CONTRACT TITLE AND NUMBER:

Exploratory Research on Novel Coal Liquefaction Concept
DOE Contract No. DE-AC22-95PC95050

CONTRACTOR NAME:

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

CONTRACT INITIATION DATE: May 24, 1995

ANTICIPATED COMPLETION DATE: May 23, 1998

GOVERNMENT AWARD: \$1,314,477 Total, \$940,000 Obligated

PRINCIPAL INVESTIGATORS: R. A. Winschel, S. D. Brandes

SUBCONTRACTORS/PRINCIPAL INVESTIGATORS:

- University of Kentucky Center for Applied Energy Research - F. J. Derbyshire, G. M. Kimber, R. K. Anderson, A. Vego, T. Rantell, D. Jacques
- LDP Associates - M. Peluso

CONTRACTING OFFICER'S REPRESENTATIVE: M. A. Nowak

REPORTING PERIOD: January 1 through March 31, 1997

CONTRACT OBJECTIVES: No Change

SUMMARY OF TECHNICAL PROGRESS - OVERALL

Microautoclave tests confirmed that first-stage subbituminous coal conversions were greater in a more aromatic first-stage solvent. First-stage liquefaction tests with hydride ion "E" showed that high coal conversions can be obtained with a number of different first-stage water-gas-shift catalysts. Eight one-liter autoclave tests were completed. All tests used Black Thunder Mine subbituminous coal and Reilly Industries anthracene oil. Differences among the tests were the hydride ion reagent used, the post-run flash of water, and the shift catalyst. Filtration tests were conducted with five one-liter autoclave products of subbituminous coal. The filtration rates were slower than those that had been obtained with North Dakota lignite products, but were still within a commercially acceptable range. The influence of the first-stage shift catalyst on filtration rates is being investigated. Second-stage hydrotreating of products of tests made to simulate the

British coal LSE process and the Wilsonville pilot plant preheaters had lower resid conversion and higher hydrogen uptake than the products of the hydride ion liquefaction reaction. The 300 mL second-stage reactor system went on line this quarter. Refinements in the experimental procedures are under way. A conceptual commercial plant design for the hydride ion reagent "A" case was completed. Evaluations of hydride ion reagent "D" and "E" cases were initiated, and an integrated liquefaction system balance for the hydride ion reagent "E" case was begun. A preliminary review of the final technical and economic reports from the Alberta Research Council study of low-rank coal conversion using the CO-steam process generated a number of questions on the published reports; further analysis of the reports is planned.

SUMMARY OF TECHNICAL PROGRESS BY TASK

Task 3 - Flow Sheet Development

First-Stage Solubilization

45 mL Microautoclave Tests

The effects of first-stage solvent on coal conversion were further explored this quarter. Coal conversions are higher for microautoclave tests made when Reilly Industries anthracene oil is used instead of Kawasaki Steel Corp. anthracene oil, at otherwise the same test conditions (Table 1). With Glenharold Mine lignite, an improvement of greater than 20 wt % was obtained in some tests. With Black Thunder Mine subbituminous coal, the coal conversion improved up to 10 wt %. The Reilly Industries anthracene oil has a higher aromatic hydrogen content than the Kawasaki Steel Corp. oil. As described previously, the solubility of the subbituminous coal products in the first-stage solvent appears to be greater in a more aromatic solvent.

A microautoclave-scale mass and elemental balance test (Run 217a) was completed. Test conditions were Black Thunder Mine coal, Reilly Industries anthracene oil, 375 °C, 60 min, hydride ion reagent D (HI "D"), and 500 mmoles/kg coal NaAlO₂. Mass recovery was 99.1 wt %.

Microautoclave tests were completed using several different candidate water-gas-shift catalysts. A series of baseline tests also was made. Conditions for tests with catalyst were: 375 °C, 60 min, HI "D", and 500 mmoles catalyst/kg dry coal. All tests were made with Black Thunder Mine coal (ca. 22 wt % moisture) and Reilly Industries anthracene oil. Coal conversions obtained with NaAlO₂, KOH, and Na₂CO₃ were 89.5, 90.2, and 88.8 wt %, respectively. Conversions with NaOH and K₂CO₃ were 75.8 wt % and 83.9 wt %, respectively. A duplicate microautoclave test made with NaOH confirmed the lower coal conversion. Duplicate tests were made without

catalyst at the conditions given above (Runs 210 and 223). Coal conversions were 72.5 wt % and 72.6 wt %. A test made with CO and coal (Run 224) had a coal conversion of 70.2 wt %. The addition of catalyst (Run 225a) improved the conversion to 83.6 wt %. Substituting hydride ion reagent E (HI "E") for HI "D" in a test made with no catalyst (Run 226) resulted in a coal conversion of 83.5 wt %. The addition of 500 mmol/kg dry coal of NaAlO_3 to the HI "E" system resulted in a coal conversion of 88.8 wt %.

One-Liter Autoclave

Eight one-liter autoclave tests, Runs 14-LA, 14b-LA, 15-LA, 15a-LA, 16-LA, 17-LA, 18-LA, and 18a-LA were completed. The test conditions were: Black Thunder Mine subbituminous coal, Reilly Industries anthracene oil, and a residence time of 60 min. at 375 °C. The test variables were: the hydride ion reagent, the post-run flash of water, and the shift catalyst.

Runs 14-LA and 14b-LA were made with hydride ion reagent "A". The products were flashed at 300 °C. As reported in the last quarterly report, work with HI "A" will be discontinued because of the high reagent cost. Run 14-LA and 14b-LA were made to provide sufficient material for filtration, second-stage upgrading tests, and product quality determinations to compare with other reagent systems. The 300 °C⁺ products were shipped to UK/CAER for filtration and second-stage upgrading tests (see below). Five-gram samples of 300 °C⁺ material retained from each test were pressure filtered. The coal conversion determined from these grab samples averaged 87 wt %. Gas analyses were obtained and are being examined.

The first one-liter autoclave test using hydride ion reagent "D" (HI "D") was Run 15-LA. NaAlO_2 catalyst was used. A replicate of Run 15-LA (15a-LA) was completed to provide additional sample for filtration and second-stage upgrading tests. Coal conversion for Run 15a-LA, determined from a five-gram grab sample was 83.6 wt %; the conversion was the same for Run 15-LA.

One-liter autoclave test Run 16-LA, the first test in the one-liter reactor to use HI "E", was made with NaAlO_2 catalyst. In this test, the flash at 300 °C was followed by a second flash at 200 °C. After the second flash, the pressure in the 1-L autoclave was reduced to atmospheric. Post-run reactor inspection revealed a blonde, crystalline, wax-like material coating the cooling coil. This coating was combined with the product remaining in the autoclave. The THF washings were stripped of THF. They were combined with the product prior to taking a five-gram grab sample

for pressure filtration to determine coal conversion. The remainder of the sample was sent to UK/CAER for filtration tests.

One-liter autoclave test Run 17-LA fed NaAlO_2 catalyst and HI "E" at double the ratio of HI "E" to dry coal that was used for Run 16-LA. Run 18-LA conditions were the same as Run 17-LA, except that Na_2CO_3 , a less costly catalyst, was used. A preliminary indication is that the filtering rate of the one-liter autoclave product of Run 18-LA was very slow. A duplicate one-liter autoclave test (Run 18a-LA) was made to produce more sample for filtration and second-stage upgrading tests. A portion of the product will be water washed to attempt to remove the catalyst and improve filtration. Results will be reported next quarter.

Large-Scale Filter

The large-scale (300 mL capacity) filter apparatus was received from the manufacturer. A standard operating procedure was drafted to integrate the filter with the one-liter reaction system. Installation of the filter on the one-liter unit was completed. During low-pressure testing of the filter system, the ball valves at the inlet and outlet of the filter did not seal. The valves were returned to the vendor for repair or replacement. Shake down runs are scheduled for next quarter.

Filtration Studies

Duplicate samples (Runs 14-LA and 14b-LA), prepared by the dissolution of Black Thunder Mine coal in Reilly Industries anthracene oil and hydride ion reagent "A" (HI"A") at 375 °C for 60 minutes, were analyzed. Coal conversions, based on the measurement of quinoline insolubles in the reactor product and both quinoline- and tetrahydrofuran (THF)-insolubles in the filter cakes, were almost identical and were between 93 and 95% (Table 2). Slightly lower conversions of ~87% were determined from the THF-solubles of the reactor product, indicating the presence of a small amount of material that is THF-insoluble but soluble in both quinoline and the process solvent.

Each sample of Run 14-LA and 14b-LA was split into two parts for filtration tests. Cake resistivities were significantly higher than those determined for the reactor products from previous tests using Glenharold Mine lignite. However, the measured flow rates of 40 to 100kg/h/m² still fall in the range of commercial acceptability (Table 3). The first sub-sample of Run 14-LA was filtered through glass fiber paper. Midway through this test, the differential pressure (ΔP) across the filter was increased. The increase in the cake resistivity (α) shows that

the filter cake is compressible; the value of the compressibility parameter ($n = 0.6$) is typical of coal residue solids ($n = 0.3$ to 0.7). The second sample was filtered using a $100\text{ }\mu\text{m}$ Conidur screen. Initially, a rapid filtration rate occurred. However, a subsequent refiltration of this filtrate through glass fiber showed that, on average, only about $\frac{1}{4}$ of the solids was retained on Conidur. During the next increase in pressure, the rate slowed in accordance with the expected cake build up, and the cake resistance was comparable with the first sub-sample run. When the pressure was increased by a factor of $2\frac{1}{2}$, the rate increased dramatically (by a factor of 10). Subsequent analysis of this sample showed that large quantities of solids were forced through the screen. This behavior and the relatively high cake resistivity are indicative of a small average size of the particles of ash and IOM. Bridging across the $100\text{ }\mu\text{m}$ orifices is marginal, but a reduction in the orifice size could make a significant improvement in bridging.

In the first test on the sub-sample from Run 14b-LA, using GFA glass fiber filter paper as the membrane, the differential pressure across the filter was periodically increased during the test. The results appear to indicate that α increases as the differential pressure is increased, again indicative of a compressible cake. However, the calculated value of the compressibility parameter, n , is greater than 1 ($n = 0$ for an incompressible cake). This suggests that α changes with time, but not solely as a function of ΔP . For example, the characteristics of the solids being deposited may change with time (very fine particles may be left suspended in the liquid phase after the coarser particles have settled to form the initial cake). A similar observation was made when the second part of the feed was filtered through a Conidur screen. In this test, using a Conidur screen with $60\text{ }\mu\text{m}$ apertures, bridging of the apertures appeared to occur satisfactorily despite the low solids content ($\sim 4\%$) and, possibly, the very small size of the particulates in the filter feed. However, at the end of the test (just prior to gas breakthrough), there was a significant increase in the filtration rate, suggesting that some solids were being carried through into the filtrate. This was confirmed from the measurement of the solids content of the filtrate.

Two samples of one-liter autoclave Run 15-LA reactor product were characterized. Both had been prepared from Black Thunder Mine coal and Reilly Industries anthracene oil at $375\text{ }^{\circ}\text{C}$ for 60 minutes. However, in Run 15-LA, hydride ion reagent "D" was used as the hydrogen transfer agent in place of HI"A" used previously, while in Run 16-LA, hydride ion reagent "E" was employed. A water gas shift catalyst, NaAlO_2 , was used in both tests. In order to generate sufficient material for second-stage hydrotreating, Run 15-LA was duplicated (Run 15a-LA) and the two products combined. Coal conversions for Run 15-LA, based upon the measurement of THF- and quinoline-insolubles of the reactor product and filter cakes, were in the range 86% to

92%; for Run 16-LA, they were generally in the range 91% to 94% (Table 1). However, in the latter material the conversion based upon THF solubility of the feedstock was only 78%. This indicates that, in this case, a significant amount of material was quinoline-soluble/THF-insoluble, but soluble in the process solvent. It will mainly report to the filtrate.

The large batch from Run 15-LA product was filtered through GFA paper in a 118 mm diameter unit operating at 250°C. The pressure was increased step-wise from 150 to 580 kPa during the test. Cake resistivities were very high and flow rates were low, ($<20 \text{ kg/m}^2/\text{h}$). The compressibility of the cake, like that from Run 14-LA, appeared to be >1 , again suggesting that a change in the character of the solids being deposited was occurring. To investigate the unusual filtration behavior of these materials, the product of Run 16-LA was well mixed before dividing it into four parts for filtration tests in the 35 mm diameter filter unit. The first batch filtered relatively easily through GFA at 265 °C and 570 kPa. The flow rate of $70 \text{ kg/m}^2/\text{h}$ was acceptable, although the cake resistivity (α) was relatively high. However, when the test was repeated using a step-wise increase in pressure from 135 to 560 kPa during the test, cake resistivities were significantly higher and flow rates much lower ($20 \text{ to } 24 \text{ kg/m}^2/\text{h}$). Duplication of the first test at 300°C and 570 kPa gave equally low rates of filtration ($\sim 16 \text{ kg/m}^2/\text{h}$). These inconsistent results may be associated with the feedstock character changing with time, or possibly to the nature of its physical properties and the handling it undergoes (the viscosity of non-Newtonian fluids is dependent upon the shear stress imposed, for example).

The last sub-sample of Run 16-LA was filtered using a Conidur screen with $60 \mu\text{m}$ apertures. Bridging of the apertures was only marginal as evidenced by sporadic increases in the flow rate imposed on the normal progressively reducing rate as cake builds and flow resistance increases. Three consecutive samples of filtrate were collected and analyzed for their solids content. The values obtained, 4%, 2% and 1%, confirm the above observations, indicating that the particulate bridges across the screen apertures were periodically collapsing to allow solids to enter the filtrate stream.

The causes for the substantially reduced flow rates and somewhat erratic behavior of these materials from Runs 15-LA and 16-LA during filtration are as yet unclear, but could possibly be attributed to two competing factors: (1) to the influence of the finely divided catalyst being deposited with the cake, or alternatively (2) to the use of the mild extraction conditions employed, which without the mitigating influence of HI"A", produces plastic, deformable residual particles that inhibit flow through the cake.

Second-Stage Hydrotreating

Microautoclave Runs

Microautoclave tests in which two grams of filtered resid were hydrotreated at 440 °C for 60 min, with 10,000 ppmw Mo (total solid reactor feed basis; Molyvan L catalyst precursor) were conducted. The experimental results are summarized in Table 4. Selected data are presented in Figure 1. In Table 4, data from first-stage one-liter autoclave tests that used Black Thunder Mine coal (Runs 14-LA, 14b-LA, 15-LA, and 15a-LA) are compared to tests that used Glenharold Mine lignite (Runs 9-LA through 13-LA). From those data, it would appear that the Glenharold Mine lignite-derived material, digested at milder temperatures and using HI"A" as the hydride ion source in the first stage, gave higher resid conversions. The tests with Black Thunder Mine coal show lower resid conversion, particularly with HI"D" substituted for HI"A" as the hydride ion source.

There is little difference among the resid conversions of tests with products of Runs 8-LA through 11-LA, in which only solvents and solvent-to-coal ratios varied (Table 4). In contrast, the resid conversions of Runs 8-LA through 11-LA are significantly higher than those of Runs 12-LA and 13-LA. Run 12-LA was made with a H-donor solvent (Lummus pasting solvent), a high temperature (410 °C) and long contact time (60 min). Run 12-LA was designed to simulate the British Coal LSE liquefaction process. Run 13-LA was made with a poor H-donor solvent (Wilsonville Run 263, V1067), and was designed to simulate the Wilsonville two-stage liquefaction pilot plant preheater conditions (410°C, 15 min, 2500 psi H₂ (hot). The resids generated in Runs 12-LA and 13-LA were less reactive than those produced in the hydride ion donation liquefaction system. The lower conversions and hydrogen uptake per unit resid conversion are more typical of the diluted feedstocks examined earlier in the program.

The boiling range distributions of the hydrotreated distillates from the hydrotreating runs described above are summarized in Table 5. The data presented are derived by simulated distillation of the micro-distilled product. Elemental analyses of some of the micro-distilled distillate and resid fractions are presented in Table 6. Hydrotreated distillates uniformly show 8-9% hydrogen content, while the matching resids have 4-5% hydrogen content. The three filtrate feedstocks shown in Table 5 contain about 5-6% hydrogen.

Equipment Installation and Testing

The high pressure, continuous flow hydrogen gas supply modifications to the 300 mL continuously stirred tank reactor (CSTR) were completed this quarter. This system can be charged with nominally 100 g, commensurate with the quantities of material produced in the one-liter first-stage autoclave. Shake-down tests of the CSTR were conducted. 100 g of Wilsonville Run 258A deashed resid was used as feedstock, along with ca. 12 g Molyvan L catalyst precursor. The reactor heat-up rate was slow, and hydrotreating temperature was not achieved after 4½ hours. To correct the slow heat-up, additional Fiberfrax insulation was applied to the vessel, and two 400W heat tapes were added. After these modifications, the autoclave heated from 20 °C to 440 °C in 2 hrs, 9 min, with a nearly linear heat up. A zero duration run will be made to determine the extent of conversion during the 30 min between 350 °C and 440°C.

An extension added to the stirrer shaft will allow a minimum feed charge of 34 mL. A 50 mL reactor charge should be ample to give stirring action through a one hour run (Table 7).

Task 5 - Engineering and Economic Study

Technical Evaluation of Hydride Ion Reagent "A" Case

The technical evaluation of a conceptual commercial plant based on Run 8-LA was completed (Confidential Appendix 2). Fuel gas requirements were identified, and the quantity of coal to be gasified was determined in order to close the make-up hydrogen, make-up carbon monoxide, and fuel gas balances. The required MF coal feed rate to gasification is approximately 40 pounds per 100 pounds of MF coal fed to liquefaction. No further work is planned on this case due to the adverse economic impact of the high consumption rate of hydride ion source "A".

Evaluation of First-Stage Microautoclave and One-Liter Autoclave Data

A material balance for microautoclave Run 217a (made with Black Thunder Mine coal, Reilly Industries anthracene oil and hydride ion reagent "D") was corrected for the Reilly oil elemental analysis. Several first-stage material balances were calculated using a variety of assumptions for closing the carbon balance. Light hydrocarbon gas yield was approximately 3 wt % on MF coal. Data from microautoclave Run 227a made with Black Thunder Mine coal, Reilly Industries anthracene oil, and hydride ion reagent E (HI"E") was evaluated. Light hydrocarbon gas yield was approximately 0.6 wt % on MF coal. This gas yield is much lower than the 3 wt % for Run 217a (made at the same reaction conditions, except with HI"D").

A gas yield was calculated for the one-liter autoclave test Run 15-LA made with HI"D". Gas analysis data indicated that the light hydrocarbon gas yield was approximately 3 wt %. One-liter

autoclave Run 16-LA was made with HI"E". Gas analysis indicate a 1.4% light hydrocarbon gas yield.

Gasification yields for the filter cake and unconverted second-stage resid also were determined for the higher first-stage CO₂ yield cases. An overall make-up CO, make-up hydrogen, and fuel gas balance was determined for the above cases. The effect of first-stage catalyst quantity on gasification efficiency also was determined.

Identification of Shift Catalyst Candidates

A short study was done to identify potential shift conversion catalysts whose costs would be substantially lower than the cost of NaAlO₂ (sodium aluminate), which was first tested for first-stage coal conversion. Potential suppliers were contacted to determine the cost of large quantities of candidate materials. It was determined that the use of Na₂CO₃ (soda ash) or NaOH (caustic soda) could potentially reduce shift catalyst cost to less than \$1 per barrel of product, a very significant savings vs the use of sodium aluminate. It was recommended that microautoclave tests be conducted with these materials to determine their effectiveness in shift and coal conversion.

Integrated Liquefaction System Balance for Hydride Ion Reagent "E" Case

An Integrated Liquefaction System Balance (ILSB) was developed using the first-stage gas and liquid yield data from microautoclave Run 227a. Due to the uncertainty of first-stage liquid yields, several cases were developed. Distillate product upgrading (to gasoline) balances were prepared for each of the cases, but the factors used for predicting hydrotreating/hydrocracking yields were found to be inconsistent with the analogous factors used in the upgrading balances for the Base Case (Wilsonville Run 263J). Therefore, the hydrotreating/hydrocracking yield factors were revised to be consistent with the analogous factors used in the upgrading balances for the base case. A second set of balances for the system was prepared to investigate the effect of a higher first stage CO₂ yield on process performance. A significant improvement in hydrogen consumption resulted. Gasification yields for the filter cake and unconverted second-stage resid were determined for higher first stage CO₂ yield cases. An overall make-up CO, make-up hydrogen and fuel gas balance was determined for one of the cases. The effect of reducing the first stage catalyst quantity on gasification efficiency was found to be significant.

Literature Review

A review was initiated of the final technical and economic reports from the Alberta Research Council (ARC) study of low-rank coal conversion using the CO-steam process. The report describes experimental work and a conceptual commercial plant design for a Black Thunder Mine coal-based CO-water liquefaction system. The commercial plant study was prepared by Kilborn Inc., under subcontract to ARC. A review of the economic aspects of these reports was begun. Reported capital costs were compared with those generated by Bechtel in their DOE-sponsored low-rank coal study. A number of questions concerning the methodology and results reported by Kilborn were identified. Further analysis of the results is planned.

Task 6 - Reporting

All reporting is on schedule. Bi-monthly conference calls were held as scheduled among the project participants. Reports were issued documenting the calls. The quarterly technical review meeting was held on March 25, 1997, at UK/CAER in Lexington, KY, to discuss progress and plans for the direction of the project. A meeting record was issued. A summary report of the project accomplishments through March 1997 was issued to DOE/FETC.

FORECAST AND PLANS

Microautoclave tests will be made with HI"E" and different amounts of water gas shift catalysts to determine the minimum catalyst loading required to maintain high coal conversion and product quality. In-line filtration will be implemented next quarter.

The cause for the slow filtration rates of products of Black Thunder Mine coal and HI"E" liquefaction will be sought.

Second-stage testing will begin in the 300 mL reactor; the distilled filtrates of Runs 14-LA and 14b-LA products made with HI"A" are planned to provide a comparison for products of HI"D" and HI"E"-promoted liquefaction.

Work on the integrated liquefaction system balance (ILSB) with hydride ion source "E" will continue as data are made available from first-stage, filtration, and second-stage experiments. The CO, hydrogen, and fuel balance for the case of high first-stage CO₂ yield will be completed.

TABLE 1

COMPARISON OF CONVERSION WITH KAWASAKI ANTHRACENE OIL AND REILLY INDUSTRIES ANTHRACENE OIL,
BLACK THUNDER MINE COAL, 60 MIN

Run No. (K-AO)	Run No. (R-AO)	Temp, °C	Coal, g	Solvent, g	HI"A", g	Methanol, g	CO, psig	NaAlO ₂ , g	Coal Conv. K-AO	Coal Conv. R-AO
163c	163/163b	350	5.00	8.00	4.00	-----	-----	-----	79.2	84.1/84.3
185	209	350	4.47	7.15	-----	-----	975	-----	54.9	59.5
186	210	350	4.47	7.15	-----	2.88	975	-----	71.3	72.5
187	211	350	4.47	7.15	-----	2.88	975	0.28	79.5	88.2
189	212	350	4.47	7.15	-----	2.88	975	0.56	70.5	84.5
190	213	350	4.47	7.15	0.69	2.88	975	0.28	80.6	87.7
192	215/215a	350	4.47	7.15	-----	3.84	1299	0.28	80.4	88.8/90.2
193	216	350	4.47	7.15	-----	3.74	1299	0.37	80.1	89.6
194	194b/217a	375	4.47	7.15	-----	2.88	975	0.28	85.4	90.1/96.0

TABLE 2
MASS BALANCE DATA

Run No	Yields				Cake Insolubles		Solids in Feed		Coal Conversion*			
	Distillate (%)	Filtrate (%)	Cake (%)	Total (%)	THFI (%)	QI (%)	THFI (%)	QI (%)	(a) (%)	(b) (%)	(c) (%)	(d) (%)
14-LA	1.6	88.0	7.6	97.2	53.5	49.2	6.1	4.0	86.7	92.7	93.3	94.0
14b-LA	5.1	85.4	6.4	96.9	58.8	53.3	5.8	4.0	86.8	93.4	92.7	94.7
15-LA	7.1	80.0	12.9	100	59.1	55.0	7.7	5.6	85.6	85.9	92.3	87.7
16-LA	1.5	79.0	15.2	95.6	43.7	38.5	11.4	6.2	78.3	90.8	93.1	94.1

*SO₃ free basis.

Coal conversions determined from: (a) THF insolubles in a sample of the reactor product; (b) THF insolubles in the filter cake; (c) Quinoline insolubles in a sample of the reactor product; (d) Quinoline insolubles in the filter cake.

TABLE 3
SUMMARY OF FILTRATION TESTS

Run No.	NCF No.	Filter membrane	Aperture retention, μm	Temp., $^{\circ}\text{C}$	Viscosity (mPa s)	Solids in feed, %	Pressure, kPa	Cake resistivity ($\text{m/kg} \times 10^{10}$)	Total flow after 30min. (kg / m^2)
14-LA	51	GFA	1	290	0.6	4.0	143	450	67
							366	760	83
	52	Conidur	100 x 500	280	0.7	4.0	141	233*	116*
	53	GFA	1	300	0.5	3.1	143	439	84
	54	GFA	1	300	0.5	1.7	143	586	97
14b-LA	55	GFA	1	300	0.6	4.0	138	630	56
							283	1400	54
							436	3450	43
							576	4260	44
	56	Conidur	60	300	0.6	4.0	143	560	61
							288	1510	52
							438	2340	52
							586	4470	43
15-LA	57	GFA	1	250	1	5.7	148	2660	18
							295	4720	19
							579	16600	14
16-LA	58	GFA	1	265	1	5.7	572	890	70
	59	GFA	1	270	1	6.2	135	1920	20
							286	2680	24
							564	6360	22
	60	Conidur	60	265	1	6.2	568	870	68
	61	GFA	1	300	0.6	6.2	566	12500	16

*High flow rates associated with marginal bridging of the screen.

TABLE 4

COMPARISON OF HYDROTREATING RESULTS FOR VARIOUS HYDRIDE PREPARED FEEDSTOCKS (a)

CONSOL Sample no.	Coal source Hydride Ion Reagent	Solvent Temp., Time	Filtration and Hydrotreat Run No.	566 °C ⁺ to Hydrotreater, wt % MAF feed	566 °C ⁺ resid conversion, wt % MAF resid	C ₁ -C ₃ gas yield, wt% feed	C ₁ -C ₃ gas yield per g resid converted	H ₂ uptake, mg/g MAF feed	H ₂ uptake per g resid converted
8-LA (A&B) heavy extract(c)	Glenharold HI"A"	488 °C- HD 350 °C 60 min	NCF 37DR R6-227-1	44.4	55.3	4.5	18.2	20	81
			NCF 37DR	44.4	59.6	4.1	15.4	26	97
8c-LA DR lights	Glenharold HI"A"	488 °C- HD 350 °C 60 min	R6-309-1	32.5	53.5	4.5	25.6	35	201
8c- LA heavies (b)	Glenharold HI"A"	488 °C- HD 350 °C 60 min	NCF 42F1 R6-309-2	36.0	58.2	4.5	21.7	46	219
			NCF 42F1 R7-013-1	36.0	55.4	6.4	32.0	50	252
			NCF 42F1 R7-043-1 [1.5g sample]	36.0	53.0	4.6	23.8	44	230
10-LA	Glenharold HI"A"	Lummus 350 °C 60 min	NCF 44FDR R6-323-1	99.0	57.3	na	na	na	na
			NCF 44FDR R6-009-3	99.0	58.0	8.2	14.4	51	89
11-LA	Glenharold HI"A"	Reilly AO 350 °C 60 min	NCF 46FDR R6-346-1	98.0	65.1	na	na	na	na
			NCF 46FDR R7-013-2	98.0	59.2	8.5	14.6	47	81
			NCF 46FDR R7-041-1	98.0	57.3	8.6	15.4	49	87

a. Hydrotreated at 440°C for 60 min with 10,000 ppm Mo in Molyvan L.

b. Insoluble in Wilsonville solvent at 60°C; redissolved in aromatic solvent and concentrated to give higher extract concentrations.

c. Hydrotreated at 440°C for 60 min with 1,000 ppm Mo in Molyvan L.

TABLE 4 (cont.)

COMPARISON OF HYDROTREATING RESULTS FOR VARIOUS HYDRIDE PREPARED FEEDSTOCKS (a)

CONSOL Sample No.	Coal Source Hydride Ion Reagent	Solvent Temp., Time	Filtration and Hydrotreat Run No.	566° C ⁺ to Hydro-treater, wt % MAF Feed	566° C ⁺ Resid Conver, wt % MAF Resid	C ₁ -C ₃ Gas Yield, wt % Feed	C ₁ -C ₃ Gas Yield per g Resid Converted	H ₂ Uptake, mg/g MAF Feed	H ₂ Uptake per g Resid Converted
9-LA (b)	Glenharold HI"A"	Lummus 350 °C 60 min	NCF 43F2DR R7-048-1	99.7	37.5	7.8	20.8	27	72
			NCF 43F2DR R7-048-2	99.7	38.9	7.8	20.0	33	85
12-LA	Glenharold NONE	Lummus 410 °C 60 min	NCF 47FDR R6-352-1	94.8	33.1	na	na	na	na
			NCF 50FDR R7-055-1	96.9	37.1	7.2	19.9	42	116
13-LA	Glenharold NONE	R-263 V1067 410 °C 15 min	NCF 49FDR R6-352-2	95.1	43.5	na	na	na	na
			NCF 48FDR R7-063-2	97.2	44.7	7.3	16.8	49	113
14-LA	Black Thunder HI"A"	Reilly AO 375 °C 60 min	NCF 51,52, 55&56 FDR R7-056-1	95.9	50.9	na	na	na	na
			NCF 51,52, 55&56 FDR R7-063-1	95.9	53.3	11.2	21.9	58	114
15-LA	Black Thunder HI"D"	Reilly AO 375 °C 60 min	NCF 57FDR R7-064-1	93.5	44.1	8.7	21.2	43	105

(a) Hydrotreated at 440 °C for 60 min with 10,000 ppm Mo in Molyvan L.

(b) No catalyst added, 440 °C for 60 min with 2% H₂S in H₂.

TABLE 5

BOILING RANGE DISTRIBUTIONS OF HYDROTREATED FILTRATE DISTILLATES ^a

CONSOL Run & Distillate Product No.	C ₄ + vapor, % MF feed	naphtha	600- 800 °F	800- 910 °F	910- 1050 °F	Total distillable, % MF feed
9-LA Thermal R7-048-1	1.4	5.4	16.2	3.4	0.4	25.4
9-LA R6-261-1	1.6	10.6	21.8	13.3	10.1	55.8
10-LA R7-009-3	3.5	4.3	20.8	13.0	5.2	43.3
11-LA R7-013-2	3.2	4.9	20.7	12.6	6.7	44.9
12-LA R7-055-1	4.0	4.4	12.1	7.2	3.6	27.3
13-LA R7-063-2	3.3	7.1	13.2	14.0	1.1	35.3
14-LA R7-063-1 ^b	5.2	-	-	-	-	36.7
15-LA R7-064-1	3.3	5.1	12.5	9.7	4.1	31.4

- a. Hydrotreatment at 440°C for 60 min with 10,000 ppm Mo in Molyvan L (except "Thermal" run).
- b. 14 La distillate sample was not available for SIMDIS analysis.

TABLE 6
HYDROTREATING RUN ELEMENTAL ANALYSES

Run No.	Sample ID	Nitrogen, %	Carbon, %	Hydrogen, %	O+S % (diff)	H /C ratio
NCF 50 FDR (12 La)	44890	1.6	88.8	5.4	4.2	0.06
R7-055-1D	44883	2.0	90.3	8.2	-0.5	0.09
R7-055-1R	44884	1.5	86.6	4.7	7.2	0.05
NCF 48 FDR (13 La)	44889	1.3	85.6	5.6	7.5	0.07
R6-352-2D	44887	1.7	90.2	8.8	-0.7	0.10
R6-352-2R	44888	1.3	85.4	5.0	8.3	0.06
NCF 51,52,55, 56 FDR (14 La)	44891	1.6	82.7	6.0	9.7	0.07
R7-056-1D	44885	1.8	90.0	9.1	-0.9	0.10
R7-056-1R	44886	1.3	78.5	4.4	15.8	0.06

TABLE 7
CAPACITY OF 300 ML CSTR VS. DEPTH OF FILL

Height above vessel bottom, inches	Cumulative capacity, mL	Height above vessel bottom, inches	Cumulative capacity, mL
0	0	2	85.8
0.25	3	3	137.3
0.5	10.7	4	188.8
0.75	21.7	5	240.3
1	34.3	6	291.7

Figure 1. Filtrate Hydrotreating Data

TABLE 1

CONCEPTUAL COMMERCIAL PLANT

BASIS: RUN 8-LA (FEED COAL: GLENHAROLD LIGNITE)

FUEL GAS BALANCE

• **Consumption (millions of Btu {LHV}/hr):**

- Coal Slurry Preheating	491
- Water Preheating	745
- Methyl Formate Preheating	317
- Filtrate Vacuum Tower	319
- Second-Stage Vacuum Tower	169
- Distillate Hydroprocessing	182
- Catalytic Reformer	826
- Steam Reformer	635
- Drying Coal Gasification	391
- Sulfur Recovery Unit	<u>176</u>
TOTAL	4,251

• **Production ((millions of Btu {LHV}/hr):**

- Liquefaction Off Gas	2,621
- Upgrading	<u>2,597</u>
Sub-Total	5,218
- Steam Reforming Feed	<u>-967</u>
TOTAL	4,251

Note: Based on 17,929 T/D MF Coal to Liquefaction