeting.

SECTION ONE

**Center for Applied Energy Research
University of Kentucky**

WORK PERFORMED

TASK 2.1 LABORATORY SUPPORT (UK/CAER)

Task 2.1.1 Development of a standard catalyst screening test

The development of a catalyst screening test (CST) was discussed in the previous Quarterly Technical Progress Report DOE/PC/91040-64. In this test, catalyst precursors are evaluated for their liquefaction performance in a solvent comprising the solids-free components of a recycle solvent generated in Wilsonville Run 258, namely, ROSE SR V-130 deashed resid from period A and V-1074 heavy distillate from period B. The deashed resid has an elemental composition that is very nearly the same as the solids-free fraction of the recycle solvent, as shown in Table 1. The only significant difference is a slightly higher hydrogen content in the ROSE deashed resid which is about 0.4% higher than in the virgin CI solubles of the recycle solvent. This slight enrichment of H-containing materials in the deashed resid could be responsible for some change in relative resid activity in the two materials. The differences in the carbon and nitrogen compositions are relatively small.

Table 1. Elemental Composition of Deashed Resid and Total Recycle Solvent from Wilsonville Run 258A		
	Cresol Insolubles-free Recycle Solvent	V-130 Deashed Resid
Elemental Composition, solids-free basis, wt%		
Carbon	90.1	90.80
Hydrogen	6.47	6.86
Nitrogen	1.20	1.13
Sulfur	not reported	0.06
Oxygen	not reported	1.05
Ash	not reported	0.10
Source of elemental data	Table 4, DOE/PC/50041-130	Table 15, DOE/PC/50041-130

Since the amounts of distillate, deashed resid and dry coal in the CST are nearly the same as in Run 263J, the overall composition should approximate the feed stream used in the Wilsonville pilot plant except for the absence of the solids component. In this mixture the actual concentration of the dry BT Wyodak coal is 35 wt %, versus the 30 wt % used in the Wilsonville feed stream. Removing the solids from the reaction mixture should simplify the interpretation of the results since considerable

catalyst is contained in this fraction.

Activity of ALC-1 Catalyst in CST (B. Demirel)

The performance of the catalysts used in ALC-1 Condition #1, i.e. Molyvan A and HTI's proprietary FeOOH/SO_4 catalyst, was determined in the CST in order to have a comparison in our micro reactors between other catalysts we are developing and the HTI continuous bench-scale. In ALC-1 these catalysts were used at a make-up rate of 100 mg Mo/kg and 1.0 g Fe/100 g dry coal, respectively. Molyvan A is a molybdenum oxysulfide dithiocarbamate that is insoluble in petroleum oils and contains 29 w% Mo. The HTI FeOOH/SO_4 is a solid containing 10 w% Fe. Because it is proprietary to HTI, it was not otherwise analyzed or characterized.

This series of tests were run in a solvent comprising ROSE SR V-130 deashed resid from Wilsonville Run 258A and V-1074 heavy distillate from Run 258B, as described in the previous Quarterly Technical Progress Report DOE/PC/91040-64. The feed composition was:

HTI Black Thunder Coal, mf	35%
Heavy Distillate, R258B	25%
Deashed Resid, R258A	40%

The reaction conditions were:

Temperature	450°C
H ₂ Pressure	850 psig
H ₂ S (7.93%)/H ₂ Pressure	500 psig
Total Pressure	1350 psig
Time	30 minutes

The results from this series of runs are shown in Table 2. Experiments run in the absence of any added catalyst either with or without H₂S gave essentially the same result, indicating that H₂S does not effect resid or THF conversion in the absence of catalyst. With Molyvan A, H₂S had a very definite effect giving higher coal and resid conversion upon adding H₂S. In this series, the lower H₂S concentration gave a higher resid conversion although the THF conversions were essentially the same. It should be noted that the composition of molybdenum changes for each catalyst due to difficulties in weighing.

The HTI FeOOH/SO_4 catalyst when used alone gave lower activity than Molyvan A, both in the presence and absence of H₂S. However, conversions were much better in the presence of H₂S. Combining the Mo-Fe catalysts gave better performance, all in the presence of H₂S, especially at higher iron concentrations. The Fe concentrations were evaluated over a range of 0.2 to 0.8 w% on dry coal.

Because experiments in the microautoclaves are run in a closed system without any exchange of gas, the amount of water present with the HTI FeOOH/SO_4 catalyst was sufficient, when run under CST

Table 2. Activity of ALC-1 catalyst and PMA in CST

Run	Catalyst	Concentration, w% dry coal		Added Sulfur	Conversion, w%		
		Mo, ppm	Fe, g/100g	H ₂ S or S	Coal	Coal +IOM +Resid	Coal+IOM+ Resid (daf)
B6-142-1	None	-	-	None	65.7	13.2	28.5
B6-149-1		-	-	H ₂ S	66.3	13.3	28.1
B6-199-1	Molyvan A	295	-	None	76.7	16.6	35.1
B6-192-1		300	-	1/2H ₂ S	90.5	24.3	51.5
B6-128-1		330	-	H ₂ S	88.2	20.2	42.9
B6-136-1	HTI Fe ¹	-	0.880	None	64.7	14.4	30.4
B6-142-2		-	0.715	H ₂ S	78.1	19.2	40.6
B6-205-1	Molyvan A+HTI Fe ¹	250	0.200	H ₂ S	91.6	28.5	60.4
B6-200-1		250	0.470	H ₂ S	91.7	28.4	60.1
B6-137-1		365	0.775	H ₂ S	92.2	30.3	65.5
B6-179-1	Fe #1 ²	-	0.695	none	74.7	19.2	40.6
B6-177-1		-	0.705	H ₂ S	80.4	23.0	49.0
B6-185-1		-	0.730	S	75.5	14.2	30.1
B6-179-2	Fe #2 ³	-	0.705	none	73.6	14.3	30.2
B6-170-1		-	0.710	H ₂ S	82.0	23.3	49.3
B6-191-1		-	0.695	S	74.6	16.3	34.4
B6-149-3	PMA ⁴	325	-	H ₂ S	80.7	20.1	42.7
B6-163-1	PMA (I on C w/ excess W) ⁵	300	-	H ₂ S	90.0	30.5	64.6
B6-207-1	PMA (I on C w/ IWW) ⁶	300	-	H ₂ S	91.6	23.9	50.6
B6-166-1	PMA (I on 10% C w/ excess W) ⁷	340	-	H ₂ S	90.8	33.3	70.4
B6-197-1	PMA (I on 10% C w/ IWW) ⁸	300	-	H ₂ S	88.2	24.0	50.8
B6-197-2	PMA (I on 10% C w/ IWW) ⁸ +Fe ¹	300	0.700	H ₂ S	90.8	24.7	52.4

(1) HTI FeOOH/SO₄ catalyst

(2) HTI Fe catalyst calcined 490 °C/3 h; humidified in a desiccator over water

(3) HTI Fe catalyst dried 96 °C/0.3 atm; humidified in a desiccator over water

(4) Phosphomolybdic acid added directly.

(5) Phosphomolybdic acid impregnated on coal with excess water; dried at 96 °C and 0.3 atm overnight.

(6) Phosphomolybdic acid impregnated on coal with 0.45 g water/g dry coal and dried to 3.93% moisture prior.

(7) Phosphomolybdic acid impregnated on 10% coal with excess water and dried at 96 °C and 0.3 atm overnight.

(8) Phosphomolybdic acid impregnated on 10% coal with 0.45 g water/g dry coal (not dried prior to use).

condition, to exceed the upper pressure limit on the reactors. These microreactors are equipped with rupture discs rated at ~2700 psi at 21 °C. Since the CST operates typically at pressures of about 2500 psi, any significant excess water over and above the amount in the normal charge stock can result in rupture of the relief valve. In order to guard against this, careful control of the water content in the feed is necessary to prevent exceeding the maximum allowable pressure with the existing relief system. Fe #1 catalyst was prepared by calcining of HTI Fe catalyst at 490°C for 3 h and humidifying in a desiccator over water. Fe #2 catalyst was prepared by first drying at 95°C and 250 torr overnight and then humidified over water in a desiccator. In the presence of H₂S, both catalysts were more active than the FeOOH/SO₄ with the water still present. In the continuous run, unlike the microautoclave, much of the water is removed in the slurry feed tank which results in a relatively dry reaction environment in the reactor.

Because of the interest in possibly using elemental S as a sulfiding agent, a few runs were made with these two dried Fe catalysts. The activities of both Fe #1 and Fe #2 were lower in the presence of elemental sulfur than when H₂S was present. Upon the addition of elemental sulfur, resid conversion decreased to 30-34% (daf) whereas it was about 49% with H₂S.

Task 2.1.2 Activation of impregnated metal catalysts (R. K. Anderson)

Studies continued on the pretreatment of incipient wetness (IW) impregnated coals. In the previous quarterly report, we presented data on the Ni/Mo and Fe/Mo impregnated coals, using 30 minute pretreatments at temperatures from 275 °C to 440 °C, immediately followed by 30 minutes reaction time at 440 °C in coal derived liquids from Wilsonville run 258A. Here, data are shown for Mo-only impregnated coals (See Table 3), using ammonium molybdate as the starting salt and as-received Black Thunder coal. Again, the entire coal was impregnated (vs the vector approach). These experiments required three coal preparations to complete, and covered batches CB-51, -58 and -59. Each carried about 500 ppmw Mo (mf coal basis), and all contained about 6-8% more moisture than the starting coal.

Figure 1 summarizes both resid and THF coal conversion for each of the impregnated coals tested. As was seen in Phase I, the Mo-only impregnated coals gave better resid conversion than the Fe/Mo impregnated coal. Overall however, Ni/Mo gives the best results with enhanced coal dissolution.

Task 2.1.3 Optimizing Metal Concentration in the Catalyst

In an effort to determine what an upper conversion limit would be for the conditions used here, and unlimited by catalyst availability, an experiment was run using crushed AKZO AO-60 at 10 wt% Mo+Ni (mf coal basis), while holding other feedstocks the same as in the pretreatment studies. Reaction conditions were 440 °C for 30 minutes, without any pretreatment period. A check of the product gas composition shows almost 2% H₂S, so that there was sufficient available to sulfide the catalyst. As shown in Table 4, 524 °C+ (975 °F+) resid conversion was found to be 39.7%, and coal conversion (THF) was 95.8%. When viewed in the context of the other experimental work [no

catalyst, 17-19% resid conversion; and with 500 ppmw Mo, ~30 % resid conversion], this result suggests that the economic employment of catalyst in this system would probably be in the range up to 1000 ppmw. Figure 2 presents this information graphically, summarizing selected runs using Mo and Ni/Mo catalysts without pretreatment, and shows the catalyst concentration on the axis using a logarithmic scale.

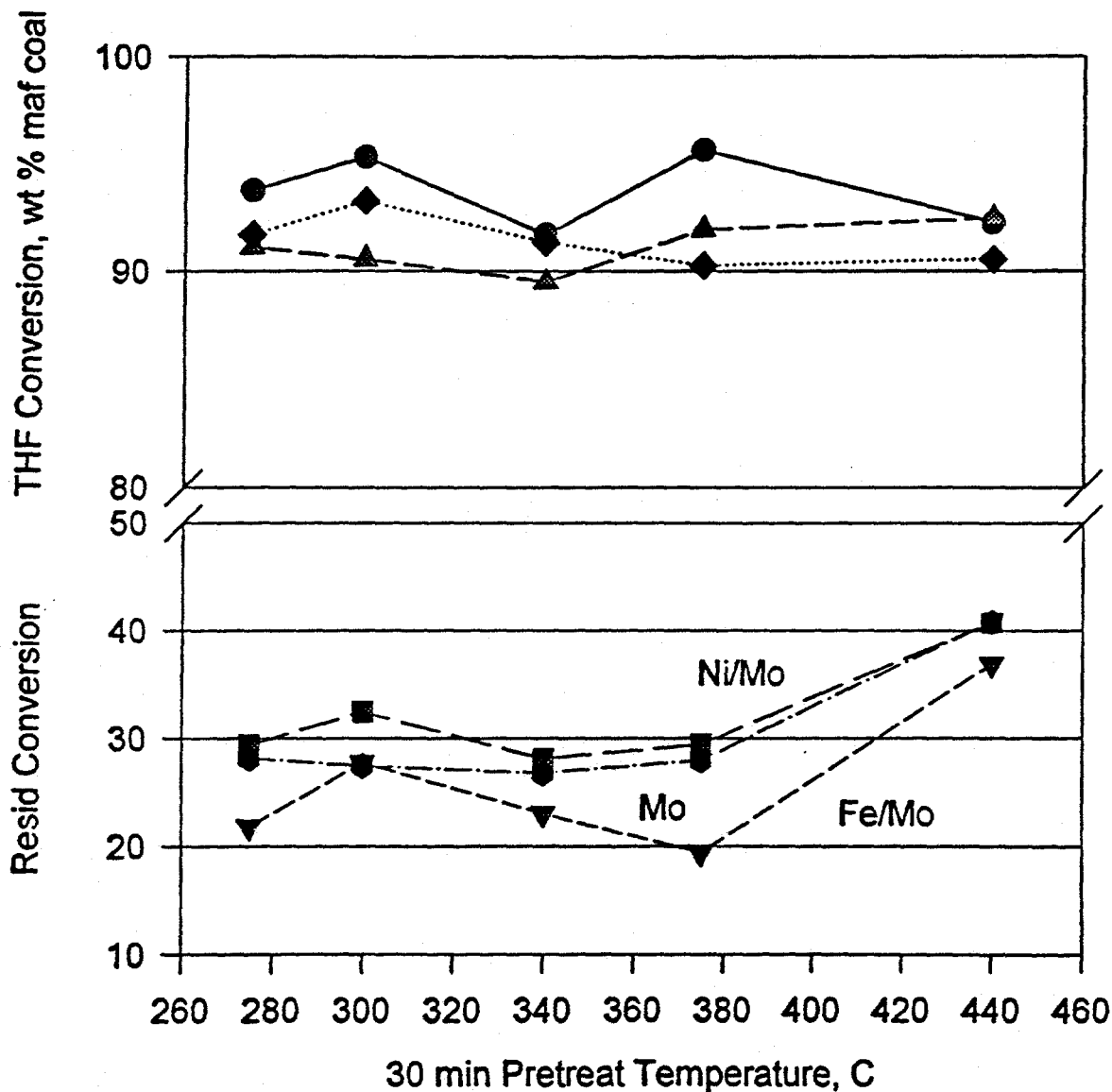
Table 3. Results of 30 minute liquefaction experiments.*						
Catalyst preparation	CB-58	CB-58	CB-59	CB-58	CB-58	CB-51
Mo added, ppmw	504	504	482	504	504	552
Pretreat time, min @ °C	none	30 @ 275	30 @ 300	30 @ 340	30 @ 375	30 @ 440
Products, wt% maf Coal						
HC Gases	14.0	14.6	13.6	14.1	13.2	19.7
CO+CO ₂	11.6	12.2	11.5	11.1	11.0	11.0
524 °C- (975 °F-)	38.3	39.5	39.5	37.9	41.7	64.8
524 °C+ (975 °F+)	36.1	33.7	35.4	36.9	34.1	4.5
Total	100.0	100.0	100.0	100.0	100.0	100.0
THF Conv	90.4	91.7	93.3	91.4	90.3	90.6
Resid Conv, wt% maf resid	27.1	28.1	27.4	26.8	28.0	40.8
Material Bal index	102	104	100	104	96	97
H ₂ consumed, mg/g maf Coal	50	56	51	57	61	70
Coal moisture, wt %	31	31	31	31	31	30
Run No.	R6-128-1	R6-136-2	R6-141-1	R6-130-1	R6-130-2	R5-346-1
a. Liquefaction experiments at 440 °C for 30 minutes, using 3 g Black Thunder coal, 2.90 g V-130 deashed resid and 1.43 g V-1074 heavy distillate from Wilsonville Run 258A. 1350 psig cold charge pressure, including 3% H ₂ S. SO ₃ -free ash basis.						

Table 4 shows other work designed to test the effect of added Ni, above the level selected for the earlier studies. For those experiments an impregnated coal, CB-60, was prepared which carried nominally 500 ppm Mo and 500 ppm Ni. Duplicate experiments (R6-144-2 and R6-151-1) were run without pretreatment, and a single run with 30 min pretreat at 300 °C was also checked (R6-169-2). These are shown in Table 4 in comparison to the other coal liquefaction experiments where 500 ppm

Figure 1.

Ni/Mo, Mo and Fe/Mo IW Impregnated Coal

Effect of Pretreatment Temperature



3% H₂S, 1350 psig total pressure, cold

Ni/Mo coal: 500 ppm IW Mo plus 100 ppm IW Ni

Fe/Mo coal: .67% IW Fe plus 500 ppm IW Mo

Mo coal: 500 ppm IW Mo

30 min hydrotreatment at 440 C, 60 min total run

- % THF Conv, Ni/Mo coal
- % Rc, Ni/Mo coal
- ▲ % THF Conv, Fe/Mo coal
- ▼ % Rc, Fe/Mo coal
- ◆ % THF Conv, Mo coal
- % Rc, Mo coal

Figure 2.

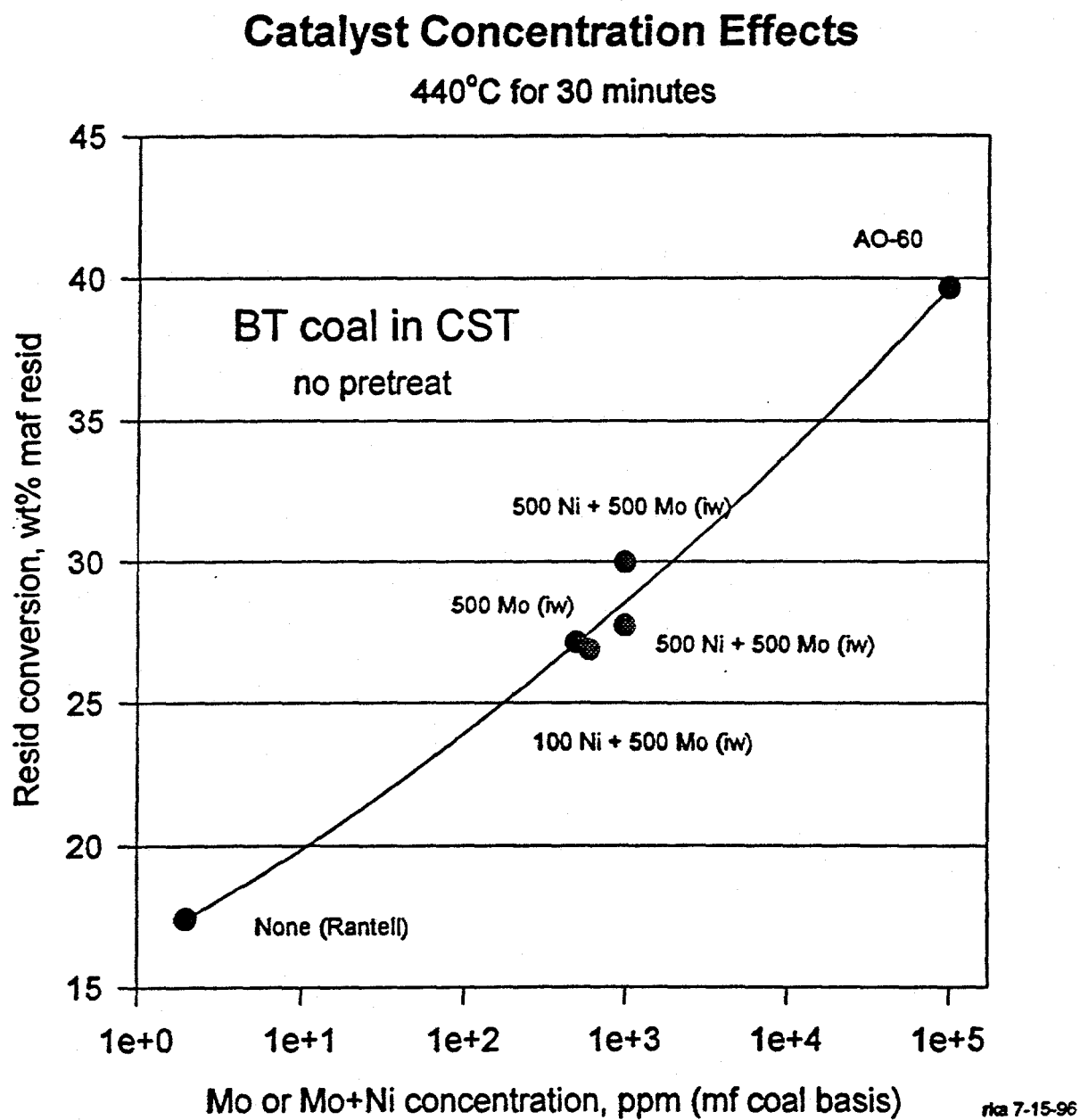


Table 4. Results of 30 minute liquefaction experiments. ^a						
Catalyst preparation	None	CB-52	CB-60	CB-60	CB-52	CB-60
Mo/Ni added, ppmw (mf coal)	85,600/ 18,200	538/101	528/528	528/528	538/101	528/528
Pretreat time at °C, mins	none	none	none	none	30 @ 300	30 @ 300
Products, wt% maf Coal						
HC Gases	14.2	12.0	12.4	13.5	14.3	14.6
CO+CO ₂	8.8	11.4	10.9	11.6	11.8	11
524 °C-	65.5	35.4	39.1	42.4	44.9	36.2
524 °C+	11.5	41.2	37.6	32.5	29	38.2
Total	100.0	100.0	100.0	100.0	100.0	100.0
THF Conv	95.8	90	93.8	92.9	95.3	89.5
Resid Conv, wt% maf resid	39.7	26.9	27.7	30	32.4	27.5
Material Bal index	95	98	99	99	100	106
H ₂ consumed, mg/g maf Coal	73	54	54	55	61	62
Coal moisture, wt %	24	21	25	25	21	25
Run No.	R6-144-1	R6-26-1	R6-144-2	R6-151-1	R6-66-1	R6-169-2
a. Liquefaction experiments at 440 °C for 30 minutes, using 3 g Black Thunder coal, 2.90 g V-130 deashed resid and 1.43 g V-1074 heavy distillate from Wilsonville Run 258A. 1350 psig cold charge pressure, including 3% H ₂ S. SO ₂ -free ash basis.						

Mo and only 100 ppm Ni were used. As can be seen from that data, without pretreatment there is a nominal increase in both 524 °C+ (975 °F+) resid conversion and THF conversion. However, this increase is only to levels seen earlier with the pretreatment experiments using coals impregnated at the lower 100 ppm Ni level, and one wonders if the increase in resid conversion found here is only due to improved Ni dispersion.

With the 30 min pretreatment, a slight *decrease* in conversion is seen, but hydrogen consumption was found to be the same. In total, the data collected to date suggests that most of the improvements in conversion from added Ni can be accrued at low concentrations.

The effect of lower H₂S partial pressure was also tested using a Ni/Mo impregnated coal (CB-61) in 30 min liquefaction at 440 °C. The outcome was similar to run R6-26-1 shown in Table 4, but showing slightly lower resid conversion. Since conclusive evidence would be needed to support a run condition change for ALC-2, this work will be duplicated in the next period.

Resid Concentration in Ashy Resid - A sample of the ashy resid from Wilsonville Run 262E was analyzed by SIMDIS and was found to be ca 16% 565 °C- (1050 °F-). Since this was somewhat higher than the 2.5% value used to determine resid conversion in earlier studies, a second sample was submitted, combining heavy distillate and ashy resid to see if there were some interactive effects when this mixture was distilled together. This was found to be 51% 565 °C- (1050 °F-) (inferring an ashy resid distillate content of about 13%), vs the value used earlier of 45%, as reported by Southern Electric. If the newer numbers are more accurate, it would lower resid conversion about 4% or 5%.

It was also observed, working with SIMDIS generated data, that resid conversion is pretty sensitive to the cut point temperature selected. For example, increasing the cut point from 524 °C (975 °F) to 565 °C (1050 °F) increases resid conversion in a typical experiment by about 8%, even though the amount of resid in the feedstock is reduced.

Laboratory analysis - To verify the expected low background concentration, a sample of the DAR was submitted to an outside lab for Mo determination, and was found to contain <10 ppmw Mo.

Phosphomolybdic acid as catalyst precursor (B. Demirel)

The activity of phosphomolybdic acid ($\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O}$) was determined in several different runs. When added as a crystalline material to the reaction mixture, its activity was quite low. However, if impregnated onto coal, its activity improved significantly. Impregnated coals were prepared by two different methods. In one case, all of the coal was impregnated with the PMA using the incipient wetness volume of water. In the other cases, the PMA was impregnated onto 10% of coal and then blended with the remaining coal, the vector approach. The vector impregnated coal using excess water gave higher resid conversion in two different cases than the other coals in which an incipient wetness volume of water was used. The excess water may have resulted in better dispersion during impregnation.

Ammonium Tetrathiomolybdate as Catalyst Precursor (H. Von Woert)

Liquefaction catalysis using ammonium tetrathiomolybdate (ATTM) as a precursor was studied during this reporting period. A fresh sample of ATTM was prepared starting with ammonium paramolybdate. A corresponding sample obtained from Aldrich Chemical was considerably different suggesting that the commercially available material had decomposed upon standing. Subsequently, a sample of fresh ATTM was also observed to become less soluble after standing about 2 months. Therefore, it will be necessary to prepare fresh material if this material will be tested in a continuous run.

Impregnated coal samples were prepared by adding a 1% aqueous solution of ATTM to obtain a Mo concentration of 300 mg/g dry coal. Both as-received as well as dry coal samples were prepared. At this level of moisture addition, as-received impregnated coal contained 15.1% moisture. A coal sample that had been previously dried overnight at 100 °C under vacuum contained 9.9% moisture after impregnating to a Mo level of 300. Of course, when impregnating with only 100 ppm Mo, as will be done when testing in the continuous bench-scale unit, the impregnated as-received coal samples will contain approximately 11% moisture.

Each catalyst impregnated coal was evaluated in the CST starting with deashed resid and distillate from Wilsonville Run 258. The results of these runs are shown in Table 5.

Liquefaction of both as-received and dry coal impregnated with ATTM in the presence of H_2S gave nearly the same 524 °C+ (975 °F+) resid conversions of 24.9 and 28.4%. On a maf coal basis, the resid conversions were 50.9 and 60.5%, respectively. The corresponding coal conversions were 84 and 88% in the presence of H_2S . The yield of gases were slightly higher for the dry coal run. Therefore, there is little difference between using as-received and dry coal as feed.

Table 5. Wyodak Coal Liquefaction with Ammonium Tetrathiomolybdate Precursors Conditions: 440 °C, 1000 psig (cold), 2% H ₂ S in H ₂ , 0.5 hrs.					
Coal	As-received	Dry coal	As-received	Dry coal	As-received
Moisture in coal, wt% ^a	8.9	7.5	16.4	7.5	15.1
Mo conc, mg/kg dry coal	none	350	370	350	370
H ₂ S, wt% dry coal	7.3	4.3	4.3	0	0
HC gases, wt% maf coal	ND	10.0	9.6	9.6	9.7
Co _x gases, wt% maf coal	ND	8.5	7.9	9.3	8.7
524 °C+ Resid Conv, wt% maf IOM+resid	not determined	24.9	28.4	24.0	17.6
524 °C+ Resid Conv, wt% maf coal	not determined	50.9	60.5	56.1	37.5
THF IOM, wt% maf coal	48.4	15.6	11.6	34.1	34.2
THF Conversion	51.6	84.4	88.4	65.9	65.8
Run No.	V6-101-1	V-111-1	VSMSR-1 V6-206-1 V6-206-2	V6-185-2	V6-185-1 V6-213-1
a. Includes moisture used in impregnating coal with ATTM.					

Because sulfur was already present in the precursor, the runs were made in the absence of H₂S to determine whether H₂S had an effect on product distribution. Gas yields and 524 °C+ resid conversions were essentially the same. Surprisingly, the yield of IOM products were considerably higher in the absence of H₂S. This could be the result of decreased conversion of the original coal macerals or it could indicate coking of the heavier products back to insoluble matter, i.e. a retrogressive type reaction. At any rate, the H₂S had a very definite effect on THF soluble yield. To check this result a new batch of impregnated coal was prepared and is now being run.

TASK 2.1.5 Coal Cleaning by Column Flotation

Beneficiation Studies of Black Thunder Coal (B. K. Parekh)

Introduction: An exploratory experimental study was performed at the University of Kentucky Center for Applied Energy Research to determine the possibility of producing a low ash Black Thunder coal using froth flotation technique. Flotation tests were performed on a fine ground (80% passing 325 mesh) coal using the Denver and 'Ken-Flote' column techniques.

It was observed that despite Black Thunder coal being classified a sub-bituminous coal, it responded favorably to froth flotation technique. High recovery of combustibles were obtained at a low or moderate ash rejection, suggesting that a majority of ash may be present as 'inherent' ash. Both conventional and column flotation provided a 3.4% ash clean coal at 75% combustible recovery at a ash rejection of about 65%.

Objectives: The main objective of the study was to develop optimum column flotation operating conditions for cleaning of Black Thunder coal to obtain a 2% ash clean coal at 90% combustible recovery.

Results: A one 55-gallon drum of the Black Thunder coal of approximately 2-in. top size was received at CAER. After crushing the coal to minus 1/4 in. a representative sample of the crushed coal was analyzed for proximate analysis. Table 6 lists the analysis of the 'as received' coal. Note, that the coal had about 27% moisture and 7.01% ash (on dry basis).

Table 6. Proximate Analysis of As-Received Black Thunder Coal.		
	Weight %	Weight %, dry basis
Moisture	26.92	-
Ash	5.12	7.01
Sulfur	0.37	0.51
Pyritic	0.05	0.07
Sulfate	0.06	0.06
Organic	0.26	0.36
Volatile Matter	32.54	44.53
Fixed Carbon	35.4	48.4
Heating Value	8900 Btu/lb	12192 Btu/lb (db)

Grinding studies of the crushed coal were conducted in the Attritor Mill, which is efficient in fine grinding of coal. For grinding studies a 50% suspension of the coal in water was utilized. Table 7 lists the particle size of the coal obtained at various grind times. Note, that the median particle size of the ground coal remains unchanged after 15 minutes of grinding. The average particle size of 10 minutes grind was about 11 μ m. Figure 3 shows the particle size distribution of the 10 minutes ground coal. All the flotation tests reported were conducted with the 10 minutes ground coal.

Froth Flotation: Initial froth flotation tests were conducted using the laboratory Denver

flotation machine. Table 8 lists the flotation data for the coal. Note, that the lowest ash content of 3.9% and combustible recovery of 52.5% was obtained using 2 lb/t of fuel oil and MIBC. The data also showed that 0.5 lb/t of reagent dosage was not sufficient to clean the coal.

A release analysis of the fine ground coal was conducted using 2 lb/t of reagents. Release analysis is similar to float sink analysis of coarse coal. The release analysis provides a guideline on practical limit of recovery and ash content of clean coal that could be obtained using the conventional froth flotation technique. Figure 4 shows the release analysis data of the fine ground Black Thunder coal. According to the figure, 3.7% ash clean coal could be obtained at 80% combustible recovery. A 3% ash clean coal could be obtained at only 30% combustible recovery.

Table 9 lists flotation tests conducted using various types of operating conditions and reagents. Note, that acid scrubbing at 30% solids concentration (Test 1) and floating using 5% solids suspension gave higher combustible recovery compared to scrubbing conducted using lower 5% solids. Addition of sodium metaphosphate, a dispersant, did not provide any improvement in clean coal ash content or combustible recovery.

Column Flotation Studies: Column flotation studies were conducted using a 2-in. I.D., 20-ft. high column. Figure 5 shows the effect of air flow rate on combustible recovery and ash content of clean coal. As expected, both the ash content and combustible recovery increasing with increase in air flow rate. The lower ash content at 3.5 L/min. air flow could be due to some experimental error.

Effect of wash water on column flotation performance is shown in Figure 6. Note, that the ash content of clean coal remained mostly constant at ~4.5% as wash water rate is increased, however, combustible recovery decreased from 70% to 30%.

Effect of feed rate on the column flotation performance is shown in Figure 7. It shows that the ash content increased from 3.2 to 3.7% and combustible recovery decreased from 75 to 45% with increase in feed rate from 0.5 to 1 liter/minute.

Conclusions: Based on the above-cited data, it can be concluded that

- conventional flotation of the Black Thunder coal ground to an average particle size of 11 μm , provided a 3.5% ash clean coal at about 70% combustible recovery using acid scrubbing step. In other words, conventional flotation provided about 65% ash rejection from the feed coal.
- column flotation of the Black Thunder coal, using a feed rate of 0.5 liter/minute, wash water rate of 0.2 liter/minute, provided the best result, i.e., 3.4% ash clean coal at about 75% combustible recovery. The results obtained using conventional and column flotation technique were very similar which could be due to very low ash content of the feed. Column is effective in rejecting ash, however, when the ash content is low, the column flotation will not show any improvement over the conventional flotation.

Table 7. Particle Size of the Fine Coal Obtained at Various Grind Time					
	Grind Time (minutes)				
	5	10	15	20	30
Medium Size (μm)	13.60	11.55	6.47	5.67	4.56
Diameter of 10% passing (μm)	1.77	1.29	1.06	1.02	0.93
Diameter of 90% passing (μm)	41.52	40.09	18.21	17.09	15.25

Table 8. Effect of Reagent Dosages on Ash Content of Clean Coal and Combustible Recovery for the Black Thunder Coal			
Reagent Dosage (lb/t)		% Ash	% Combustible Recovery
Fuel Oil	MIBC		
0.5	0.5	5.0	81.9
1.0	1.0	4.1	26.9
2.0	2.0	3.9	52.5
4.0	4.0	3.9	52.5

Table 9. Flotation Test Data Obtained Using Various Reagents and Process Conditions			
Test No	Process Conditions	% Ash	% Combustible Recovery
1	Acid scrubbing at pH 2.0, at 30% solids, flotation at 5% solids	3.4	72.63
2	Acid scrubbing at pH 2.0 using 5% solids	3.4	58.41
3	7 lb/t of sodium metaphosphate	4.2	53.7

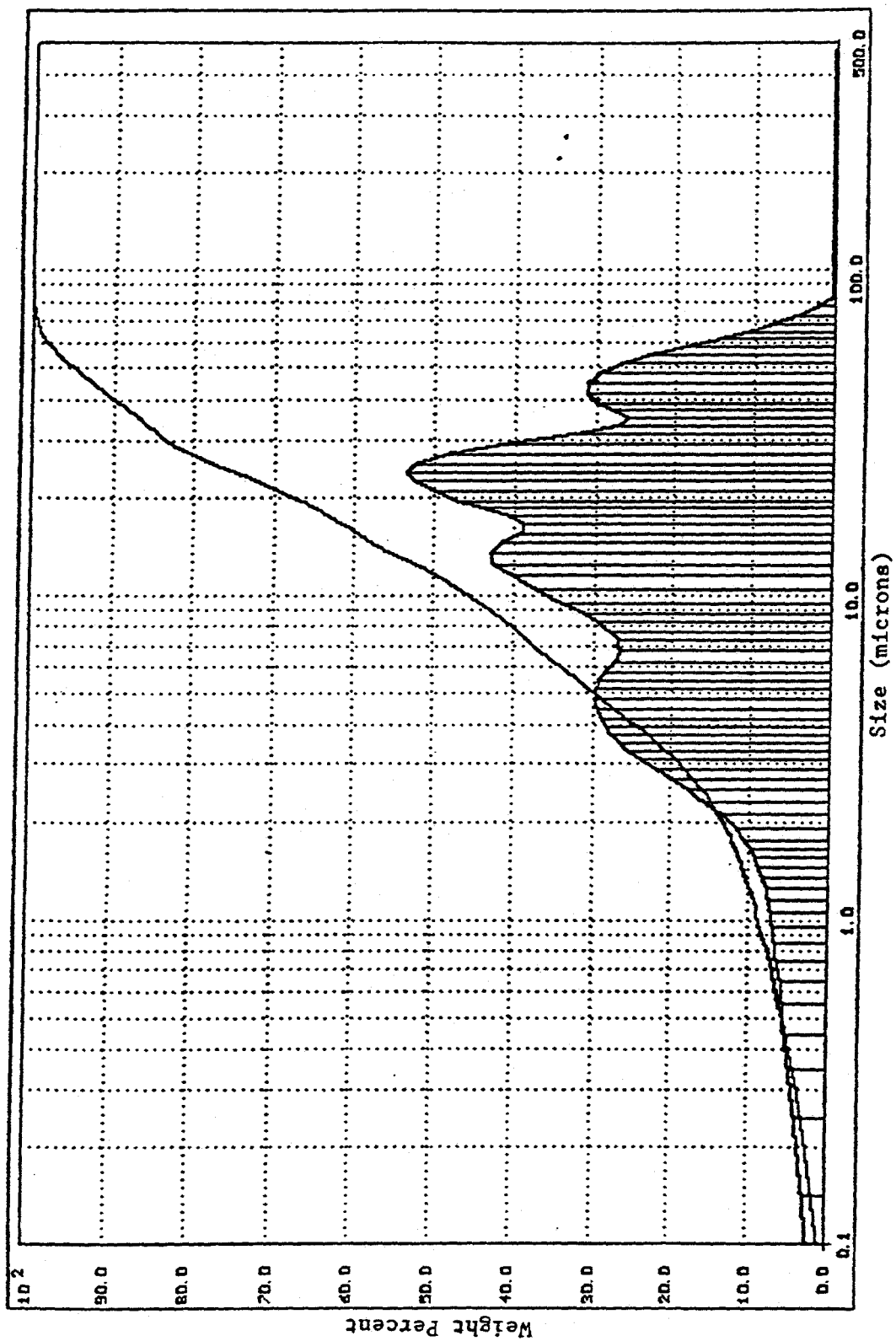


Figure 3. Particle size distribution of 10 minutes ground coal

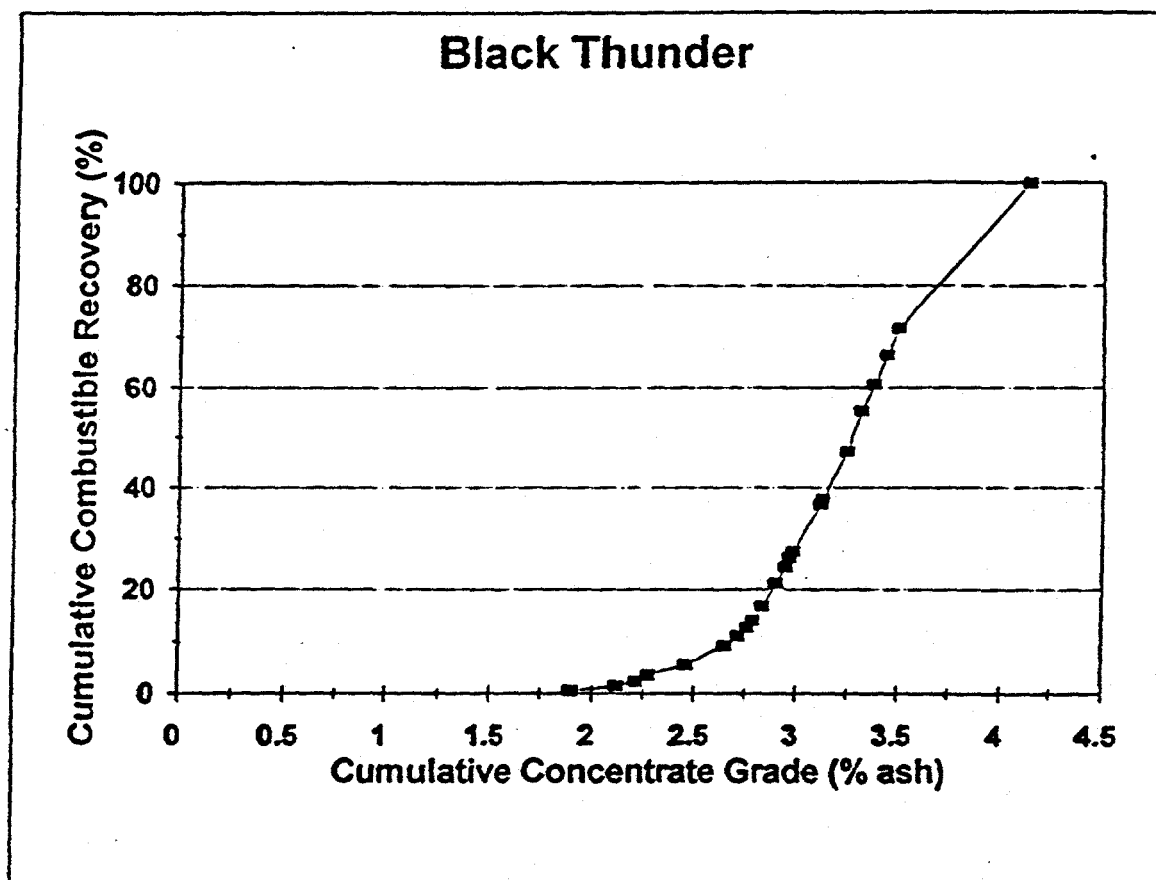


Figure 4. Release analysis of the 10 minutes ground coal.

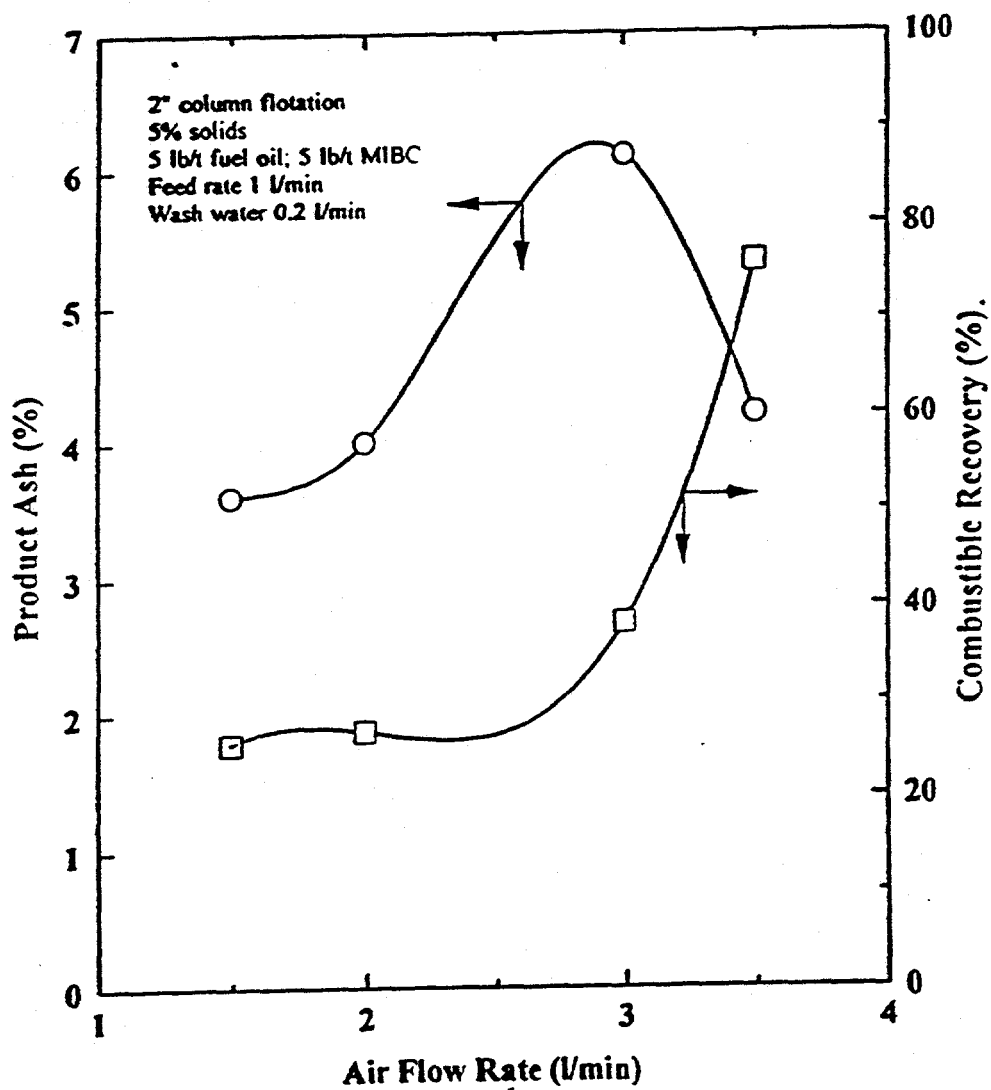


Figure 5. Effect of air flow rate on combustible recovery and product ash content of Black Thunder coal.

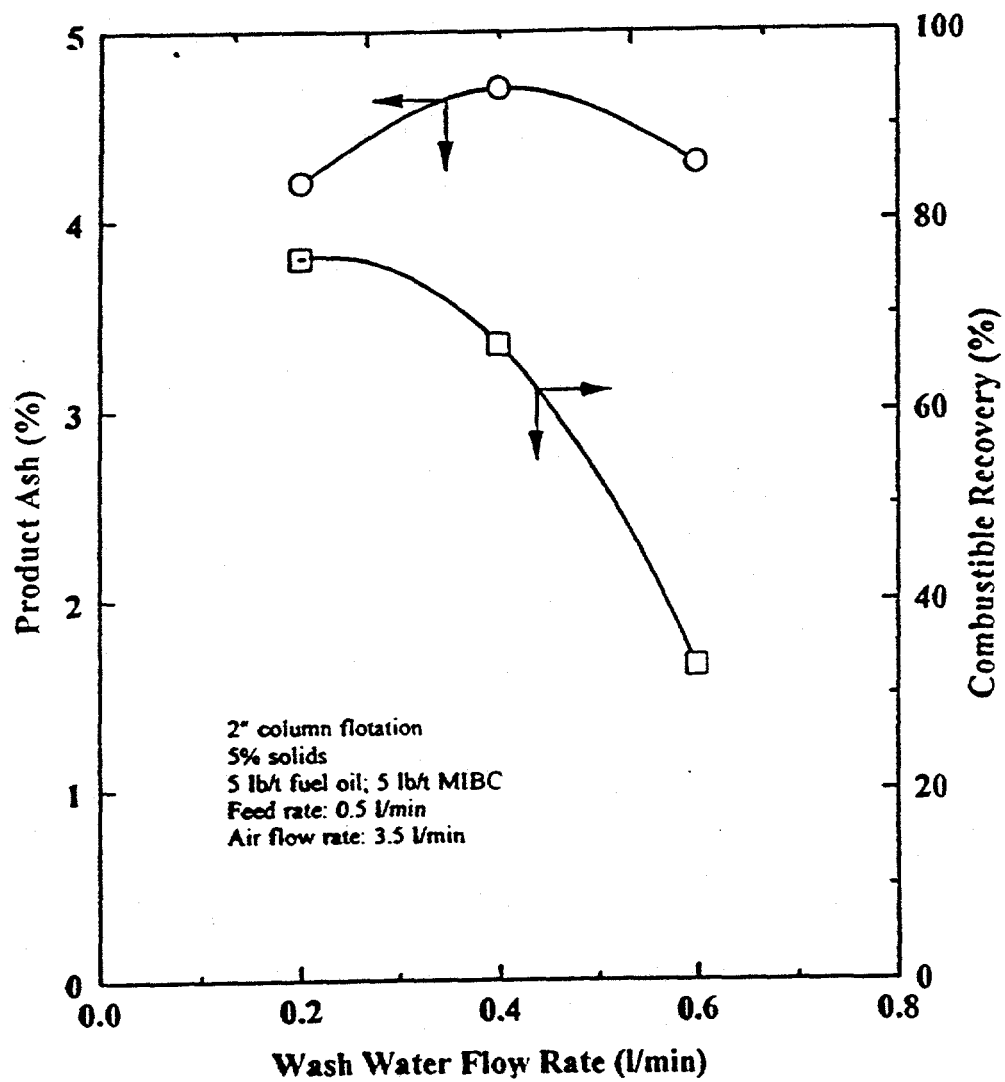


Figure 6. Effect of wash water rate on product ash and combustible recovery of Black Thunder coal.

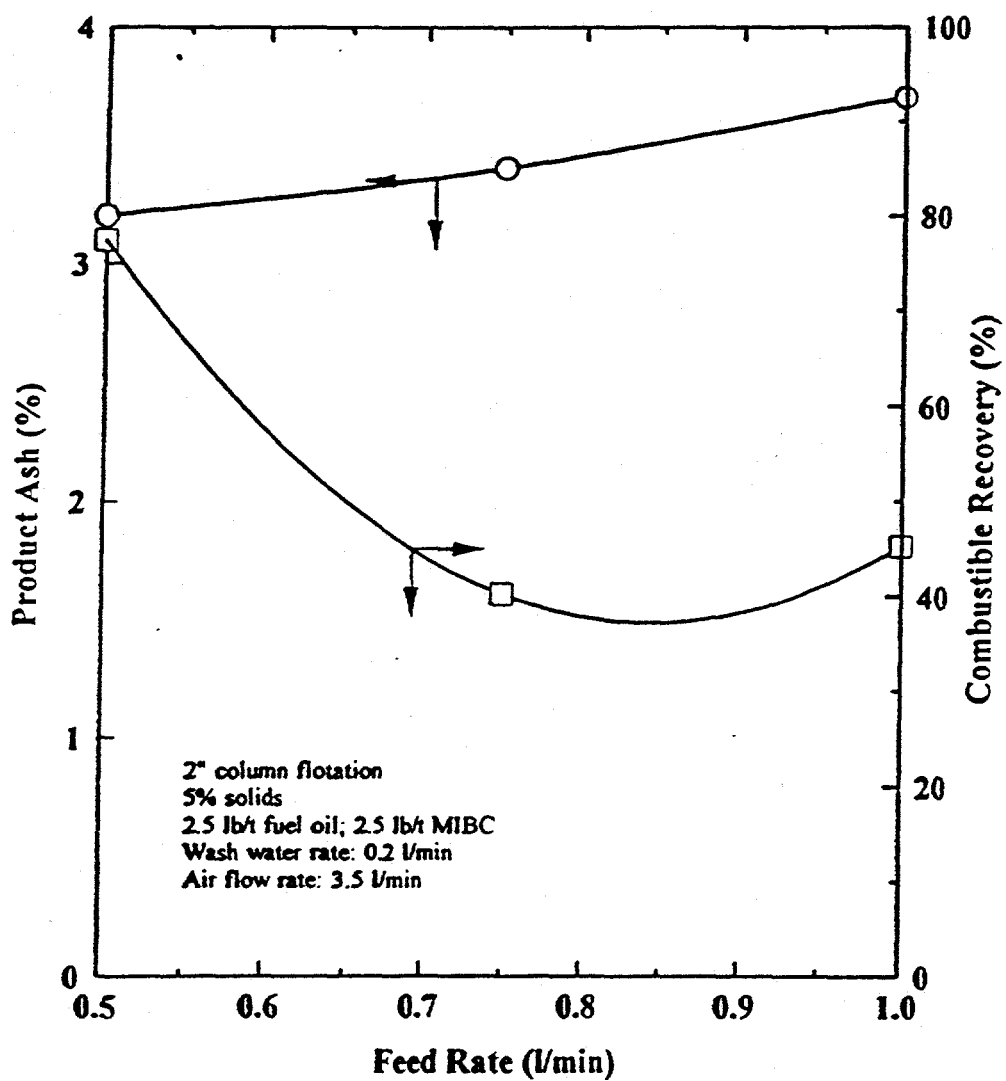


Figure 7. Effect of feed flow rate on column flotation performance for the Black Thunder coal (air flow rate 3.5 l/min.)

SECTION TWO

CONSOL, INC.

January 16, 1997
Report Period: April 1 - June 30, 1996

SUBCONTRACT TITLE AND NUMBER:

Subcontract UKRF-4-25582-92-76 to CONSOL Inc.
Under DOE Contract No. DE-AC22-91PC91040, "Advanced Coal Liquefaction Concepts for the PETC Generic Bench-Scale Unit"

SUBCONTRACTOR NAME:

CONSOL Inc.
Research & Development
4000 Brownsville Road
Library, PA 15129

SUBCONTRACT PERIOD: November 26, 1991 - September 30, 1996

PRINCIPAL INVESTIGATORS: F. P. Burke, R. A. Winschel, G. A. Robbins

SUBCONTRACT OBJECTIVES: No change.

SUMMARY OF TECHNICAL PROGRESS - OVERALL

All CONSOL's support activities for Run ALC-1 were completed on schedule. This included characterization of materials produced in Sandia's and HTI's hydrotreating and dewaxing work conducted in advance of Run ALC-1; on-site monitoring of Run ALC-1; characterization of the coal-agglomerates produced by CONSOL for Run ALC-1 and analysis of the rejected solids and waters to generate elemental and material balances of the agglomeration process; review of HTI's preliminary results from Run ALC-1; and organization of the post-run meeting to discuss Runs ALC-1 and ALC-2.

Activities planned for next quarter include: generate elemental and material balances for the agglomerate production runs; analyze the Run ALC-1 separator overhead water samples to determine if there is unreported carbon, nitrogen and sulfur that may improve the elemental balances; characterize the wax produced in Run ALC-1; evaluate the solvent quality of the Run ALC-1 dewaxed and hydrotreated recycle oils; participate in planning Run ALC-2. The process oil samples collected during Run ALC-1 are being characterized by CONSOL under DOE Contract DE-AC22-94PC93054.

SUMMARY OF TECHNICAL PROGRESS - BY TASK

Task 2 - Laboratory Support

Laboratory Coal/Oil Agglomeration Testing Completed in Preparation for Run ALC-1. The laboratory agglomeration tests were completed to prepare for the large-scale production agglomeration runs. The base-line agglomerates from laboratory-scale test A75 were analyzed (Table 1). The ash elemental analysis shows that calcium, magnesium, sodium, and the resulting ash SO_3 are selectively rejected from the ash of the agglomerates (compare with ash elemental analysis of the feed coal, Table 9).

The elemental analysis of the dried (but not ashed) filter cake from laboratory-scale agglomeration test A78 indicates that the filter cakes of these low-pH agglomerations tests with Black Thunder coal consist principally of gypsum. The analysis (Table 2) is consistent with material that is 83 wt% gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), 17 wt% silica, and less than 1 wt% of anything else. It was also determined that the ashed filter cake from Run A79 contained 19.3% sulfur; this is consistent with material that is 82% CaSO_4 .

Characterization of Sandia Hydrotreating Samples Produced in Preparation of Run ALC-1. Samples of the feedstock and eight products from Sandia's hydrotreating tests with Criterion C-411 catalyst were characterized. Sandia conducted these tests to help plan solvent hydrotreating conditions for Run ALC-1. Table 3 lists Sandia's operating conditions and simulated distillation data of the products. Sandia provided CONSOL with partially-full 20 mL vials of the various samples for characterization. CONSOL's elemental analyses are shown in Table 4. CONSOL's $^1\text{H-NMR}$ proton distributions and microautodave solvent quality assay data are shown in Table 5. Key observations are:

- The feedstock has an unexpectedly high S content for a coal-derived HTI VSOH.
- Five of the eight products had a strong ammonia odor. Some of the product nitrogen is probably dissolved ammonia.
- The products appear to belong to two groups on the basis of elemental analysis and proton distributions: the earliest five and the latter three. The latter three are generally less hydrogenated. For example, even though samples 100-35-96C and 100-313-96B were

produced at ostensibly similar conditions, the latter has more N and S and less H and is more aromatic. This may reflect catalyst age.

- The first five products were made at the high hydrogen treat rate. The primary effects of increasing the temperature were to decrease S and N contents and decrease the boiling point distribution of the product. There was not a substantial effect on H content or proton distribution of the product. At higher temperatures, there was greater heteroatom removal and cracking, and about the same level of liquid product hydrogenation. The similar H contents but lower boiling point distributions of the products made at high temperatures may indicate that there is lower H content in each given narrow boiling point fraction of the high-temperature products.
- For the last three products (made at 365 °C, 689 °F), decreasing the hydrogen treat rate increased the S and N contents and the aromaticity and boiling point distribution of the products. There was less hydrogenation, and apparently less cracking, at the lower treat rates. In the absence of gas/liquid recovery data, the cause of the high-boiling tail in the two products with the lowest treat rates is unclear. It could be caused by loss of light products, polymerization, sample contamination, or analytical error
- Solvent quality assays at a single set of conditions on the feedstock and one product in each of the two groups show that the products are better hydrogen donor solvents than the feedstock.

We recommended to HTI that Run ALC-1 VSOH hydrotreater operating conditions be selected to promote hydrogenation, but not cracking. Neither cracking nor heteroatom removal are important functions for this step. We recommended that HTI operate their solvent hydrotreater at 365 °C (689 °F) or lower and at high H₂ treat rate. We discussed our Run ALC-1 hydrotreating recommendations with LDP and HTI. On the basis of prior experience, HTI expected their originally planned conditions ((379 °C)715 °F, about 2.4 WHSV, 15.2 MPa (2200 psig) H₂, and treat rate about 1.07 std m³/L (6000 SCF/bbl)) to produce only about 10% 343 °C (650 °F) material from the VSOH. However, they agreed to lower the temperature to 371 °C (700 °F), to further reduce the possibility of over-hydrogenation or cracking. Sandia produced a high yield of light material at an even lower temperature, but their space velocity was only about one-half of HTI's.

Characterization of HTI Dewaxing Sample in Preparation of Run ALC-1. HTI provided a sample of the dewaxed VSOH inventory (the solvent hydrotreater feed) from HTI. ¹H-NMR analysis shows that it contains about 8% acetone (the dewaxing solvent). This was reported to HTI, who performed distillation testing to verify this result and plan corrective measures. The original material was only flashed (not distilled), and the acetone removal efficiency was lower than HTI anticipated. HTI found 5% to 7.5% acetone in their distillation of the original material. HTI found suitable distillation procedures that leave only traces of acetone in the stripped product. We stripped the acetone from our sample by rotary evaporation and analyzed the stripped material by elemental analysis, ¹H-NMR, and GC/MS (Table 6). It was our understanding that the oil was to be predominantly a 343 °C⁺ (650 °F⁺) distillate. However, the GC/MS analysis shows that the dewaxed oil has a substantial quantity of 343 °C⁻ (650 °F⁻) material in it (for example, cresol and methylnaphthalene are major components). Clearly, the starting oil selected by HTI for dewaxing and hydrotreating had a lower boiling point distribution than originally desired.

HTI sent dewaxed oil samples (343 °C⁻ (650 °F⁻) and 343 °C⁺ (650 °F⁺) fractions) that were stripped of acetone, hydrotreated, and fractionated at 343 °C (650 °F). CONSOL analyzed the fractions by ¹H-NMR (Table 6) and GC/MS. The aromaticity in both fractions of the hydrotreated oil was reduced relative to the dewaxed oil, which confirms substantial hydrogenation. The GC/MS chromatograms show that the fractions of the hydrotreated oil contain more hydroaromatics and naphthenes than the dewaxed oil.

Characterization of Run ALC-1 Process Samples. HTI provided the requested process oil samples collected during Run ALC-1. These samples are being characterized by CONSOL under DOE Contract DE-AC22-94PC93054.

Task 3 - Continuous Operations

The various process streams produced during the production of the coal-oil agglomerates for Run ALC-1 were analyzed. HTI required the coal-oil agglomerate analyses for input to their material balance and yield calculations for Run ALC-1. The analyses of the rejected solids and water streams will be used to calculate material and elemental balances for the agglomeration runs.

Properties of Coal-Oil Agglomerates Used in Run ALC-1. The complete analyses of the feed coal and feed oil used to produce the agglomerates are provided in Tables 7 and 8, respectively.

The agglomerating oil analysis (Table 8) used to obtain these results is an average of two CONSOL analyses. HTI's re-analysis of this oil generally confirmed these analyses (Table 8). The analyses of the agglomerates produced for Run ALC-1 are presented in Table 9.

The elemental compositions of the oil-free agglomerated coal were calculated for each production agglomeration run (Table 10). These were obtained by backing out the C, H, N, S, and O (by difference) contents of the agglomerating oil (Table 8) from that of the product agglomerates (Table 9) from each batch (C2, C3, etc.) by: 1) adjusting the compositions of the agglomerates to an MAF basis, 2) accounting for the contribution of agglomerating oil as one of two MAF organic components (the other being MAF coal), then 3) adjusting the calculated composition of the coal portion back to an MF basis. Thus, the elemental composition of the MF coal component of each batch of agglomerates was derived by calculation from the analytically-determined elemental compositions of the agglomerating oil and the agglomerates from each batch. The weighting factors for the elemental compositions of each of these components were obtained by normalizing the percent agglomerating oil and percent MAF coal in the agglomerates to represent percent MAF organics. Table 9 includes two entries for cake moisture content and for ash SO_3 . These calculations were based on the Leco ash- SO_3 values and the oven moisture values presented in Table 9.

Some aspects of the agglomerated coal elemental compositions deserve comment. Relative to the composition of the agglomeration feed coal (Table 7), the coal portion of the agglomerates is calculated to be similar in carbon and nitrogen content, higher in hydrogen, sulfur, and oxygen content, and lower in ash content. The sulfur content of the coal was increased by about 0.5 wt % by the acid treatment. If the sulfur is present as sulfate ion, it accounts for about 1 wt % of the oxygen. Thus, the concentration of every element except hydrogen appears to be reasonable. The hydrogen content of the agglomerated coal appears to be about 0.5 wt % high. We have no explanation for this apparent increase; the amount of hydrogen that would be present from ion-exchange should be small. If moisture is under-accounted, it would result in high hydrogen contents. However, we have no reason to think that the moisture content is much higher than is reported here.

Microautoclave tests performed with agglomerated coal and with the agglomeration feed coal seem to indicate lower reactivity of the agglomerated coal. Tests were conducted at three sets of conditions, none employing catalyst. The conditions and conversion of coal to THF solubles

(on a SO_3 -free MAF basis) follow. 1) At 440 °C (824 °F), 30 min, 10.3 MPa (1500 psi) H_2 , 1.5/1 solvent to MF coal ratio. The feed coal gave SO_3 -free coal conversion of $82.9\% \pm 1.7\%$ ($n=3$). Tests of individual Runs C1 through C9 plus a duplicate of the Runs C2 through C9 composite sample gave SO_3 -free coal conversions of $74.5\% \pm 1.4\%$ ($n=11$). The Run C10 agglomerates, produced with a different batch of feed coal, gave a coal conversion of 69.6% ($N=1$). 2) At 5 min, but otherwise the same conditions, the feed coal gave 67.2% conversion and the Run C6 agglomerates gave 68.2% conversion. 3) At 399 °C (750 °F), 30 min, 2/1 solvent to whole coal and no H_2 , the feed coal gave 72.9% conversion and the Run C2 agglomerates gave 69.1% conversion (at the same solvent to MAF coal ratio). Thus, the agglomerates were more difficult to convert to solubles in these tests. Nevertheless, the actual conversion of the agglomerates in Run ALC-1 was excellent.

The sample of composite agglomerates from Run C2 through Run C9 was separated by acetone solubility to see if this method accurately determines the MF coal/oil ratio of the agglomerates. The recovered dry solubles accounted for 26.26% of the feed, the recovered dry insolubles accounted for 69.38% of the feed, and there was 4.35% unrecovered material, including moisture. The estimated composition of this sample is 23.51% agglomerating oil, 68.91% MF coal, and 7.60% moisture (Table 10). The acetone solubility does a fair approximation of separating the two components. The two fractions were analyzed (Tables 11 and 12) to determine, among other things, the form of the sulfur added during low-pH agglomeration. A comparison of the analyses of the acetone solubles (Table 11) and the agglomerating oil (Table 8) shows that the acetone solubles have a similar proton distribution but a higher oxygen-by-difference content, caused, at least in part, by some remaining acetone (apparent in the NMR spectrum). A comparison of the analyses of the acetone insolubles (Table 12) and the feed coal (Table 7) shows the acetone insolubles to have a sulfur content approximately 0.5% higher, primarily because of sulfate retention.

To determine the approximate boiling point range of the agglomerating oil used in the large-scale agglomerate production runs, the oil was analyzed by gas chromatography/mass spectrometry. The earliest eluting major component is pyrene (b.pt. 393 °C). The oil contains the n-paraffins from C_{20} through C_{37} (b.pts. 343 °C through 506 °C), but only C_{23} through C_{32} (b.pts. 380 °C through 467 °C) have substantial concentrations. Thus, its nominal boiling point range is 380-470 °C.

Properties of Solids and Waters Rejected During Run ALC-1 Agglomeration. The elemental analyses of the ashed, rinsed filter cakes are presented in Table 13. The rinsed filter cake is the rejected solids. The analyses indicate that the filter cake ashes are composed primarily of calcium sulfate and silica. These analyses plus information obtained in Task 2 (see above), indicate that the filter cakes (unashed) are composed primarily of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and a siliceous component.

The analyses of the filtrates (the aqueous phase remaining after agglomeration, filtration, and rinsing) are presented in Table 14. The filtrates include the contribution from the agglomerate rinse water. The filtrates were analyzed for a group of elements (reported here as the oxide), acidity, and pH. The rinse water volume varied from run to run, and the dilution of the filtrates varied. However, the data indicate that the principal dissolved species include sulfates of calcium (major), magnesium (medium), and aluminum, iron, and sodium (minor).

The analyses of the feed coal (Table 7), agglomerate product (Table 9) and rejected solids and waters (Tables 13 and 14) indicate that elements selectively rejected from the coal by agglomeration at low pH are Ca, Mg, and Na.

Special measurements and samples were taken during Runs C7, C9, and C11 to provide additional characterization of the reject solids and waters. The special samples included the filter cakes and filtrates of the screen underflow stream (i.e., the material that passes through the screens used to recover the product agglomerates), prior to rinsing the agglomerates. These materials are collected prior to any rinse water dilution. The analyses of the filter cakes are given in Table 15 and those of the filtrates are given in Table 16. The analyses performed on the filter cakes include moisture, ash, and ash elemental analyses. A comparison of the compositions of the samples obtained after rinsing (Tables 13 and 14) with those of the samples obtained without rinsing (Tables 15 and 16) shows that rinsing depletes calcium from the filter cake and concentrates it in the filtrate.

The filter cake (primarily gypsum) produced during agglomeration Run C9 was examined with the optical and scanning electron microscopes. The gypsum was white and composed of 10 to 50 μm crystals. The analyses of the ashed filter cakes from three production agglomeration runs in Table 15 (filter cakes obtained prior to agglomerate rinsing) show them to consist of ca. 89% anhydrite, with the balance mostly silica.

Run ALC-1 Operations. Run ALC-1 started coal feed on April 19 and continued through 4:00 on May 14. CONSOL personnel were on site at HTI to monitor operations for the periods April 23 through May 2 and May 7 through May 12.

Task 4 - Technical Assessment

CONSOL sent a letter to HTI suggesting specific items which should be included in the draft Run ALC-1 report. CONSOL reviewed HTI's draft report on Run ALC-1 and provided comments. We met with Mike Peluso on June 5 in Library to discuss the Run ALC-1 results. CONSOL organized and participated in a post-run review meeting at HTI on June 18 to evaluate results from Run ALC-1 and to plan for future work. Several options were developed for future liquefaction runs under this program. A meeting report was issued.

The paper "Testing of Advanced Liquefaction Concepts in HTI Run ALC-1: Coal Cleaning and Recycle Solvent Treatment" was submitted to the DOE First Joint Power and Fuel Systems Contractors Conference. A shorter version was submitted to the Thirteenth Annual International Pittsburgh Coal Conference.

R. A. Winschel
Project Manager

/s

TABLE 1
ANALYSIS OF RUN A75 AGGLOMERATES

Moisture, wt % As Determined	2.72
Ash, wt % MF, Including SO ₃	2.91
<u>Proximate, wt % MF, SO₃-Free Ash Basis</u>	
Ash, SO ₃ -Free	2.59
Volatile Matter	55.25
Fixed Carbon	42.16
<u>Ultimate, wt % MF, SO₃-Free Ash Basis</u>	
Carbon	75.57
Hydrogen	6.24
Nitrogen	0.94
Sulfur	0.68
Chlorine	<0.02
Oxygen (by difference)	13.98
Ash, SO ₃ -Free	2.59
HHV, Btu/lb (MF)	13,473
<u>Ash Elemental Analysis, wt %</u>	
SiO ₂	38.94
Al ₂ O ₃	24.57
TiO ₂	2.17
Fe ₂ O ₃	8.16
CaO	9.33
MgO	1.29
Na ₂ O	0.29
K ₂ O	0.54
P ₂ O ₅	2.03
SO ₃	10.92
Undetermined	1.76

TABLE 2

ANALYSIS OF RUN A78 FILTER CAKE
DRIED, BUT NOT ASHED

Element Oxide	wt %
SiO ₂	16.53
Al ₂ O ₃	0.05
TiO ₂	0.06
Fe ₂ O ₃	0.18
CaO	27.04
MgO	0.06
Na ₂ O	0.04
K ₂ O	0.02
P ₂ O ₅	0.01
SO ₃	39.05
Undetermined	16.97(a)

(a) Believed to consist principally of gypsum hydration water.

TABLE 3

OPERATING CONDITIONS AND SIMULATED DISTILLATION DATA
SANDIA HYDROTREATING SAMPLES FOR RUN ALC-1
(Data supplied by Sandia)

	Temp., °C	Total Pressure, psig	H ₂ Treat Rate, SCF/bbl	WHSV	Sim Dist Data			
					IBP, °F	wt % off @ 600 °F	wt % off @ 650 °F	FBP, °F
Feedstock HTI PFL VSOH 227-90-10	-	-	-	-	-445	-9.2	20.4	950
Products								
100-35-96C	365	1800	9000	1	269	21.7	38.0	947
100-37-96A	375	1800	9000	1	218	23.3	39.5	941
100-38-96B	385	1800	9000	1	215	24.8	40.8	936
100-311-96B	395	1800	9000	1	215	26.6	42.4	935
100-312-96B	405	1800	9000	1	209	31.3	47.0	937
100-313-96B	365	1800	9000	1	<400	20.7	35.8	944
100-315-96A	365	1800	4500	1	<400	13.9	24.4	69.7% off @ 1000 °F
100-316-96A	365	1800	3000	1	<400	12.6	22.4	68.8% off @ 1000 °F

Catalyst - Criterion C-411 Trilobe (Ni/Mo/Al)

Source: Fax of J. Miller to M. Peluso 4/3/96, personal communication with J. Miller 4/10/96, and fax of J. Miller to R. Winschel 4/10/96.

TABLE 4
ELEMENTAL ANALYSES OF SANDIA HYDROTREATING SAMPLES
FOR RUN ALC-1

Analysis, wt % as Determined	C	H	N	S	O (diff.)	Comment
Feedstock HTI PFL VSOH 227-90-10	88.24	8.38	0.67	1.17	1.54	Coal liquid odor, black
Products						
100-35-96C	88.38	10.47	0.08	0.18	0.89	NH ₃ odor
100-37-96A	88.65	10.68	0.04	0.13	0.50	NH ₃ odor
100-38-96B	88.56	10.52	<0.01	0.08	0.84	Sweet odor
100-311-96B	88.81	10.55	0.01	0.06	0.57	Sweet Odor
100-312-96B	88.60	10.60	<0.01	0.03	0.77	Sweet Odor
100-313-96B	88.32	9.79	0.22	0.30	1.37	NH ₃ odor
100-315-96A	88.50	9.86	0.28	0.46	0.90	NH ₃ odor
100-316-96A	89.07	9.72	0.31	0.53	0.37	NH ₃ odor

TABLE 5
PROTON DISTRIBUTIONS OF SANDIA HYDROTREATING SAMPLES FOR RUN ALC-1

	Proton Distribution, %							Microautoclave (a) Coal Conversion wt % MAF SO ₂ - free
	Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma	
Feedstock HTI PFL VSOH 227-90-10	20.4	8.4	16.2	11.9	11.8	21.0	10.3	80.9
Products								
100-35-96C	7.7	7.1	14.4	10.3	20.1	25.6	14.8	-
100-37-96A	8.0	6.7	14.5	10.0	20.4	25.8	14.7	-
100-38-96B	8.2	6.1	14.2	9.4	20.6	26.3	15.3	89.2
100-311-96B	9.2	6.4	13.5	9.6	19.5	26.0	15.8	-
100-312-96B	8.6	6.3	13.0	8.9	20.0	26.6	16.6	-
100-313-96B	10.1	6.9	15.6	10.1	19.4	24.9	13.0	-
100-315-96A	11.3	6.8	15.6	11.3	18.1	24.0	12.9	-
100-316-96A	13.5	6.5	16.5	10.6	16.7	24.0	12.3	88.4

(a) Conditions: 5.45 g solvent, 4.55 Black Thunder Mine coal (1621-22-1), 840 °F, 30 min, 1500 psig cold H₂, 45 mL vessel, THF solubles.

TABLE 6
ANALYSIS OF HTI DEWAXED AND DEWAXED-HYDROTREATED OILS

Sample	WT %						
	C	H	N	S	O (Diff)	Btu/lb	
HTI Dewaxed Oil for ALC-1 (a)	87.60	8.80	0.72	0.70	2.18	17,354	
	Proton Distribution, %						
Sample	Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
HTI Dewaxed Oil for ALC-1 (a)	16.6	9.5	15.2	12.7	13.6	19.4	130
HTI Dewaxed-Hydrotreated Oil, 650 °F Fraction	10.0	6.1	17.4	10.4	19.6	22.2	14.2
HTI Dewaxed-Hydrotreated Oil, 650 °F Fraction	5.2	6.5	13.6	9.8	22.6	24.6	17.8

(a) Acetone removed by CONSOL before analysis.

TABLE 7

ANALYSIS OF HTI BLACK THUNDER MINE COAL FOR AGGLOMERATION

	Black Thunder Mine Coal, HTI No. 6213					HTI No. 6392 Drum 6 (b)	Lab Composite Drums 1-4 (c)
	Drum 1	Drum 2	Drum 3	Drum 4	Average Drums 1-4 (a)		
Moisture, wt % As Determined	10.00	10.00	9.98	9.65	9.91	8.47	9.70
Ash, wt % MF, Including SO ₃	6.12	6.31	6.22	6.28	6.23	6.34	6.28
<u>Proximate, wt % MF, SO₃-Free Ash Basis</u>							
Ash, SO ₃ -Free	5.39	5.56	5.49	5.55	5.50	5.78	5.23
Volatile Matter	43.65	43.72	43.63	43.61	43.65	43.69	44.89
Fixed Carbon	50.96	50.72	50.88	50.84	50.85	50.55	49.88
<u>Ultimate, wt % MF, SO₃-Free Ash Basis</u>							
Carbon	71.00	71.21	71.05	71.05	71.08	69.83	70.72
Hydrogen	4.67	4.69	4.68	4.73	4.69	4.67	4.74
Nitrogen	0.92	0.92	0.91	0.91	0.92	0.96	1.00
Sulfur	0.49	0.52	0.49	0.48	0.50	0.44	0.49
Chlorine	-	-	-	-	-	-	-0.02
Oxygen (by diff.)	17.53	17.10	17.36	17.28	17.32	18.34	17.82
Ash, SO ₃ -Free	5.39	5.56	5.49	5.55	5.50	5.76	5.23
SO ₃ , wt % of Ash	11.94	11.81	11.81	11.57	11.78	9.20	16.42
HHV, Btu/lb (MF)	12,066	12,061	12,050	12,088	12,069	11,753	11,967
<u>Sulfur Forms, wt % ME</u>							
Pyritic							0.07
Sulfate							0.01
Organic							0.41
<u>Ash Elemental Analysis, wt %</u>							
SiO ₂							31.21
Al ₂ O ₃							16.00
TiO ₂							1.21
Fe ₂ O ₃							5.57
CaO							21.55
MgO							4.51
Na ₂ O							1.46
K ₂ O							0.43
P ₂ O ₅							1.14
SO ₃							16.42
Undetermined							0.50

- (a) Drums 1-4 were the feed to agglomeration production Runs C1-C9.
 (b) Drum 5 was the feed to agglomeration production Runs C10 and C11.
 (c) Lab composite was the feed to laboratory agglomeration tests.

TABLE 8

ANALYSES OF AGGLOMERATING OIL USED TO PRODUCE RUN ALC-1 FEEDS

Sample description: Agglomerating Oil for Run ALC-1 Feed, V-1074 Heavy Distillate from Wilsonville Run 263 End-of-Run Inventory				
Elemental Analysis, wt % As-Determined				
	CONSOL Analysis, Det'n 1	CONSOL Analysis, Det'n 2	CONSOL Average*	HTI Analysis
Carbon	89.85	89.63	89.74	89.45
Hydrogen	8.82	8.80	8.81	9.16
Nitrogen	0.65	0.52	0.59	0.61 to 1.03
Sulfur	0.02	0.03	0.03	0.02
Oxygen (by diff.)	0.66	1.02	0.84	-
Heating Value (Btu/lb)	17,958	17,949	17,954	-
<u>Proton Distribution, %</u>				
Cond Arom	14.9			
Uncond Arom	5.4			
Cyclic Alpha	16.7			
Alkyl Alpha	8.7			
Cyclic Beta	14.6			
Alkyl Beta	27.6			
Gamma	12.1			

*Average value used to calculate elemental composition of coal component of agglomerates.

TABLE 9
COMPOSITIONS OF PRODUCTION BATCHES OF AGGLOMERATES

	C1	C2	C3	C4	C5	C6	C6/C7	C7	C8	C9	Composite C2-C9	C10	C11
Moisture, wt % as det. (a)													
Leco	4.78	4.80	6.28	4.48	6.64	6.42	6.54	6.21	6.83	6.97	6.91	6.88	7.20
Oven	5.48	5.77	8.10	6.65	7.90	7.64	7.48	7.79	8.12	8.67	7.31	9.32	8.50
Proximate, wt % dry													
Ash, SO ₃ -Containing	3.10	2.73	2.74	2.86	2.80	2.67	2.78	2.71	2.72	2.72	2.77	2.78	2.67
Volatile Matter	55.80	56.15	56.35	56.29	56.30	55.91	56.21	55.53	56.02	56.28	-	56.52	56.96
Fixed Carbon	41.10	41.12	40.91	40.85	40.90	41.42	41.03	41.76	41.26	41.00	-	40.70	40.17
Ultimate, wt % dry													
Carbon	75.16	75.06	74.84	74.44	74.85	74.71	74.86	74.76	74.70	74.08	75.00	73.19	75.29
Hydrogen	6.20	6.07	6.30	6.27	6.32	6.25	6.09	6.08	6.37	6.13	6.07	6.21	6.75
Nitrogen	0.86	0.85	0.84	0.84	0.85	0.85	0.86	0.85	0.86	0.84	0.85	0.83	0.86
Chlorine	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Sulfur	0.66	0.74	0.73	0.75	0.72	0.74	0.71	0.71	0.73	0.71	0.74	0.69	0.68
Ash, SO ₃ -free	2.82	2.47	2.50	2.59	2.53	2.43	2.51	2.49	2.45	2.42	2.51	2.44	2.57
Oxygen (diff)	14.30	14.81	14.79	15.11	14.73	15.02	14.96	15.11	14.89	15.84	14.83	16.64	13.85
HHV, Btu/lb dry	13462	13430	13412	13489	13487	13514	13458	13459	13443	13367	13559	13115	13452
Major Ash Elements, %													
Ignited at 750 °C													
SiO ₂	45.06	42.89	43.86	42.90	42.40	43.27	42.69	44.41	42.68	41.62	41.59	39.83	43.00
Al ₂ O ₃	23.66	24.46	24.97	23.65	24.46	24.46	24.20	25.05	24.06	23.82	23.68	22.87	21.95
TiO ₂	2.17	2.36	2.35	2.26	2.30	2.30	2.31	2.28	2.25	2.21	2.32	2.03	1.92
Fe ₂ O ₃	8.86	8.22	8.28	8.87	7.76	7.52	7.50	7.56	7.36	7.76	8.68	7.32	5.96
CaO	7.84	8.04	7.86	8.21	7.98	7.85	7.57	7.52	8.67	7.88	7.70	8.66	9.38
MgO	1.02	1.27	1.38	1.33	1.38	1.35	1.33	1.35	1.30	1.31	1.27	1.30	1.35
Na ₂ O	0.29	0.37	0.36	0.37	0.40	0.36	0.38	0.39	0.41	0.36	0.36	0.33	0.39
K ₂ O	0.46	0.47	0.50	0.45	0.47	0.48	0.48	0.50	0.56	0.47	0.48	0.40	0.41
P ₂ O ₅	1.83	1.74	1.90	1.76	1.91	1.97	1.97	2.02	1.94	1.88	1.82	2.10	2.01
SO ₃	9.79	9.77	9.33	10.04	10.18	9.59	9.59	9.09	10.93	11.93	9.70	12.90	12.43
SO ₃ (Leco) (b)	8.99	9.22	8.73	9.53	9.51	8.97	8.96	8.24	10.00	11.1	9.32	12.3	10.5
Mo (mg/kg)	38.5	62.5	43	42	45	45	52	41	38.5	44	49	43.5	41
Undetermined	-0.98	0.41	-0.83	0.16	0.76	0.83	1.98	-0.17	-0.16	0.76	2.40	2.26	1.20
Sieve Size, mesh													
+4, wt %											4.8		
4 x 8, wt %											41.4		
8 x 14, wt %											52.7		
4 x 28, wt %											0.7		
8 x 48, wt %											0.0		
48, wt %											0.4		

- a) Two moisture methods used: Leco TGA-501 Instrument, and 100 °C oven overnight. Oven method used to calculate composition of shipped material. Leco method used to calculate dry analyses shown here.
- b) Sulfur by Leco SC-32 Instrument. This value used to calculate SO₃-free ash content. This value was excluded from the calculation of the "Undetermined" components.

TABLE 10

COMPOSITION OF AGGLOMERATE PRODUCTS FROM RUNS C1-C10
(HTI Run ALC-1 Feedstocks)

	Bag No.											
	C2	C3	C4	C5	C6	C7	C8	C9	C6+C7	C1	C10	C2-C9 Avg. ± Std. Dev.
Composition, wt % of Whole Sample												
Moisture	5.77	8.10	6.65	7.90	7.64	7.79	8.12	8.67	7.48	5.48	9.32	7.58 ±0.93
Agglom. Oil	24.06	23.40	23.62	23.36	23.46	23.43	23.41	23.34	23.49	23.28	22.95	23.51 ±0.24
Ash, SO ₃ -Free	2.37	2.34	2.47	2.37	2.27	2.33	2.29	2.26	2.35	2.68	2.28	2.34 ±0.07
MAF Coal	67.81	66.15	67.25	66.37	66.63	66.44	66.18	65.73	66.68	68.55	65.45	66.57 ±0.68
MF Coal (MAF Coal + Ash)	70.17	68.50	69.73	68.74	68.90	68.78	68.47	67.99	69.03	71.24	67.73	68.9 ±10.71
Additional Analyses, wt % of Whole Sample, As-Determined												
Carbon	71.61	70.14	71.11	69.88	69.92	70.12	69.76	68.90	69.97	71.57	68.17	70.18 ±0.84
Hydrogen*	6.31	6.61	6.49	6.64	6.58	6.40	6.69	6.48	6.42	6.44	6.55	6.52 ±0.13
Nitrogen	0.82	0.79	0.81	0.80	0.80	0.80	0.81	0.79	0.80	0.82	0.77	0.80 ±0.01
Sulfur	0.71	0.69	0.72	0.67	0.70	0.67	0.69	0.67	0.68	0.63	0.64	0.69 ±0.02
Heating Value, Btu/lb	12,812	12,570	12,886	12,592	12,647	12,624	12,553	12,435	12,578	12,819	12,216	12,640 ±145
Elemental Composition, wt % of MF Coal Portion of Agglomerates (Calculated on Oil-Free, Moisture-Free, Ash-SO ₃ -Free Basis)												
Ash, SO ₃ -Free	3.27	3.28	3.46	3.37	3.23	3.27	3.25	3.02	3.37	3.56	3.28	3.27 ±0.13
Carbon	71.36	71.84	71.64	71.22	70.98	71.46	71.26	70.74	70.84	71.29	70.29	71.31 ±0.35
Hydrogen**	5.05	5.31	5.26	5.37	5.30	5.04	5.43	5.09	5.09	5.31	5.15	5.23 ±0.15
Nitrogen	0.96	0.95	0.96	0.96	0.96	0.96	0.98	0.98	0.98	0.96	0.94	0.96 ±0.01
Sulfur	1.01	0.99	1.03	0.97	1.00	0.96	0.99	0.97	0.97	0.88	0.94	0.99 ±0.02
Oxygen (by diff.)	18.36	17.62	17.66	18.12	18.53	18.31	18.09	19.21	18.77	18.00	19.41	18.24 ±0.51

* Includes hydrogen from moisture (as-determined basis).

** Corrected to exclude hydrogen from moisture.

TABLE 11

**ANALYSIS OF DRY ACETONE SOLUBLES
OF COMPOSITE RUNS C2-C9 AGGLOMERATES**

	wt %
C	86.94
H	8.06
N	0.42
S	0.09
O (diff.)	4.49

Proton Distribution, %						
Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
13.8	5.2	15.1	8.6	14.7	29.6	13.0

TABLE 12
ANALYSIS OF DRY ACETONE INSOLUBLES
OF COMPOSITE RUNS C2-C9 AGGLOMERATES

Moisture, wt % As Determined	1.37
Ash, wt % MF, including SO ₃	3.38
<u>Proximate, wt % MF, SO₃-Free Ash Basis</u>	
Ash, SO ₃ -Free	3.02
Volatile Matter	46.77
Fixed Carbon	50.21
<u>Ultimate, wt % MF, SO₃-Free Ash Basis</u>	
C	70.68
H	4.84
N	1.08
S, Total	0.93
S, Pyritic	0.26
S, Sulfate	0.46
S, Organic	0.21
O (diff.)	19.45
Ash, SO ₃ -Free	3.02
HHV, Btu/lb, MF Basis	11,885
<u>Ash Elemental Analysis, wt %</u>	
SiO ₂	42.91
Al ₂ O ₃	25.63
TiO ₂	2.04
Fe ₂ O ₃	6.63
CaO	8.19
MgO	1.32
Na ₂ O	0.49
K ₂ O	0.62
P ₂ O ₅	1.86
SO ₃	10.58
Undetermined	-0.27

TABLE 13

ANALYSES OF ASHED FILTER CAKES OBTAINED WITH RINSING

	wt %, as determined										
	C1*	C2	C3	C4	C5	C6	C7	C8	C9	C10	C11
SiO ₂		20.24	14.22	27.15	18.41	21.91	41.54	20.04	24.30	24.60	13.16
Al ₂ O ₃		0.23	0.41	0.47	0.52	0.57	2.07	0.56	0.80	0.24	0.27
TiO ₂		0.03	0.02	0.03	0.03	0.04	0.06	0.06	0.04	0.03	0.03
Fe ₂ O ₃		0.21	0.25	0.48	0.49	0.45	0.88	0.42	0.50	0.29	0.62
CaO		32.06	29.90	25.49	28.40	27.74	20.45	31.40	30.27	30.50	34.69
MgO		0.03	0.08	0.03	0.04	0.05	0.04	0.21	0.05	0.03	0.09
Na ₂ O		0.02	0.05	0.02	0.03	0.03	0.03	0.06	0.06	0.02	0.04
K ₂ O		0.04	0.04	0.06	0.09	0.05	0.08	0.05	0.06	0.04	0.03
P ₂ O ₅		0.03	<0.01	<0.01	<0.01	<0.01	0.07	<0.01	0.01	<0.01	<0.01
SO ₃		46.62	52.54	44.65	49.95	47.36	33.23	46.45	43.80	44.73	51.01
Undetermined		0.49	2.49	1.62	2.04	1.80	1.55	0.75	0.11	-0.48	0.06

*No analysis

TABLE 14

ANALYSES OF FILTRATES OBTAINED WITH RINSING

	Concentration, mg/L, as determined										
	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10	C11
SiO ₂	12.09	17.83	24.85	11.27	16.23	13.31	11.15	13.07	14.20	12.45	12.51
Al ₂ O ₃	133.56	251.00	436.75	170.36	260.36	214.42	133.19	208.10	169.56	218.01	218.43
TiO ₂	0.01	0.08	0.04	0.18	0.17	0.13	0.15	0.14	0.12	0.13	0.14
Fe ₂ O ₃	139.66	257.36	363.70	152.48	213.67	208.51	116.23	164.45	151.17	216.44	226.09
CaO	741.13	731.07	733.40	742.92	724.11	754.64	790.35	730.48	764.61	745.50	743.89
MgO	250.91	368.86	641.53	263.09	398.22	321.33	207.60	310.13	268.17	345.67	369.30
Na ₂ O	108.46	147.94	340.94	181.98	293.49	194.36	169.63	202.12	168.77	187.01	175.02
K ₂ O	13.37	14.66	28.08	10.61	16.14	12.80	8.56	37.05	12.36	12.11	14.08
P ₂ O ₅	3.40	11.93	15.03	6.54	8.01	8.41	4.48	6.76	5.37	10.22	9.01
SO ₃	2628.50	3729.50	5449.00	2843.00	3597.00	3283.00	2470.00	3216.00	2851.00	3455.00	3517.00
Acidity, mg/L CaCO ₃	1093	2105	3251	1208	1653	1568	823	1550	1195	1793	1844
pH	2.30	2.01	1.88	2.28	2.25	2.19	2.61	2.22	2.31	2.08	2.06

TABLE 15

ANALYSES OF FILTER CAKES OBTAINED WITHOUT RINSING

	C7	C9	C11
Moisture, wt %, as determined	0.59	1.60	3.96
Ash, wt % of dry cake	97.83	98.16	98.31
<u>Ash Elemental, wt % of Ash</u>			
SiO ₂	8.44	11.24	6.63
Al ₂ O ₃	0.72	0.13	0.08
TiO ₂	0.01	0.01	0.01
Fe ₂ O ₃	0.13	0.10	0.09
CaO	35.64	34.35	36.83
MgO	0.13	0.09	0.03
Na ₂ O	0.06	0.05	0.02
K ₂ O	0.02	0.01	0.01
P ₂ O ₅	<0.01	<0.01	<0.01
SO ₃	53.51	50.96	53.78
Undetermined	1.34	3.06	2.52

TABLE 16

ANALYSES OF FILTRATES OBTAINED WITHOUT RINSING

	Concentration, mg/L, as determined		
	C7	C9	C11
SiO ₂	40.07	36.33	28.09
Al ₂ O ₃	1061.00	979.33	866.62
TiO ₂	0.19	0.18	0.09
Fe ₂ O ₃	769.89	729.36	764.01
CaO	675.39	676.63	680.25
MgO	1589.00	1502.00	1436.00
Na ₂ O	543.67	522.67	549.35
K ₂ O	61.13	70.72	53.10
P ₂ O ₅	31.25	30.36	36.25
SO ₃	11050	10690	10440
Acidity, mg/L CaCO ₃	7879	7719	7702
pH	1.53	1.49	1.46

SECTION THREE

Hydrocarbon Technologies, Inc.

Hydrocarbon Technologies, Inc.
P.O. Box 6047
New York & Puritan Avenues
Lawrenceville, New Jersey 08648
609/394-3102 Fax 609/394-9602



Theo L.K. Lee
Vice President

June 5, 1996

Via Facsimile & Regular Mail

Dr. Ed Givens
Center for Applied Energy Research
University of Kentucky
3572 Iron Works Pike
Lexington, KY 40511-8433

RE: DOE Advanced Coal Liquefaction Concepts - Phase II Program
Sub Contract: UKRF 425582-96-152
May/96 Report

Dear Ed:

Works undertaken during this report period include:

1. Completed Run ALC-01
2. Continuing with Product Workup on TBP fractions
3. Preparing the ALC-01 Run Report
4. Forwarded all the required samples to Consol

A list of operational recommendations is attached for your comment. A draft run report will be issued by the week of June 10-14 for your review and comment.

Sincerely,

Theo L.K. Lee

LKL/dms
Enclosures

cc: R. Winschel - via regular mail
M. Peluso - via regular mail

96/LKL/105

Future Unit 227 operations that mimic Run 94 parameters would have the following operational issues to address:

- A - Feed Blending
- B - Achieving 650+ Extinction
- C - Improving Filtration Cycle Time
- D - Controlling Distillation Cut Points
- E - Shortening Equipment Cycle Times for Dewaxing

A short discussion of each of these items follows.

A - Feed Blending

Prior to run 94 operations, it was thought that in order to quickly change the solids content of the feed it would be necessary to produce the feed blends on a 1 to 2 hour basis. However, such fine control of the feed blend solids content never really became an issue. Utilizing already established 6 hour blending procedures would be acceptable for future operations. In addition, this procedure would enable a more thorough and longer feed blend mixing.

B - Achieving 650+ F Extinction

The procedures established during run 94 operations, namely diluting the filter feed blend with a distillate material to control the feed viscosity and then vacuum distilling the pressure filter liquid (PFL) should be adopted as part of the operating strategy. This will enable an extinction of the 650 + F fraction of the PFL. In addition, the temperatures of the hot separator (O-1) and atmospheric flash vessel (O-3) should be adjusted to remove as much 650- F material as possible prior to pressure filtration.

C - Improvements to Filtration Cycle Time

The filtration cycle time is mainly determined by unit material production and the physical work needed to turn a filter around. A two hour cycle time is about the practical minimum. For the majority of run 94, this was achieved. Long filtration times are usually attributed to a high viscosity feed. The only improvements would be to increase the surface area, decrease the amount of feed or lower the viscosity of the feed. A larger plate filter is presently under going commissioning. The addition of distillate material to the feed is used to control the viscosity.

D - Controlling Distillation Cut Points

The bench unit vacuum distillation system is a simple single stage flash. To ensure that the distillation cuts meet specifications, the comparison between laboratory analysis and plant vacuum distillation should be closely monitored. Any needed adjustments to the batch vacuum still could be quickly made.

E - Shortening Equipment Cycle Times for Dewaxing and Hydrotreating

The relatively long dewaxing/hydrotreating equipment cycle time is not easily reduced. A number of different equipment cycles must be optimized to show a significant change in the present two day lag. However, the impact of the following equipment and procedure modifications should be investigated.

- A - Larger dewaxing vessels that could substitute for blending vessels
- B - Product flash system to remove a large portion of the acetone prior to distillation
- C - Possibly a larger hydrotreater may be needed if the preceding items decrease the lag time significantly

SECTION FOUR

LDP Associates

LDP ASSOCIATES

Michael Peluso, Proprietor
609-586-2301

32 Albert E. Bonacci Dr.
Hamilton Square, N.J. 08690

August 12, 1996

Dr. Ed Givens
Center for Applied Energy Research
3572 Iron Works Pike
Lexington, Kentucky 40511-8433

Dear Ed:

Subject: QUARTERLY PROGRESS REPORT FOR APRIL THRU JUNE 1996

For the quarter ending June 30, 1996 the following subcontract services (UKRF-4-25582-92-75) were performed in support of the DOE Advanced Concepts Program (DE-AC22-91PC91040):

SUPPORT FOR ALC-1

During the quarter the first continuous bench scale run, ALC-1 was made at HTI.

Prior to the run, the results of SANDIA's work on hydrotreating a vacuum still overhead (VSOH) supplied by HTI was reviewed. It was found that, at the conditions used, a significant amount of 343°C+(650°F+) distillate present in the feed was converted to 343°C-(650°F-) material. Since this is not advantageous in this particular situation, CONSOL and HTI were advised of this finding and it was recommended that HTI conduct its pre-ALC-1 dewaxed oil hydrotreating at the lowest possible thermal severity. The use of an alternative catalyst to achieve the desired high degree of aromatics saturation was briefly investigated.

During ALC-1, LDP participated in the daily meetings at HTI, monitored run conditions and results, performed data analysis and assisted in planning operating conditions. CONSOL and CAER were kept up to date on run activities when their representatives weren't onsite. Post run meetings were held with HTI to collect and review run and analytical data and discuss material balancing procedures.

An elementally balanced computerized material balance procedure was developed and used to calculate yields, hydrogen consumption, distillate boiling range yields and estimated distillate product properties for the liquefaction section of the five run conditions (see Attachment #1, Condition #5). An estimated material balance was also prepared for the distillate solvent dewaxing and hydrotreating system of Condition #5 (see Attachment #2) in order to estimate overall yields and hydrogen consumption.

A preliminary analysis of ALC-1 was presented at the ALC-1 Review Meeting at HTI on June 18th (see Attachment #3). Suspect run data was identified and recommendations were made for achieving more reliable results in future ALC runs at HTI.

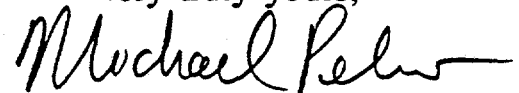
ALC-2 RUN PLAN

A meeting was attended with CAER and HTI representatives to discuss the preliminary concept and process configuration improvements for run ALC-2.

ECONOMIC ASSESSMENT

No activity.

Very truly yours,

A handwritten signature in black ink, appearing to read "Michael Peluso", with a long horizontal flourish extending to the right.

Michael Peluso
LDP Associates

cc: F. Derbyshire @ CAER
R. Anderson @ CAER
R. Winschel @ CONSOL
T. Lee @ HTI

ATTACHMENT #1

**ELEMENTALLY BALANCED MATERIAL BALANCE
FOR CONDITION #5**

CAER AC : ALC-1, CONDITION #5 (Period 25)

	1	2	3	4	5	6	7	8
1	ALC-1							
2	Condition #5 Liq'n Only							
3	Period #25							
4								
5	INPUT STREAMS	Grams	C	H	O	N	S	SO3-Free Ash
6	MF Coal	22399	71.07	4.69	17.32	0.92	0.50	5.50
7	Preprepared DW-HT	6868	88.10	8.47	2.02	0.65	0.76	0.00
8	Molyvan A	5.36	11.00	1.80	0.00	4.30	39.10	43.80
9	Fe2O3	224	0.00	0.00	0.00	0.00	0.00	100.00
10	H2S	960	0.00	5.92	0.00	0.00	94.08	0.00
11	TNPS	1893	53.52	9.48	0.00	0.00	37.00	0.00
12	VSB	6630	89.70	5.51	3.12	1.41	0.26	0.00
13	Recycle DW-HT(P23)	12663	90.20	9.34	0.11	0.23	0.12	0.00
14	PRC	7397	50.46	3.57	7.12	0.51	7.22	31.12
15	TOTAL	59039	44085	3681	4766	411	2336	3760
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								

2-66

CAER AC : ALC-1, CONDITION #5 (Period 25)

1	2	3	4	5	6	7	8
29	MAF/MF Coal	0.945	Frac. of C1-C3		Frac. of C4-C7		Fraction of COx
30	% C1-C3 Yld MAF Coal	10.32	C1 0.305	C4	0.47		CO 0.26
31	% C4-C7 Yld MAF Coal	4.27	C2 0.305	C5-C7	0.53		CO2 0.74
32	% COx Yld MAF Coal	6.76	C3 0.390				
33	OUTPUT STREAMS	Grams	C	O	N	S	SO3-Free Ash
34	H2O Net	0.00	0.00	88.81	0.00	0.00	0.00
35	H2S	0.00	0.00	5.92	0.00	94.08	0.00
36	NH3	0.00	0.00	17.76	0.00	0.00	0.00
37	CO	372.03	42.88	57.12	0.00	0.00	0.00
38	CO2	1058.86	27.29	72.71	0.00	0.00	0.00
39	C1	666.25	74.86	0.00	0.00	0.00	0.00
40	C2	666.25	79.88	0.00	0.00	0.00	0.00
41	C3	851.93	81.71	0.00	0.00	0.00	0.00
42	C4	422.09	82.86	0.00	0.00	0.00	0.00
43	C5+ Gas	481.74	83.81	0.00	0.00	0.00	0.00
44	SOH Oil	8657.00	86.74	0.25	0.11	0.11	0.00
45	VSOH 1	0.00	0.00	0.00	0.00	0.00	0.00
46	975F- VSOH	22586.00	89.51	1.13	0.53	0.15	0.00
47	VSF	7526.00	89.70	3.12	1.41	0.26	0.00
48	PFC	11237.00	50.46	7.12	0.51	7.22	31.12
49	Total	54525.17	43075.04	2294.15	292.65	874.28	3496.95
50			CARBON				
51	ASH BALANCE		C in PFC Out				
52	OUT	3496.95	Bal. of C Out				
53	IN	3760.24					
54	PFC Out Adjust Factor	1.0753	C In	44085.06			
55	PFC Out for Ash Bal.	12083.03	C In - C In PFC	37987.97			
56			Bal C Out A.F.	1.01559			

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CAER AC : ALC-1, CONDITION #5 (Period 25)

	1	2	3	4	5	6	7	8
57								
58								
59								
60	BALANCED							
61	OUTPUT STREAMS	Grams	C	H	O	N	S	SO3-Free Ash
62	H2O Net	0.00	0.00	11.19	88.81	0.00	0.00	0.00
63	H2S	0.00	0.00	5.92	0.00	0.00	94.08	0.00
64	NH3	0.00	0.00	17.76	0.00	82.24	0.00	0.00
65	CO	377.83	42.88	0.00	57.12	0.00	0.00	0.00
66	CO2	1075.37	27.29	0.00	72.71	0.00	0.00	0.00
67	C1	676.64	74.86	25.14	0.00	0.00	0.00	0.00
68	C2	676.64	79.88	20.12	0.00	0.00	0.00	0.00
69	C3	865.21	81.71	18.29	0.00	0.00	0.00	0.00
70	C4	428.67	82.66	17.34	0.00	0.00	0.00	0.00
71	C5+ Gas	489.25	83.81	16.19	0.00	0.00	0.00	0.00
72	SOH Oil	8791.96	86.74	12.79	0.25	0.11	0.11	0.00
73	VSOH1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
74	975F- VSOH	22938.10	89.51	8.68	1.13	0.53	0.15	0.00
75	VSB	7643.33	89.70	5.51	3.12	1.41	0.26	0.00
76	PFC	12083.03	50.46	3.57	7.12	0.51	7.22	31.12
77	Total w/H2O, H2S, NH3	60357.58	44085.06	4998.94	4765.69	411.28	2336.36	3760.24
78	Total exH2O, H2S, NH3				2377.68	300.64	936.35	
79	O BALANCE		N BALANCE		S BALANCE		H BALANCE	
80	IN	4765.69	IN	411.28	IN	2336.36	OUT	4998.94
81	OUT - H2O	2377.68	OUT - NH3	300.64	OUT - H2S	936.35	IN	3680.73
82	O IN H2O	2388.01	N IN NH3	110.64	S IN H2S	1400.02	CONSUMED	1318.22
83	H2O OUT	2688.90	NH3 OUT	134.53	H2S OUT	1488.11	Wt % MF Coal	5.89
84								

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CAER AC : ALC-1, CONDITION #5 (Period 25)

	1	2	3	4	5	6	7	8
8 5								
8 6								
8 7		GROSS YIELDS	Wt. % MF Coal INPUTS	NET YIELDS			Wt. % MAF Coal NET YIELDS	
8 8		H2O	12.00	0.00	12.00		12.70	
8 9		H2S	6.64	7.41	-0.77		-0.81	
9 0		NH3	0.60	0.00	0.60		0.64	
9 1		O	1.69	0.00	1.69	COX	6.87	
9 2		CO2	4.80	0.00	4.80			
9 3		C1	3.02	0.00	3.02	C1-C3	10.48	
9 4		C2	3.02	0.00	3.02			
9 5		C3	3.86	0.00	3.86			
9 6		C4	1.91	0.00	1.91		2.03	
9 7		C5+ Gas	2.18	0.00	2.18		2.31	
9 8		SOHOL	39.25	5.32	33.93		35.90	
9 9		Recycle DW-HT(P23)	0.00	56.53	-56.53		-59.82	
1 0 0		975F- VSOH	102.41	0.00	102.41		108.37	
1 0 1		VSBL	34.12	29.60	4.52		4.79	
1 0 2		PPC	53.94	33.02	20.92		22.14	
1 0 3		Prepared DW-HT	0.00	30.66	-30.66		-32.45	
1 0 4		MOLYBANA	0.00	0.02	-0.02		-0.03	
1 0 5		Fe2O3	0.00	1.00	-1.00		-1.06	
1 0 6		Total	269.47	163.58	105.89		112.05	
1 0 7								
1 0 8				H2 Consumed	5.89		6.23	
1 0 9				H2 for TNPS	0.24		0.25	
1 1 0				Net H2 Consd	5.65		5.97	
1 1 1								
1 1 2								

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CAER AC : ALC-1, CONDITION #5 (Period 25)

1	2	3	4	5	6	7	8
113	BOILING RANGE	SUMMARY					
114	NET YIELD						
115	Wt. % MF Coal	IBP-350 F	350-500 F	500-650 F	650-850 F	850-975 F	Solids - Free 975 F+
116	C4	100.00	0.00	0.00	0.00	0.00	0.00
117	C5+	100.00	0.00	0.00	0.00	0.00	0.00
118	SOHOIL	29.80	24.40	30.40	15.40	0.00	0.00
119	Recycle DW-HT(P23)	0.00	0.00	4.80	82.70	12.50	0.00
120	975F- VSOH	0.00	2.50	18.60	70.60	8.30	0.00
121	VSBI	0.00	0.00	0.00	0.00	2.30	97.70
122	PFC	0.00	0.88	6.48	24.54	3.15	11.03
123	Preprepared DW-HT	0.00	0.00	3.90	87.70	8.40	0.00
124	Total	14.21	11.02	26.81	9.01	-0.38	6.73
125		Wt. % MAF Coal					
126	C4 - 975 F	60.67	64.20				
127				Wt. % Solids in PFC	Wt. % SO3 Free Ash in PFC	Wt. % 975 F+ in PFC	Wt. % 975 F- in PFC
128	Solids Free 975 F+	6.73	7.12	53.92	31.12	11.03	35.05
129				Wt. % MAF Coal			
130	Solids	11.28	11.94	Conversion			
131	• IOM	4.77	5.05	94.66			
132	• SO3 Free Ash	6.51					
133		IBP-350F	15.04				
134		350-500F	11.66				
135		500-650F	28.37				
136		650-850F	9.54				
137		850-975F	-0.40				
138	Total	64.20					
139							
140							

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CAER AC : ALC-1, CONDITION #5 (Period 25)

	1	2	3	4	5	6	7	8
		Wt. % MF Coal	C	H	O	N	S	SO3-Free Ash
141								
142	Gross SOH	39.25	86.74	12.79	0.25	0.11	0.11	0.00
143	- C9H19 from TNPS	5.32	84.95	15.05	0.00	0.00	0.00	0.00
144	Net SOH	33.93	87.02	12.44	0.29	0.13	0.13	0.00
145								
146	VSb	4.52	89.70	5.51	3.12	1.41	0.26	0.00
147	- 975+ in VSb	4.42	89.69	5.50	3.13	1.42	0.26	0.00
148	975- in VSb	0.10	90.12	5.93	2.70	0.99	0.26	0.00
149								
150	VSOH-1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
151	975F- VSOH	7.28	89.51	8.68	1.13	0.53	0.15	0.00
152	975 F- in VSb	0.06	90.12	5.93	2.70	0.99	0.26	0.00
153	975 - in PFC	7.33	89.51	8.66	1.14	0.53	0.15	0.00
154								
155	Agglomerating Oil	0.00	0.00	0.00	0.00	0.00	0.00	0.00
156	- 975+ in Agglom Oil	0.00	0.00	0.00	0.00	0.00	0.00	0.00
157	975- in Agglom Oil	0.00	0.00	0.00	0.00	0.00	0.00	0.00
158								
159	C4 to 975F Distillate from Liquef'n							
160	• C4	1.91	82.66	17.34	0.00	0.00	0.00	0.00
161	• C5+ Gas	2.18	83.81	16.19	0.00	0.00	0.00	0.00
162	• Net SOH Oil	33.93	87.02	12.44	0.29	0.13	0.13	0.00
163	Recycle DW-HT(P23)	-56.53	90.20	9.34	0.11	0.23	0.12	0.00
164	975F- VSOH	102.41	89.51	8.68	1.13	0.53	0.15	0.00
165	• 975F- in VSb	0.10	90.12	5.93	2.70	0.99	0.26	0.00
166	• 975F- in PFC	7.33	89.51	8.66	1.14	0.53	0.15	0.00
167	Prepared DW-HT	-30.66	88.10	8.47	2.02	0.65	0.76	0.00
168	TOTAL C4 to 975F	60.67	87.77	10.81	1.09	0.49	-0.15	0.00

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ATTACHMENT #2

**ESTIMATED MATERIAL BALANCE FOR
DISTILLATE SOLVENT DEWAXING &
HYDROTREATING - CONDITION #5**

1063

CAER AC : ALC-1, CONDITION #5 (Period 25)

	1	2	3	4	5	6	7	8
169								
170	DEWAXING &	HYDROTREATG	BALANCE					
171		Wt.% of						
172		975F- VSOH	NET YIELD					
173	INPUT	from Liquef'n	Wt.% MF Coal					
174	975F- VSOH from Liq.	100.00	102.41	C	H	O	N	S
175				89.51	8.68	1.13	0.53	0.15
176	MEASURED OUTPUTS							
177	DW-HT VSOH-1	28.86	29.55	88.99	10.74	0.00	0.16	0.12
178	DW-HT VSOH-2	66.05	67.64	89.76	9.30	0.62	0.21	0.12
179	Unrefined Wax	4.37	4.48	85.86	13.22	0.59	0.21	0.12
180	Total Measured	99.28	101.67	88.72	9.82	0.43	0.19	0.11
181			IN - OUT	0.79		0.70	0.34	0.04
182	NON-MEASD OUTPUTS							
183	C1-C3	0.40	0.41	78.82	21.18	0.00	0.00	0.00
184	C4	0.10	0.10	82.66	17.34	0.00	0.00	0.00
185	C5+ Gas	0.19	0.19	83.81	16.19	0.00	0.00	0.00
186	IBP-350F	0.28	0.29	85.00	15.00	0.00	0.00	0.00
187	H2O	0.78	0.80	0.00	11.19	88.81	0.00	0.00
188	NH3	0.41	0.42	0.00	17.76	0.00	82.24	0.00
189	H2S	0.04	0.04	0.00	5.92	0.00	0.00	94.08
190	Total Meas'd+Non-Me	101.48	103.92	89.51	10.16	1.13	0.53	0.15
191	Hydrogen Consumed	1.48	1.51		1.48			
192	Non-Meas'd Carb. Split	%						
193	C1-C3	40.00						
194	C4	10.00						
195	C5+ Gas	20.00						
196	IBP-350F	30.00						

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CAER AC : ALC-1, CONDITION #5 (Period 25)

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	1	2	3	4	5	6	7	8
197	C4-975F							
198	DEWAXING & HT CUTS							
199	OUT	Wt. % MF Coal	IBP-350F	350-500F	500-650F	650-850F	850-975F	
200	• DW-HT VSOH-1	29.55	0.00	13.20	52.50	32.30	2.00	
201	• DW-HT VSOH-2	67.64	0.00	0.00	9.70	81.80	8.50	
202	• Unrefined Wax	4.48	0.00	0.00	19.10	72.40	8.50	
203	• C4	0.10	100.00	0.00	0.00	0.00	0.00	
204	• C5+ Gas	0.19	100.00	0.00	0.00	0.00	0.00	
205	• IBP-350F	0.29	100.00	0.00	0.00	0.00	0.00	
206	TOTAL OUT	102.25	0.58	3.90	22.93	68.12	6.72	102.25
207	- IN	102.41	0.00	2.56	19.05	72.30	8.50	102.41
208	NET DW & HT	-0.16	0.58	1.34	3.88	-4.18	-1.78	-0.16
209	+ LIQUEFACTION	60.67	14.21	11.02	26.81	9.01	-0.38	60.67
210	TOTAL LIQ'N+DW+HT	60.51	14.78	12.36	30.69	4.83	-2.16	60.51
211								
212	Total, Wt. % MAF Coal	64.03	15.65	13.08	32.48	5.11	-2.28	64.03
213								
214	Total Wax, Wt. % MAF	59.30	15.65	13.08	31.57	1.68	-2.69	59.30
215	Wt. % MF Coal		C	H	O	N	S	
216	• DW-HT VSOH-1	29.55	88.99	10.74	0.00	0.16	0.12	
217	• DW-HT VSOH-2	67.64	89.76	9.30	0.62	0.21	0.12	
218	• Unrefined Wax	4.48	85.86	13.22	0.59	0.21	0.12	
219	• C4	0.10	82.66	17.34	0.00	0.00	0.00	
220	• C5+ Gas	0.19	83.81	16.19	0.00	0.00	0.00	
221	• IBP-350F	0.29	85.00	15.00	0.00	0.00	0.00	
222	Total DW+HT	102.25	89.33	9.92	0.44	0.19	0.12	100.00
223								
224								

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CAER AC : ALC-1, CONDITION #5 (Period 25)

	1	2	3	4	5	6	7	8
225								
226								
227	NET YIELDS	LIQUEFACTION	Wt.% MF Coal DEWAX & HT	TOTAL		Wt.% MAF Coal TOTAL		
228	H2O	12.00	0.80	12.81		13.55		
229	H2S	-0.77	0.04	-0.73		-0.77		
230	NH3	0.60	0.42	1.02		1.08		
231	O	1.69	0.00	1.69		1.78		
232	CO2	4.80	0.00	4.80		5.08	C4-975F	
233	C1-C3	9.90	0.41	10.31		10.92	+Wax	
234	C4-975F	60.67	-4.64	56.04		59.30	64.03	
235	Unrefined Wax	0.00	4.48	4.48		4.74		
236	Solids Free 975F+	6.73	0.00	6.73		7.12		
237	IOM	4.77	0.00	4.77		5.05		
238	SO3-Free Ash	6.51	0.00	6.51	Sub-Total	107.84		
239	MOLYBANA	-0.02	0.00	-0.02				
240	Fe2O3	-1.00	0.00	-1.00				
241	Total	105.89	1.51	107.40				
242	H2CONSUMED	5.89	1.51	7.40		7.83		
243	TOTAL C4-975F DIST.	Wt.% MF Coal	C	H	O	N	S	
244	• C4	1.91	82.66	17.34	0.00	0.00	0.00	
245	• C5+ Gas	2.18	83.81	16.19	0.00	0.00	0.00	
246	• Net SOH Oil	33.93	87.02	12.44	0.29	0.13	0.13	
247	Recycle DW-HT(P23)	-56.53	90.20	9.34	0.11	0.23	0.12	
248	• DW+HT C4-975F	102.25	89.33	9.92	0.44	0.19	0.12	
249	• 975F- in VSB	0.10	90.12	5.93	2.70	0.99	0.26	
250	• 975F- in PFC	7.33	89.51	8.66	1.14	0.53	0.15	
251	Prepared DW-HT	-30.66	88.10	8.47	2.02	0.65	0.76	
252	TOTAL C4 to 975F	60.51	87.23	12.88	-0.09	-0.08	-0.21	

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ATTACHMENT #3

**PRELIMINARY ANALYSIS OF
BENCH SCALE RUN ALC-1**

**BY LDP ASSOCIATES
PRESENTED @ JUNE 1996 ADVANCED CONCEPTS PROJECT REVIEW
MEETING @ LAWRENCEVILLE NEW JERSEY
DOE CONTRACT DE-AC22-91PC91040**

TABLE 1 ALC-1: SUMMARY OF LDP MATERIAL BALANCE RESULTS

	CONDITION #1	CONDITION #2	CONDITION #3	CONDITION #4	CONDITION #5
	PERIOD #6	PERIOD #13	PERIOD #17	PERIOD #20	PERIOD #25
CATALYST RATE	100%	100%	70%	70%	70%
REACTOR SV	42	35	34	26	29
AS-IS BALANCES					
• ASH	96.8	98.3	98.6	101.7	92.5
• CARBON	98.5	100.3	97.1	98.0	97.7
MAF COAL CONVN	95.2	97.7	97.4	97.3	94.7
NET YIELDS w%MAF					I/C DW & HT
• COx	5.9	2.8	4.5	4.3	5.9
• C1-C3	10.1	12.5	11.6	13.0	10.9
• C4-975F	67.0	55.2	60.2	64.5	64.0
• SF 975+	5.6	16.0	11.0	5.0	7.1
• QI	12.1	7.3	7.1	7.3	11.9
• H2 CONSUMED	7.0	6.7	6.8	7.4	7.8
• NET 650-850F	16.7	12.5	15.7	16.7	5.1
• NET 850-975F	0.5	-1.7	-4.7	-7.3	-2.3
% SOLIDS IN 975F+	68.5	31.4	39.2	59.5	62.6
EST'D NET C4-975F					
• % H	11.4	11.5	11.5	11.8	12.9
• % O+N+S	1.3	2.9	2.4	1.9	0.0

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TABLE 2
CAER ADVANCED CONCEPTS PROGRAM
BENCH RUN ALC-1
USE OF DI-TERTIARY-NONYL POLYSULFIDE (TNPS)

CHEMICAL COMPOSITION: $C_9H_{19} - S_x - C_9H_{19}$ (37% Sulfur, Min.)

USAGE RATE: APPROX. 8.1% ON MF COAL NEEDED TO
ACHIEVE TARGETED 3.0% SULFUR ON MF COAL

ACCOUNTING FOR TNPS IN MATERIAL BALANCES:

- Assumed Reaction:



2.82 lb H added/100lb TNPS

0.23 lb H added/100lb MF Coal

- Assumed C_9H_{20} Product (5% on MF Coal) Subtracted From SOH Oil

CONCLUSION: USE OF TNPS MAY HAVE SIGNIFICANT IMPACT ON

NET YIELDS OF HYDROCARBON GASES &

IBP-350°F FRACTIONS

RECOMMENDATION: ELIMINATE/REDUCE USE OF TNPS

- Options:

1. Install 2nd H_2S System

2. Use DMDS

3. Don't Use Interstage Separator: Adjust H_2 Feed Rate To Maintain
Desired H_2 Partial Pressure

4. Eliminate or Significantly Reduce Sulfur Addition to 2nd Stage



TECHNICAL DATA

ATOCHEM NORTH AMERICA, INC. • Organic Chemicals Division • Three Parkway • Philadelphia • Pennsylvania 19102
01490

DI-TERTIARY-NONYL POLYSULFIDE (TNPS) $C_9H_{19}S_xC_9H_{19}$ Isomers

SPECIFICATIONS

Form	Liquid
Color and Appearance.	Bright Yellow
ASTM D-1500	Maximum 3
Total Sulfur	Minimum 37.0% ←
Ash	Maximum 0.01%
Hydrogen Sulfide Test (Lead Acetate)	Negative
Odor	Comparable to or less Offensive than Standard

The above specifications are determined according to Atochem analytical methods.

PROPERTIES

Flash Point, Cleveland Open Cup (°F)	Minimum 300
Flash Point, Pensky Martens Closed Cup (°F)	250
Refractive Index	1.540 - 1.560
Average Weight, Pounds Per Gallon	8.65
Coefficient of Cubical Expansion/°C.	0.00058
Fire Point - Open Cup (°F)	350
Viscosity at 210°F SUS	40 - 48
Specific Gravity at 60/60°F	1.026 - 1.045

SHIPPING

Samples and Drums, Cans: Lubricating Oil, NOI, other than petroleum.
Tank Trucks: Lubricating Oil Additive.
Tank Cars: Lubricating Oil Additive.
Iso Tank: Lubricating Oil Additive.

MISCELLANEOUS

CAS No.: 68425-16-1*

* CAS Number assigned for EPA use in TSCA Chemical Substance Inventory.

(Over)

HEADQUARTERS
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Technical Service 215-587-7710
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CANADA
P.O. Box 278
Oakville, Ontario
Canada, L6J5A3
416-827-9841
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TABLE 3
CAER ADVANCED CONCEPTS PROGRAM
BENCH RUN ALC-1
VARIABILITY OF SECOND STAGE GAS YIELDS

SECOND STAGE GAS COMES FROM TWO SOURCES

TWO GC RESULTS TAKEN DURING EACH OPERATING PERIOD

OFTEN THESE TWO RESULTS DIFFER SIGNIFICANTLY (e.g. Period 17)

**HYDROCARBON GAS YIELD CAN BE SIGNIFICANTLY AFFECTED BY
CHOICE OF GC RESULT USED IN MATERIAL BALANCE CALCULATION**

**RECOMMENDATION: SEPARATELY MEASURE AND ANALYZE EACH
SOURCE OF GAS FROM SECOND STAGE**

HYDROCARBON RESEARCH INC.
ANALYTICAL LABORATORY

HEWLETT-PACKARD GAS ANALYSIS
Model 5890A

Workup Period Card #3

UNIT: 227 RUN: 94 PERIOD NO: 17 DATE: 05/06/96

Component-Type	DATA WITH N2 & O2			DATA WITHOUT N2 & O2		
	VOL% B/OVHD 1ST	VOL% A/BTMS 2ND	VOL% B/BTMS 2ND	VOL% B/OVHD	VOL% A/BTMS	VOL% B/BTMS
Hydrogen	90.750	75.810	88.040	91.500	86.502	92.450
Methane	2.110	2.880	1.760	2.127	3.286	1.848
Ethylene	0.030	0.000	0.000	0.030	0.000	0.000
Ethane	0.940	1.390	0.850	0.948	1.586	0.893
Propene	0.080	0.000	0.000	0.081	0.000	0.000
n-Propane	0.750	1.430	0.850	0.756	1.632	0.893
Butenes	0.070	0.000	0.000	0.071	0.000	0.000
n-Butane	0.290	0.530	0.310	0.292	0.605	0.326
i-Butane	0.000	0.000	0.000	0.000	0.000	0.000
Pentenes	0.000	0.000	0.000	0.000	0.000	0.000
n-Pentane	0.110	0.160	0.100	0.111	0.183	0.105
i-Pentane	0.070	0.090	0.050	0.071	0.103	0.053
MeCyclo C5	0.010	0.000	0.000	0.010	0.000	0.000
Cyclo C6	0.010	0.250	0.060	0.010	0.285	0.063
n-Hexane	0.040	0.100	0.030	0.040	0.114	0.032
Me-Pentane	0.020	0.010	0.000	0.020	0.011	0.000
C6 and C7	0.000	0.000	0.000	0.000	0.000	0.000
CO	0.480	0.020	0.020	0.484	0.023	0.021
CO2	1.240	0.180	0.070	1.250	0.205	0.074
H2S	2.180	4.790	3.090	2.198	5.466	3.245
Nitrogen	0.650	12.100	4.550	----	----	----
Oxygen	0.170	0.260	0.220	----	----	----
Total	100.000	100.000	100.000	100.000	100.000	100.000
Total C1-C3	3.910	5.700	3.460	3.942	6.504	3.633
Total C4-C7	0.620	1.140	0.550	0.625	1.301	0.578
Total CO+CO2	1.720	0.200	0.090	1.734	0.228	0.095
S	2.180	4.790	3.090	2.198	5.466	3.245
Calculated molecular wt	4.889	9.022	5.487	4.691	6.332	4.349

TABLE 4
CAER ADVANCED CONCEPTS PROGRAM
BENCH RUN ALC-1
COMMENTS ON MATERIAL BALANCE CONDITION #1 (Period #6)

- SOH OIL RATE 50% HIGHER IN PERIOD#6 VS PERIOD #5.
- MAIN RECYCLE STREAM IS PFL WITH UNDESIRABLY LOW
425°F IBP.
- YIELD OF SOLIDS FREE 975°F+ IS VERY SENSITIVE TO SF 975°F+
CONTENT OF VSB. VSB COMES FROM TWO SOURCES
(Toluene Solubles from PFC Extraction & Topped PFL).
- WATER YIELD & HYDROGEN CONSUMPTION MAY BE HIGHER THAN
INDICATED DUE TO EXTREMELY HIGH 15.3% OXYGEN (By Diff.)
in PFC.

Table 5

Run ALC-1 (227-94) Condition 1 Preliminary Results

DATE PERIOD Coal Type or Bag #'s	19-Apr-96 1 Raw Coal	20-Apr-96 2 Raw Coal	21-Apr-96 3 Raw Coal	22-Apr-96 4 Raw Coal	23-Apr-96 5 Raw Coal	24-Apr-96 6 Raw Coal
FEEDS, gms						
COAL: Black Thunder	25845	33810	34770	35562	31386	35303
Was Old Agg Oil						
Wash Oil						
FF4						
FF5						
Molyvan - A	10.12	10.63	10.89	11.15	9.92	11.13
Fe - based Catalyst	2947	3097	3172	3249	2890	3242
H2S	915	908	887	903	889	1012
TNPS	2469	2667	2661	2555	2659	2723
Make-up Oil	1787	5399	6689			
VSB				2788	10253	9215
VSOH2						
VSOH1						
975F-VSOH						
ASOH						
INTERSTAGE						
SOH - OIL						
SOH - H2O						
PFL	53280	41793	42107	46626	30107	35020
PFC		2288	1904	2756	6726	8638
Water	9269	8508	8462	8567	8778	8916
Hydrogen Feed	7072	7067	7073	7068	7055	7062
TOTAL FEED	103594	105547	107736	110084	100753	111142
Products, gms						
Hydrogen in Product Gases	6076	4238	4440	4867	4596	4653
Product Gas (hydrogen & nitrogen free)	8399	8206	6936	6472	7901	6285
VSB						
VSOH2						
VSOH1						
975F-VSOH						
ASOH		4143	13929	6077	1428	
INTERSTAGE						
SOH - OIL	10444	15769	9268	8823	9035	14794
SOH - H2O	14265	19711	20138	26114	20873	18701
PFL	61620	44801	44496	45952	43402	50113
PFC	2602	4760	4357	5439	10349	13215
TOTAL PRODUCTS	103406	101629	103564	103744	97584	107761
RECOVERY, W%	99.82	96.29	96.13	94.24	96.86	96.96
FEED STREAM RATIO's (to MF Coal)						
PFL+PFC+VSB+VSOH+Makeup+Agg. Oil	2.32	1.60	1.60	1.61	1.63	1.63
IOM & ASH in PFC FEED, %		6.08	5.20	6.50	19.07	22.71
Metered H2 Cons., W% maf coal	4.46	9.77	8.85	7.22	9.07	7.92
UN-NORMALIZED GAS YIELDS, W% maf co						
K-1 & K-2 : C1-C3 Gas Yield	15.72	15.63	11.94	10.97	14.74	9.94
K-1 & K-2 : C4-C7 Gas Yield	10.78	5.70	5.44	5.05	6.40	4.88
K-1 & K-2 : COx Gas Yield	11.15	7.02	5.94	5.21	8.00	5.84
K-1 : C1-C3 Gas Yield	7.19	5.19	4.58	4.83	5.72	4.84
K-1 : C4-C7 Gas Yield	4.65	1.92	1.70	3.19	3.39	2.84
K-1 : COx Gas Yield	10.24	5.95	5.11	4.72	7.44	5.32
Dry Coal Feed, gms	23762	30845	31697	32468	28884	32405
Calculated Coal Moisture Content, W%	8.06	8.77	8.84	8.70	7.97	8.21
Coal conversion, SO3 free		93.38	93.99	95.14	95.44	94.96

Figure 3
DOWNSTREAM PRODUCT FLOW SCHEME
Condition 1

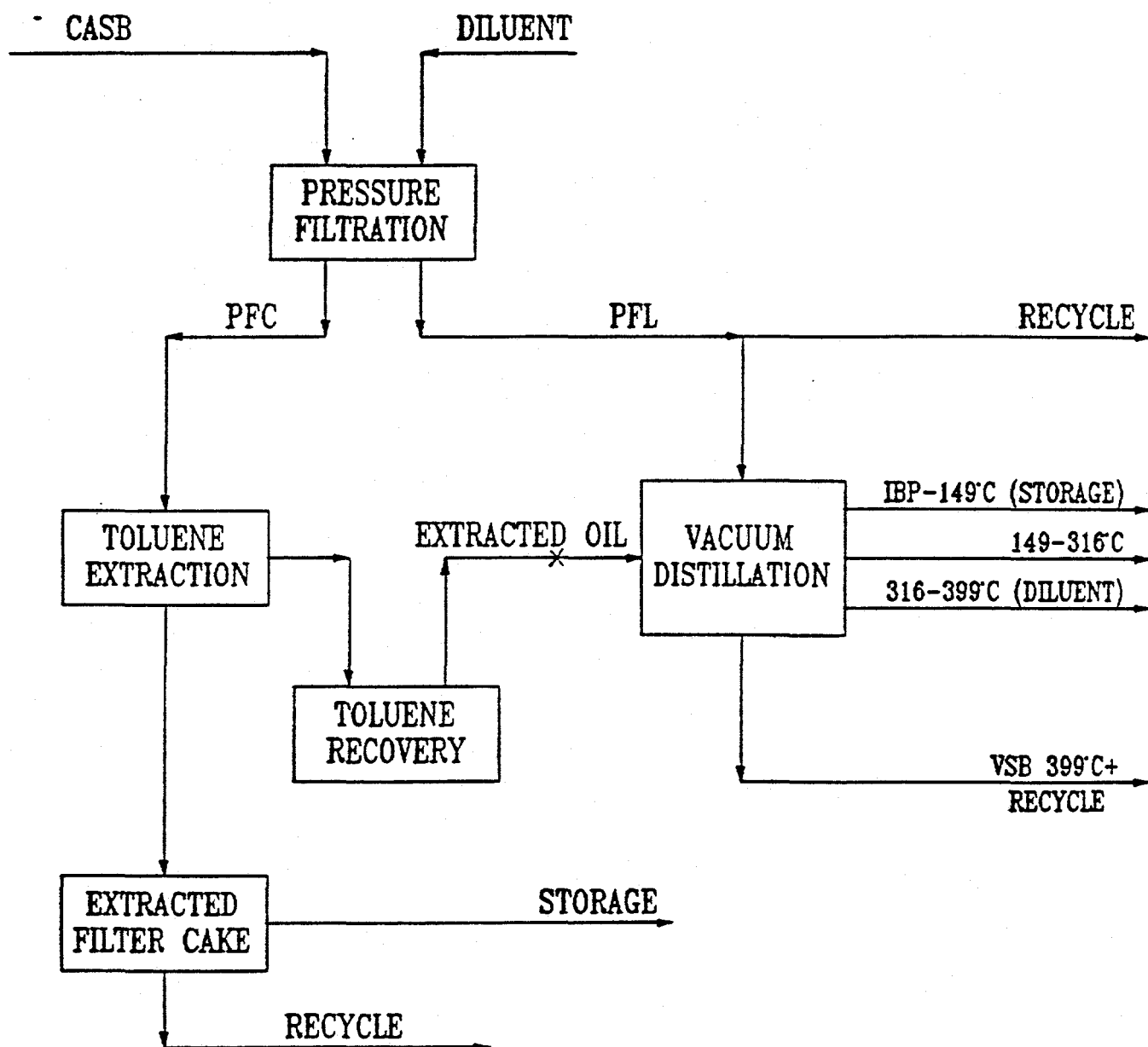


TABLE 5
CAER ADVANCED CONCEPTS PROGRAM
BENCH RUN ALC-1
COMMENTS ON MATERIAL BALANCE
CONDITIONS #2, #3 & #4 (Periods #13, #17 & #20)

- HYDROGEN CONTENT OF AGGLOMERATED COAL HIGHER THAN CALCULATED FROM RAW COAL AND AGGLOMERATING OIL.
- EFFECT OF EXTERNAL AGGLOMERATING OIL ON YIELDS UNCERTAIN
- UNABLE TO REMOVE EQUIVALENT AGGLOMERATING OIL BOILING RANGE MATERIAL FROM SYSTEM DUE TO FRONT END MIXING CONSTRAINTS.
- NEGATIVE 850-975°F YIELD FOR ALL THREE CONDITIONS
- COAL CONVERSION:
 - 2 TO 3% HIGHER THAN WITH REGULAR COAL
 - NO SIGNIFICANT EFFECT OF REDUCED CATALYST RATE OR REDUCED SPACE VELOCITY
- SURPRISING COMPARATIVE RESULTS BETWEEN CONDITIONS #2 & #3. DESPITE HIGHER CATALYST FEED RATE CONDITION #2 HAD LOWER DISTILLATE AND HIGHER SF 975°F+ YIELDS. THESE ANOMALOUS DIFFERENCES MAY BE DUE TO UPSET IN VACUUM STILL OPERATION PRIOR TO PERIOD #13.
- YIELD TRENDS AS EXPECTED WHEN SPACE VELOCITY LOWERED (CONDITION #4 VS #3).
- DISTILLATE PRODUCT QUALITY ALSO IMPROVED AT LOWER SV.
- CO_x YIELD IN CONDITION #2 (Period #13) IS SIGNIFICANTLY LOWER THAN IN PRECEDING PERIODS OF SAME CONDITION.

Table 6

Run ALC-1 (227-94) Condition 2 Preliminary Results

DATE PERIOD Coal Type or Bag #s	25-Apr-96 7 BAG C2	26-Apr-96 8 BAG C2 & C3	27-Apr-96 9 BAG C3 & C4	28-Apr-96 10 BAG C4 & C5	29-Apr-96 11 BAG C5	30-Apr-96 12 BAG C5 & C6	01-May-96 13 BAG C6 & C7
FEEDS, gms							
COAL: Black Thunder	33715	33417	29040	28109	25208	27280	27998
Was Old Agg Oil							
Wash Oil				4892	1462		
FF4							
FF5							
Molyvan - A	10.57	10.53	9.40	9.00	8.10	8.81	9.20
Fe - based Catalyst	3079	3064	2699	2585	2324	2530	2644
H2S	828	981	960	915	847	908	872
TNPS	2639	2586	2442	2120	2107	2048	2129
Agglomerating Oil	10778	10531	9213	8825	7885	8593	9109
VS8	1614	3943	22676	21720	18524	20988	21897
VSOH2	25572		2437	2626	9815	3554	3074
VSOH1							
975F-VSOH							
ASOH							
INTERSTAGE							
SOH - OIL							
SOH - H2O							
PFL	4156	22504					
PFC	9243	12103	9157	8771	7884	8584	9084
Water	6648	6154	7241	7123	6915	7014	7651
Hydrogen Feed	7074	7076	7071	7061	7071	7074	7073
TOTAL FEED	105356	102369	82945	94757	91050	88582	91540
Products, gms							
Hydrogen in Product Gases	4610	4597	4357	4695	4803	4743	4699
Product Gas (hydrogen & nitrogen free)	6169	5677	5727	5358	5756	5167	5342
VS8			33434	27309	26421	27825	32233
VSOH2			9170	14716	19894	12628	9715
VSOH1							
975F-VSOH							
ASOH							
INTERSTAGE	304						
SOH - OIL	10054	11801	10027	8465	8485	8538	9314
SOH - H2O	19183	19067	17460	18838	15593	15720	15822
PFL	52213	45516				0	
PFC	13040	15065	11986	11683	10402	10728	12537
TOTAL PRODUCTS	105574	101523	92161	91064	91354	85349	89661
RECOVERY, W%	100.21	99.17	99.16	96.10	100.33	96.35	97.95
FEED STREAM RATIO's (to MF Coal)							
PFL+PFC+VS8+VSOH+Makeup+Agg. Oil	1.63	1.60	1.60	1.60	1.94	1.65	1.61
DOM & ASH in PFC FEED, %	17.49	24.97	18.53	18.31	21.32	20.01	18.73
Metered H2 Cons., W% maf coal	8.10	8.36	10.36	9.34	10.12	9.54	9.17
UN-NORMALIZED GAS YIELDS, W% maf coal							
K-1 & K-2: C1-C3 Gas Yield	9.08	9.13	10.74	11.37	14.56	11.60	12.59
K-1 & K-2: C4-C7 Gas Yield	6.17	5.41	7.03	5.23	5.25	5.38	5.22
K-1 & K-2: COx Gas Yield	5.04	4.61	4.09	4.56	5.86	4.17	2.81
K-1: C1-C3 Gas Yield	4.99	4.95	4.66	5.71	6.08	5.44	4.07
K-1: C4-C7 Gas Yield	1.70	3.11	3.86	2.38	2.27	1.84	1.71
K-1: COx Gas Yield	4.62	4.20	3.72	4.18	5.33	3.77	2.06
Dry Coal Feed, gms	31435	30653	27100	26226	23206	25280	26786
Calculated Coal Moisture Content, W%	6.76	8.27	6.68	6.70	7.94	7.33	4.33
rsion, SO3 free	97.04	97.04	97.98	97.79	97.66	97.84	97.69

TABLE 6
CAER ADVANCED CONCEPTS PROGRAM
BENCH RUN ALC-1
COMMENTS ON MATERIAL BALANCE CONDITION #5 (Period #25)

- DUE TO LONG STARTUP TIME LAG & FILTRATION PROBLEMS, USE OF RECYCLED DEWAXED & HYDROTREATED (DWHT) SOLVENT WAS INFREQUENT.
- RECYCLED DWHT SOLVENT DIFFERED SIGNIFICANTLY FROM PREPARED (OLD) DWHT SOLVENT. EFFECT ON YIELDS UNCERTAIN.
- LONG TIME LAG FOR VSB ALSO DUE TO FILTRATION PROBLEMS.
- NO VSB WITHDRAWN FROM SYSTEM (Except for Samples), YET MATERIAL BALANCE SHOWS SIGNIFICANT POSITIVE VSB YIELD. INDICATED SF975F+ YIELD SHOULD BE LOWER.
- NO COAL CONVERSION IMPROVEMENT VS CONDITION #1.
- HIGHER HYDROGEN CONSUMPTION BUT SIGNIFICANTLY IMPROVED DISTILLATE PRODUCT QUALITY VS CONDITION #1.
- FULLY REFINED WAX YIELD WILL BE LOWER THAN "WAX" YIELD INDICATED AS ELEMENTAL ANALYSIS OF "WAX" INDICATES THAT THIS MATERIAL CONTAINS A SIGNIFICANT AMOUNT OF OIL.
- GREATER THAN 1% HYDROGEN ADDED TO C4-975°F PRODUCT IN HYDROTREATING. GAS MAKE APPEARS TO BE LOW & HETEROATOM REMOVAL IS SIGNIFICANT.

Figure 6
DOWNSTREAM PRODUCT FLOW SCHEME
 Condition 5

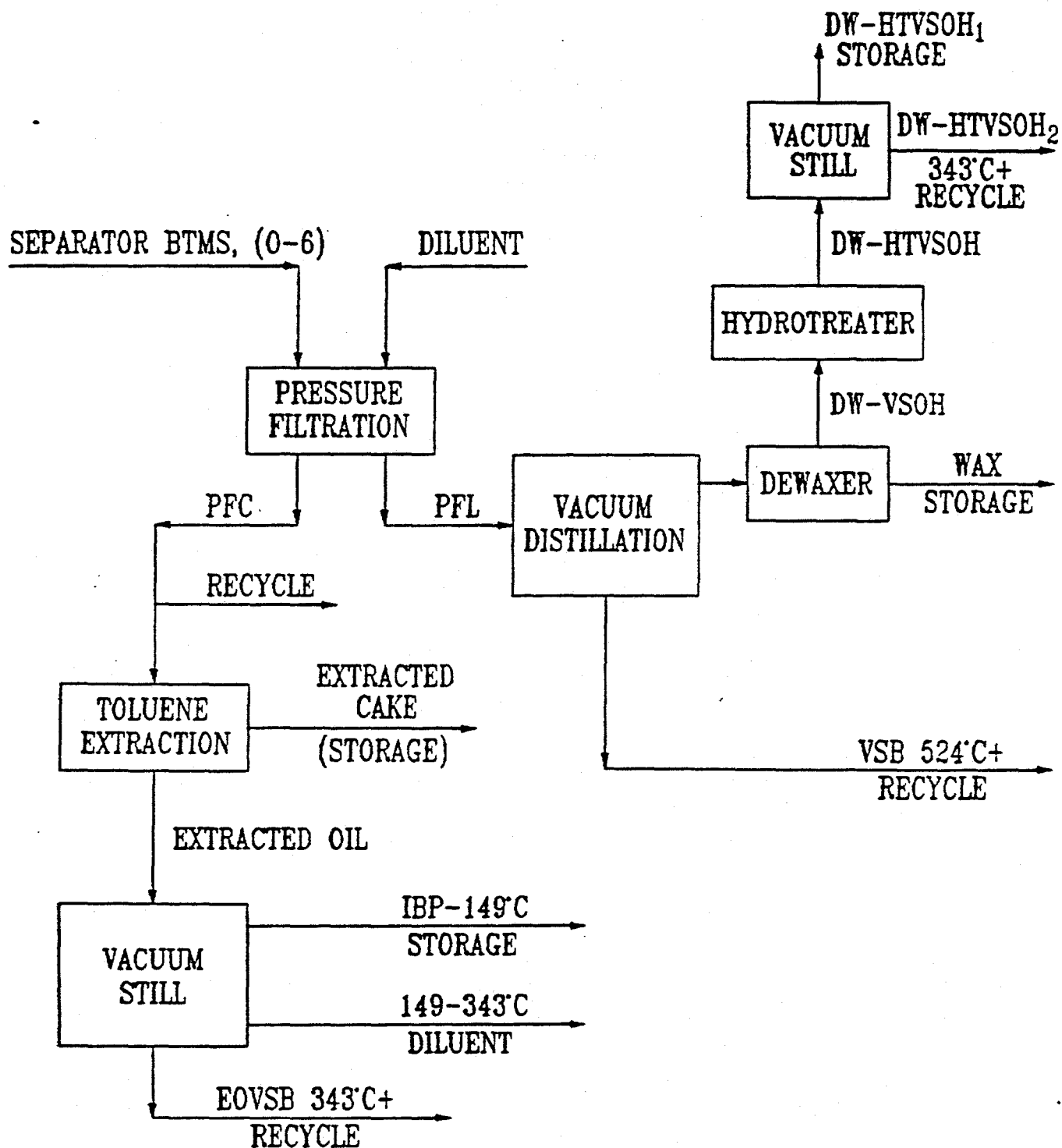


TABLE 7: ALC-1 DEWAXED-HYDROTREATED RECYCLE SOLVENT

ELEMENTALS	OLD 650F+DWHT	PERIOD #23	PERIOD #25
		VSOH-2	VSOH-2
CARBON	88.10	90.20	89.76
HYDROGEN	8.47	9.34	9.30
OXYGEN (Diff)	2.02	0.11	0.62
NITROGEN	0.65	0.23	0.21
SULFUR	0.76	0.12	0.12
CUTS			
500-650F	3.90	4.80	9.70
650-850F	87.70	82.70	81.80
850-975	8.40	12.50	8.50

6/17/96

TABLE 8
CAER ADVANCED CONCEPTS PROGRAM
BENCH RUN ALC-1
PLAN OF FUTURE WORK

- REFINE MATERIAL BALANCES
- PROJECT YIELDS AT STEADY STATE OPERATION
- PREPARE CONCEPTUAL COMMERCIAL PLANT DESIGN INFORMATION
- PERFORM ECONOMIC ANALYSIS OF COMMERCIAL PLANTS
- PREPARE RECOMMENDATIONS FOR FUTURE ALC BENCH RUNS

Table 9

Run ALC-1 (227-94) Condition 5 Preliminary Results

DATE PERIOD Coal Type or Bag #'s	09-May-96 21 Raw Coal	10-May-96 22 Raw Coal	11-May-96 23 Raw Coal	12-May-96 24 Raw Coal	13-May-96 25 Raw Coal
FEEDS, gms					
COAL: Black Thunder	21764	24596	25398	24964	23899
Was Old Agg Oil					
Wash Oil					
FF4					
FF5					
Molyvan - A	5.17	5.64	5.71	5.58	5.38
Fe - based Catalyst	1501	1637	1655	1618	1561
H2S	986	891	910	931	889
TNPS	1687	1857	1989	1877	1893
Agglomerating Oil					
VSF	10418	10337	7476	7493	6398
DW-HT VSOH2 (Old)	17237	19664	7094	16216	7138
DW-HT VSOH2 (Fresh)			14348	3243	12702
Wax					
DW-HT VSOH1					
DW-HT VSOH2					
SOH - OIL					
SOH - H2O					
PFL					
PFC	6875	7783	7871	7695	7420
Water	8335	8318	8107	7456	7649
Hydrogen Feed	7071	7073	7061	7067	7069
TOTAL FEED	75879	82162	81915	78566	76622
Products, gms					
Hydrogen in Product Gases	5200	5168	5193	5317	5260
Product Gas (hydrogen & nitrogen free)	4007	5203	5448	5527	4285
VSF	7456	8828	9365	8426	7259
DW-HT VSOH2 (Old)					
DW-HT VSOH2 (Fresh)					
Wax	1339	1274	1071	1518	986
DW-HT VSOH1	7176	4770	5536	6918	6519
DW-HT VSOH2	15408	16449	17300	13054	14919
SOH - OIL	8048	10262	8797	8970	9017
SOH - H2O	13837	14150	15234	14712	13051
PFL					
PFC	9260	12683	12391	11869	12307
TOTAL PRODUCTS	71731	78787	80335	76312	73602
RECOVERY, W%	94.53	95.89	98.07	97.13	96.06
FEED STREAM RATIO's (to MF Coal)					
PFL+PFC+VSF+VSOH+Makeup+Agg. Oil	1.64	1.64	1.56	1.50	1.51
IOM & ASH in PFC FEED, %	20.43	22.58	20.05	15.58	17.89
Metered H2 Cons., W% maf coal	9.46	8.82	8.42	8.06	8.62
UN-NORMALIZED GAS YIELDS, W% maf coal					
K-1 & K-2 : C1-C3 Gas Yield	10.49	12.85	12.55	13.51	9.87
K-1 & K-2 : C4-C7 Gas Yield	3.99	4.97	5.52	5.13	4.09
K-1 & K-2 : COx Gas Yield	5.79	6.27	6.47	6.83	6.46
K-1 : C1-C3 Gas Yield	7.24	5.96	6.53	5.86	6.05
K-1 : C4-C7 Gas Yield	2.36	2.20	2.23	2.07	2.45
K-1 : COx Gas Yield	5.64	5.89	6.14	6.27	6.31
Dry Coal Feed, gms	21061	23014	23643	23114	22362
Calculated Coal Moisture Content, W%	3.23	6.43	6.91	7.41	6.43
Coal conversion, SO3 free	96.09	95.48	96.26	96.64	94.72

Table 15. Properties of Wax Product

Periods	22	23	24	25
Elemental Analysis				
Carbon, W%	86.75	84.28	81.47	85.86
Hydrogen, W%	12.22	12.66	13.09	13.22
Sulfur, W%	0.328	0.367	0.873	0.309
Nitrogen, W%	0.32	0.25	0.20	0.21
H/C Ratio	1.69	1.80	1.93	1.85
Product Distrubution by (GC&GC-MS), W%				
C ₁₇ -C ₂₂				24.8
C ₂₃ -C ₃₀				71.9
C ₃₁ -C ₃₆				3.3
524 °C+ Resid Content, W%	1.49	0.28	4.39	3.31