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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, \$1,032,000 Obligated

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REPORTING PERIOD: April 1 through June 30, 1997

CONTRACT OBJECTIVES: No Change

SUMMARY OF TECHNICAL PROGRESS - OVERALL

Task 3 work was concluded this quarter. Filtration characteristics of first-stage products were determined in off-line tests. The one-liter autoclave first-stage reactor was successfully operated with an in-line filter. Construction of the 300 mL second-stage catalytic upgrading continuous stirred tank reactor (CSTR) was completed. The reactor system underwent shake-down trials, was modified and subsequently used in tests with first-stage products. The economic evaluation of the conceptual process design continued using data from first- and second-stage tests. First-stage catalyst costs were estimated based on usage rates currently being employed. Task 4 was authorized; a Work Plan was drafted for approval.

SUMMARY OF TECHNICAL PROGRESS BY TASK

Task 3 - Flow Sheet Development

First-Stage Solubilization and Filtration

Catalyst Studies

Ten successful one-liter autoclave tests were executed this quarter. Tables 1 and 2 give a summary of the first-stage tests and filtration data.

Studies were continued to investigate the filtration characteristics of one-liter reactor products. It previously was reported that filtration rates as low as 16 kg/m²h and inconsistent behavior (filtration rates 16-70 kb/m²h) of the material during filtration were observed when NaAlO₂ was used as the first-stage catalyst (Run 16-LA). This behavior was thought to be attributable to either finely divided catalyst particles deposited in the cake, or the mild first-stage conditions which resulted in plastic, deformable, residual particles which inhibited flow through the cake. To investigate these possibilities, Run 17-LA (a repeat of Run 16-LA) was made and the product was filtered at 560 kPa using a step- wise increase in temperature. Viscosity measurements were made using a Brookfield viscometer on samples of the Run 17-LA product. At the initial temperature of 190 °C, viscosity was 4 mPas. At 250 °C, viscosity decreased to 1.2 mPas. At the last temperature tested, 300 °C, viscosity was measured at 0.7 mPas. The corresponding filtration rates were 15, 9 , and 5 kg/m²h. The filtration rate at 190°C (15 kg/m²h) was as low as lowest rates obtained with products of Run 16-LA.

Microautoclave tests were made to determine the effectiveness of Na₂CO₃ as catalyst and the minimum amount of Na₂CO₃ catalyst required to maintain good coal conversions in the first stage (Table 3). Baseline tests (no catalyst) gave ca. 83 wt % coal conversion. Coal conversions with catalyst loadings between 850 and 38 mmole Na₂CO₃/kg dry coal are ca. 90 wt % (±2 wt %, abs.), indicating that Na₂CO₃ is an effective first-stage catalyst at low catalyst loadings.

Subsequent to the determination made on microautoclave scale that Na₂CO₃ is an effective first-stage catalyst, one-liter autoclave test Run 18-LA was performed using Na₂CO₃. The products of this test gave very low rates of filtration (5 kg/m²h) when filtered through a glass fiber filter at 290 °C and 560 kPa. An 8 g aliquot of the product of Run 18a-LA (a repeat of Run 18-LA) was water washed in boiling water to remove Na₂CO₃ catalyst. The recovered product was distilled to remove retained water and then subjected to filtration tests. The water-washed, distilled product of Run 18a-LA had the same poor filtering properties as the product of Run 18-LA.

Analysis of the wash water indicated that only about 20% of the catalyst had been removed by water washing.

Three one-liter autoclave tests (Runs 19-LA, 20-LA, and 21-LA) were completed to determine the reason for the poor filtering performance found for products of Runs 16, 17, 18, and 18a-LA. Possible causes were the use of Black Thunder Mine coal in the hydride ion "E"-promoted reaction, the use of catalysts (such as NaAlO_2 and NaCO_3) for that reaction, or the amount of catalyst used (Table 4). Runs 19-LA, 20-LA, and 21-LA were made at half reactor loadings. No analyses were made of product gases. Run 19-LA was made using no catalyst and gave an acceptable filtration rate ($64 \text{ kg/m}^2\text{h}$) with a low cake resistivity of $58 \times 10^{10} \text{ m/kg}$. Although in the range of commercial acceptability, the flow rate was lower than might have been expected from a cake with low resistivity. However, the low conversion 70.0% (QI basis) and 54.7% (THFI basis) indicates that the first-stage product sample contained a large amount of solids, which depressed the filtration rate. Run 20-LA was made using 90 mmole/kg dry coal Na_2CO_3 as opposed to the 300 mmole/kg used in Run 18-LA. Filtration rates, however, were the same as for products of Run 18-LA. Coal conversion for Run 20-LA was ca. 76% (QI basis) and ca. 53% (THFI basis). Run 21-LA used KOH as the first-stage catalyst and produced a high filtration rate, $100 \text{ kg/m}^2\text{h}$ at 300°C and 140 kPa. Coal conversion was 71.5% (QI basis) and 52.4% (THFI basis). This is much lower than was predicted from microautoclave tests in which a 92 wt % coal conversion had been obtained.

These results indicate that products made using Na_2CO_3 and NaAlO_2 give very low filtration rates even when the catalyst loading is reduced by 70%. Encouraging results were achieved in Run 21-LA when KOH was used. It is believed that the small batch size (approximately half that used in previous one-liter autoclave tests) used in Run 21-LA may have contributed to the low conversion because of low HI "E" loading. However, the high filtration rates, contrary to those found when Na_2CO_3 was used as catalyst, indicate that KOH may be a good candidate first-stage catalyst.

In-Line Filtration

To determine if filtration could be improved if the sample is maintained at filtration temperatures rather than cooled and reheated, 1-L autoclave test Run 22-LA (a repeat of Run 21-LA) was made. In this first attempt at using the filter in-line, the run had to be aborted about 30 min after heat-up began (reactor temperature 251°F) because of pressure loss in the reactor. It was found that the sample had blown through the closed valve between the reactor and the filter. The

contents of the filter were filtered into the receiver. The valve was removed, cleaned, inspected, and pressure tested at reaction temperature (ca. 420°F) before reassembly. It could not be determined why the valve failed; however, it is possible that the packing may have reseated on heating for the first time or that the valve was not fully closed.

Two tests, Runs 23-LA and 23a-LA, were made using the same feed as Run 21-LA, but with sufficient loading to produce a system pressure at reaction temperature close to 4000 psi. The products of Run 23-LA were filtered through the in line filter apparatus. The contents of the 1-gal filtrate receiver were analyzed and found to contain essentially all of the ash and catalyst fed to the reactor. No evidence was found that the sample by-passed the filter medium (glass fiber backed by Whatman 42 filter paper). It appears that the filter papers, which rested on a 1/16" stainless steel screen, punctured and allowed the ash to pass through into the filtrate. The whole product was re-filtered in the 35 mm filter unit. It gave a high filtration rate (208 kg/m²h) and a low cake resistivity (69×10^{10} m/kg). Analysis of Run 23-LA product prior to filtration gave coal conversions of 93.8 wt % (QI basis) and 87.3 wt % (THFI basis). After filtration, analysis of the filter cake gave coal conversions of 95% (QI basis) and 94% (THFI basis) with 1.7% (THFI) present in the filtrate. However, the material balance for Run 23-LA was poor (88.1 wt %).

Run 23a-LA, a repeat of Run 23-LA, was successfully executed. Products were filtered in-line through a glass fiber filter supported by a 60 µm Conidur plate. The filter cake was approximately 1/8" thick. Analysis of the filtrate showed <0.1% QI and 1.1% THFI. Analysis of the filter cake gave coal conversions of ca. 99% (QI basis) and ca. 98% (THFI basis).

Mass balance for Run 23a-LA also was poor at 90.2%. About 30 g of oil/water were expected to be recovered from the flash receiver vessel. However, no material was recovered from the flash receiver vessel for Runs 23-LA or 23a-LA. The outlet for nitrogen used to blow dry the filter cake was routed through the flash receiver vessel. It was suspected that the vented nitrogen carried with it the contents of the flash receiver vessel. The outlet of the nitrogen was rerouted to bypass the receiver vessel and instead pass through an dry ice-cooled condenser before venting.

Run 23b-LA was executed, but was unsuccessful. At run temperature, system pressure was only 3300 psi (about 800 psi below expected). It is speculated that there was a small leak in the reactor system. The run was aborted prior to transfer of material into the filter. The collected

gas quantity was unreasonably high (12 cu ft) and contained predominantly N₂. The reactor was cleaned and the products retained, but not analyzed.

Run 23c-LA was a successful test. System pressure was ca. 4100 psi. A dry 1/8" thick filter cake (10.40 g) was recovered. The flash vessel contained ca. 35 g of oil and water. Total gas collected was ca. 3.5 cu ft. A mass balance of 101.8% was obtained. The hydrogen gas determination obtained by difference was checked by direct measurement on a different apparatus calibrated for low H₂ gas content in mixtures. By difference, the value was ~5 vol %; direct measurement gave a hydrogen content of ~1.5 vol %. The discrepancy is large enough to warrant direct H₂ gas analyses to be performed on all product gas samples whenever possible. Elemental analyses were obtained for all recovered material. A THF pressure filtration of the filtrate gave 2.09 wt % THF insolubles, which includes 0.15 wt % ash (whole sample basis). THF pressure filtration of the filter cake was carried out. The cake contains 46.2 wt % THFI which includes 19.15 wt % SO₃-free ash (whole sample basis). Products of Run 23c-LA were further analyzed for quinoline and THF solubility by a *hot* THF extraction method. The filtrate gave 0.3% quinoline insoluble (QI) and 2.2% THFI. The filter cake was found to contain 44.8% QI and 45.9% THFI. Coal conversion is calculated to be ca. 95% (THFI basis). The ash balance was poor. Assuming the KOH reports to the ash, only 56% of the ash can be accounted for. Potassium analyses, however, show only ca. 0.0036 moles total K (or 0.20 g of KOH) recovered in the filter cake and the washings of the reactor system. The feed coal contained only 0.0001 moles K. If it is assumed that the KOH does not report to the ash, the ash balance is ca. 67%. A potassium analysis will be made of the filtrate and the flashed oil/water sample to close the potassium balance.

Run 23d-LA was successfully executed. Overall material balance was 99.8%. In-line filtration was monitored. It took 35 seconds at 103 kPa and 250°C to filter the entire product. This corresponds to a rate of 276 kg/m²h and can be compared to the 208 kg/m²h achieved with Run 23-LA products in the 35 mm diameter off-line filter. Yields were 167.25 g of filtrate and 23.35 g of filter cake. Analyses of all product fractions are being obtained. It is believed that the larger quantity of filter cake obtained from Run 23d-LA vs. Run 23c-LA was a consequence of reduced nitrogen blowing, with the resultant filter cake containing more filtrate. This will be confirmed by extraction of the filter cake.

Ash balances for Runs 23-LA, 23a-LA, and 23c-LA were inconsistent. Ash recoveries of 88%, 59%, and 56%, respectively, were obtained. Run 23-LA, which had the highest ash recovery,

also had the largest yield of filter cake (3.9 g or 75% of the 88% recovered). 2.6 g of ash (or 50% of the 59% ash recovery) was recovered from the filter cake of Run 23a-LA, and 2.0 g of ash (43% of 55% recovered) was found in the filter cake of Run 23c-LA. This indicates that not all the ash is being quantified in the filtrate or possibly flashed products (in the form of potassium salts). Analysis of Run 23d-LA products will be examined carefully to obtain a good ash balance.

Second-Stage Upgrading

Sample Nomenclature

A sample nomenclature system was enacted this quarter for second-stage feeds and products. Samples prepared in the 1-L autoclave first stage reactor are numbered consecutively and suffixed with 'LA'. If multiple tests are made at the same conditions, an alphanumeric system of labeling is used as follows: Run ##x - LA, where ## is numeric and x is alphabetic. These samples then are distilled and, in some cases, combined to produce sufficient sample for second-stage upgrading. Henceforth, these combined samples will be designated ##xyz (where x, y, and z are different first-stage tests). A suffix (indicating the consecutive run number for the test in which this feedstock was used) is appended, followed by the type of the second-stage equipment used (ex., ##xyz-#, 300 CSTR (for continuous stirred tank reactor) or 50 MA (for microautoclave)).

Sample Preparation for Second-Stage Testing

The filtrates from Runs 23-LA, 23a-LA, and 23c-LA were composited to give 426.4 g of filtrate. This composite was distilled to produce 96 g of distillate residue for a single hydrotreating test in the 300 mL second-stage reactor. The distillation was carried out under vacuum and was cut at an atmospheric equivalent of 454 °C (850 °F). This cut temperature gave a high yield of >566 °C material without subjecting the residue to severe conditions in the still. The products collected were in the proportions, 74.7% <454 °C distillate and 25.3% >454 °C residue. Analysis by simulated distillation of the distillate gave 100% <566 °C, and of the residue, 6% <566 °C. The softening point of the residue was 290 °C.

300 mL CSTR Tests

Three hydrotreating tests were completed in the 300 mL reactor facility. A shake-down test was completed with a 50 g sample of composited filtrate from Runs 15-LA and 15a-LA (previously designated NCF 57 FDR). It was hydrotreated in the 300 mL CSTR at the standard conditions of 440 °C for 60 min, using 1% Mo (feed basis) from Molyvan L as the catalyst precursor. This run is numbered 15oa-1, 300 CSTR. The second test was made using deashed resid from the

Wilsonville pilot plant. This test (DARo-1, 300 CSTR) was used to confirm procedures in the new reactor system and to conserve first-stage product sample until operational problems in the reactor system could be resolved (see below). The third test (23oac-1,300 CSTR) fed a composite of distilled, filtered first-stage products from Runs 23-LA, 23a-LA, and 23c-LA.

Data for each of the three runs completed this quarter in the 300 mL CSTR are shown in Table 6. For Run 15oa-1, 300 CSTR, the overall material balance was 102 wt %, carbon balance was 101 wt %, and hydrogen balance was 99 wt %. Resid conversion was 49 wt % and H₂ uptake (calculated from the H₂ gas balance) was 54 mg/g MAF feed. Comparable values (44% conversion and 43 mg/g MAF feed H₂ consumption) were obtained with this feedstock in microautoclave tests which were reported previously. However, C₁-C₃ gas yield for 15oa-1, 300 CSTR, was 23%, after correction of methane yield for decomposition of the DMDS sulfiding agent. This value is much higher than the ca. 9% obtained in the microautoclave tests. CO/CO₂ yields were the same as those obtained in the microautoclave tests. A 270% increase in the fixed carbon content of the non-distillate product suggests the possibility that coking occurred. An examination of the mass flow controller indicated that the instrument was not properly calibrated. Consequently, H₂ flow in 15oa-1, 300 CSTR, was about 20% of the set point. The instrument was returned to the manufacturer for repair.

Modifications were made to the 300 mL reactor system and start-up procedure. These include the installation of an inlet gas tube for delivery of H₂ into the reaction mixture. An extended shaft turbine blade stirrer was installed to provide better agitation of the mixture. A strobe tachometer was used to measure the actual stirring rate of the air motor driven stirrer used on the CSTR. It was found to be 497 rpm unloaded at 60 psig initial motor air pressure (52 psig flowing). It stirred at 365 rpm with a viscous load. The feed charge size was increased to assure good mixing. The H₂ gas flow rate was increased from 0.3 to 1.7 std. L/m and a continuous H₂ flow, instead of a stagnant charge of H₂, was used during heat up.

Two CSTR runs were completed after the modifications were made. Run DARo-1, 300 CSTR, was made with 100 g of Wilsonville Run 258A DAR and 12.3 g Molyvan L (1% Mo on feed). Resid conversion was 63% (Table 6). The overall material balance for DARo-1, 300 CSTR was over 100%. Because of the high material balance, the sampling and measurement system for the product gas stream was reviewed. The gas sampling point was separated from the vent meter by a 500 mL knockout scrubber. The gas to be vented first during a run should have been high in H₂ content, since the system is pressurized with pure hydrogen prior to heat up. In

Run DARo-1, 300 CSTR, the gas sample was initially 73.4% H₂. The gas sampling point was relocated to the back pressure regulating valve. In Run 23oac-1, 300 CSTR (see below), the first 10 min sample contained 99.0% H₂ as a result of this change. This change affects the overall material balance. The volume of gas vented remains the same, but the molecular weight assigned to that volume is much lower. Gas compositions for the first period of Runs 15oa-1, 300 CSTR, and DARo-1, 300 CSTR, were recalculated with the data from Run 23oac-1, 300 CSTR. Because this correction was applied only to sample collected in the initial 10 min of the run and ¾ of the product gases are released at the conclusion of the run when the cold reactor is vented, the impact on total gas yield is small. The overall material balance reported in Table 6 reflects the recalculated value for DARo-1, 300 CSTR.

The second run, Run 23oac-1, 300 CSTR (Table 6), was made with 94 g of composited first-stage distilled filtrate from Runs 23-LA, 23a-LA, and 23c-LA, and 11.6 g Molyvan L. Resid conversion was 57%, with an overall material balance of 96%. Light hydrocarbon gas yields were higher than those obtained in microautoclave tests. An internal temperature sensor will be installed in the CSTR to determine if the wall temperature is much higher than the vapor temperature presently measured. A higher temperature may result in increased gas make. Simulated distillation data for the 300 mL CSTR runs are given in Figure 1.

Microautoclave Tests

To assess the reactivity of the first-stage filtrate resid, microautoclave experiments were performed at 440 °C with 1% Mo, using the composite sample fed in Run 15oa-1, 300 CSTR. Runs were made at 30, 90, and 120 min (Table 7). A plot of resid remaining vs. reaction time is presented in Figure 2. Resid conversion asymptotically approached 53-55%. The data obtained using Wilsonville Run 258A deashed resid (DAR) as feed also is shown. The experiments with the DAR utilized a larger feed sample and 0.1% Mo rather than 1%. It is believed that the comparison is valid because resid conversions of the Wilsonville Run 258 DAR at 60 min with 0.1% and 1.0% Mo previously were found to be the same. Resid remaining at each reaction time is greater for the DAR than the distilled first-stage filtrate. Figure 3 is a plot of hydrogen consumed per gram MAF feed and hydrogen consumed per unit resid conversion for the reaction times of 30, 60, 90, and 120 min. with the 15oa-1, 300 CSTR feed. As time progresses, H₂ consumption increases. These data are consistent with data previously obtained for microautoclave tests with first-stage products.

Task 3 - Flow Sheet Development

First-Stage Solubilization

Authorization was received to initiate work under Task 4. A work plan was drafted.

Task 5 - Engineering and Economic Study

First-Stage Catalyst

The leading U.S. producer of caustic potash (KOH) was contacted to obtain technical and pricing information. A main supplier of the feed material for KOH production was contacted for additional background information. Commercial production is via electrolysis of an aqueous potash (KCl) solution. The cost of electric power is an important factor in the cost of KOH production. KOH is available mainly as a 45% aqueous solution. The major North American potash producers are located in Saskatchewan, Canada. Current U.S. KOH production capacity is 500,000 t/y. Oxychem is the leading producer, with 350,000 t/y. Oxychem's U.S. plants are located in Mobile and Muscle Shoals, Alabama, and in Delaware City, Delaware. A 20,000 t/d liquefaction plant would require 65,000 t/y of KOH at a 1% addition rate. The current list price is 34¢/lb KOH. For planning purposes, Oxychem suggested a 10% discount off the list price. Therefore, the estimated cost of using KOH at a 1% addition rate is \$2/bbl of gasoline (at a gasoline yield of 3.1 bbl/ton coal).

Plant Design

Based on the material balance methodology previously developed for liquefaction and upgrading, a conceptual commercial plant design was begun for a case representative of the HI "E" first-stage process, which has a higher first stage CO₂ yield, a lower first-stage catalyst consumption, and a lower first-stage distillate yield than previous cases examined. (Data are available in Confidential Appendix 2, Table CA2-1). The projected case has a significant hydrogen consumption efficiency advantage over the Base Case. However, these results are based on speculative process yields that will be modified once actual experimental data become available (Table CA2-2).

An analysis was made of the available data from Run 23c-LA. Light hydrocarbon gas yields were 0.6 wt % on MF coal, consistent with the prior Run 23 series data. HI "E" consumption was lower by 8 % (abs.) than in the previous Run 23 series tests. However, the excess CO₂ yield of 55.6% was ca. 40 wt % (abs) higher in Run 23c-LA than in the previous runs. The H₂ produced to HI "E" consumed molar ratio varies little for all Run 23-LA series tests and is close to 1. Direct

H₂ measurements likely will improve the reliability of this value. The as-measured carbon balance is good, but the ash balance for Run 23c-LA is poor.

Task 6 - Reporting

All reporting is on schedule. Bi-monthly conference calls were held as scheduled with UK/CAER and LDP. Reports documenting the calls were issued. The quarterly technical review meeting is scheduled for July 9, 1997, at UK/CAER in Lexington, KY, to discuss progress and plans for the direction of the project. A Task 4 Work Plan was drafted.

FORECAST AND PLANS

Distillation of the Run 23c-LA first-stage filtrate resulted in a material with a high softening point. This material may be difficult to handle in a continuous process. It is planned to distill the products of Run 23d-LA to yield 70% instead of 74-75% distillate to produce a residue with a softening point of 200-240 °C.

A series of tests at the conditions of Run 23-LA will be made in the one-liter first-stage reactor to produce a large quantity of material for second-stage upgrading tests. Three first-stage tests with 100 g coal loadings are required to make sufficient 566 °C⁺ distillate residue for a single second-stage test in the 300 mL CSTR.

An elementally balanced first-stage reaction system material balance for Run 23c-LA will be prepared after complete analytical data are obtained. A similar balance for the second-stage reaction system will be prepared once hydrotreating data become available. The results of these balances will be the input for the conceptual commercial plant design and economic evaluation effort.

TABLE 1
FIRST-STAGE ONE-LITER AUTOCLAVE TESTS

Run Number (a)	NCF No	Solvent to Dry Coal Ratio	Catalyst	
			Type	(mole/kg dry coal)
16-LA	58, 59, 60 & 61	2.0	NaAlO ₂	0.551
17-LA	63	1.8	NaAlO ₂	0.530
18-LA	62	1.8	Na ₂ CO ₃	0.308
18a-LA (b)	64	1.8	Na ₂ CO ₃	0.312
19-LA	65	1.8	None	None
20-LA	66 & 67	1.8	Na ₂ CO ₃	0.060
21-LA	68	1.8	KOH	0.205
23-LA	69	1.8	KOH	0.204
23a-LA	-	1.8	KOH	0.200
23c-LA	-	2.0	KOH	0.230
23d-LA	-	2.0	KOH	0.230

- (a) All runs made with Black Thunder Mine coal and Reilly Industries anthracene oil at 375°C for 60 min with HI "E"
- (b) sample washed with hot water to recover catalyst.

TABLE 2
SUMMARY OF FILTRATION TESTS

Run No.	NCF No.	Filter membrane	Aperture retention (µm)	Temp (°C)	Viscosity (mPas)	Pressure (kPa)	Cake resistivity (m/kg x 10 ¹⁰)	Total flow after 1 h (a) (kg / m ²)
16-LA	58	GFA	1	265	1	572	890	70
	59	GFA	1	270	1	135	1920	20
						286	2680	24
						564	6360	22
	60	Conidur	60 x 400	265	1	568	870	68
	61	GFA	1	300	0.6	566	12500	16
17-LA	63	GFA	1	190	4	560	3440	15
18-LA	62	GFA	1	290	0.5	566	221000	5
18a-LA	64	GFA	1	280	0.8	filter blinded		
19-LA	65	GFA	1	255	1	139	58	64
20-LA	66 & 67	GFA	1	260	1	filter blinded		
21-LA	68	GFA	1	296	0.4	142	73	97
23-LA	69	GFA	1	300	0.4	210	69	208
23a-LA	—	GFA	1	250	0.8	210	N.D.	N.D.
23c-LA	—	GFA	1	250	0.8	210	N.D.	N.D.
23d-LA	—	GFA	1	250	0.8	105	8	276

N.D. = not determined.

(a) Filter in operation for 30 min each hour.

TABLE 3

EFFECT OF Na₂CO₃ CATALYST LOADING ON COAL CONVERSION

Catalyst: Na₂CO₃; Coal: Black Thunder Mine;
 Solvent/dry coal: 1.8; Temperature: 375°C;
 Hydride ion source: HI "E"

Solvent: Reilly Anthracene Oil
 Time: 60 min

mmole catalyst/kg dry coal	Conversion, wt %
0	83.5
0	82.7
850	88.4
222	89.5
165	91.0
82	89.5
38	88.1

TABLE 4

FIRST-STAGE CATALYST EFFECT ON FILTRATION

Coal: Black Thunder Mine; Solvent: Reilly Anthracene Oil
 Temperature: 375°C; Time: 60 min;
 Hydride ion source: HI "E"

	Run 18a-LA (a)	Run 19-LA	Run 20-LA	Run 21-LA
catalyst	Na ₂ CO ₃	None	Na ₂ CO ₃	KOH
catalyst, g	2.65	0.00	0.25	0.45
coal, g	102.33	60.39	50.00	50.00
HI "E"	Full Charge	Half Charge	Half Charge	Half Charge
solvent, g	140.46	80.90	71.50	71.50
Conversion (b), wt %	N.D. (c)	49	53	45
Filterability	Poor	Good	Poor	Good

- (a) 8 g portion of product water washed - recovered only 1/5 Na added as Na₂CO₃
 (b) based on THF solubility
 (c) N.D. = not determined

TABLE 5

FILTRATION AND DISTILLATION YIELDS FOR RUNS 23-LA, 23a-LA, 23c-LA

Filtration		g	
23-LA	Coal In (dry)	79.50	
	Coal In (daf)	75.10	
	Solvent In	142.08	
	Filtrate	166.14	QI - 0.4%, THFI - 1.7%
	Filter Cake	21.59	QI - 43.4%, THFI - 45.7%
23a-LA	Coal In (dry)	80.40	
	Coal In (daf)	75.95	
	Solvent In	142.25	
	Filtrate	170.15	QI - 0.1%, THFI - 1.1%
	Filter Cake	14.25	QI - 43.6, THFI - 46.3%
23c-LA	Coal In (dry)	71.30	
	Coal In (daf)	67.38	
	Solvent In	139.80	
	Filtrate	168.40	QI - 0.3%, THFI - 2.2%
	Filter Cake	10.40	QI - 44.8%, THFI - 45.9%
TOTAL	Coal In (dry)	231.20	
	Coal In (daf)	218.43	
	Solvent In	424.13	
	Filtrate	504.74	
	Filter Cake	46.24	
DISTILLATION			
	Combined Feed In (b)	426.38	
	Distillate (566 °C ⁻)	312.52	
	Residue (566 °C ⁺) (c)	107.69	25.3 %
	TOTAL	420.21	98.6 %
	Losses (d)	6.17	1.4 %

(a) daf = SO₃-free basis

(b) Representative of the three runs

(c) 6% boiling below 1050 °F

(d) Assumed to be distillate lost in vacuum system and that all the residue is collected

TABLE 6
SUMMARY OF 300 ML CSTR TESTS

Run No.	15oa-1, 300 CSTR [R7-111-1]	DARo-1, 300 CSTR [R7-167-1]	23oac-1, 300 CSTR [R7-181-1]
Feedstock	15-LA, 15a-LA composited filtrate resid	Wilsonville Run 258A deashed resid	23-LA, 23a-LA, 23c-LA composited filtrate resid
Feed, g	50	100	94
Catalyst (Molyvan L), g	6.2	12.3	11.6
DMDS, g	4.8	6.9	7.2
Time, min	60	60	60
Temperature, °C	440	440	440
566°C+ in feed, wt % MAF feed	89.0	75.7	93.8
566°C+ resid conversion, wt % MAF feed	49.2	63.3	57.3
C ₁ -C ₃ gas, wt % MAF feed	22.6	12.6	17.3
C ₁ -C ₃ gas yield/gram resid converted	51.6	26.3	32.2
H ₂ uptake (by diff), mg/g MAF feed (a)	54	18	29
H ₂ uptake/gram resid converted	123	38	54
H ₂ balance (uptake basis), % (b)	99	110	107
Fixed carbon ratio, out/in (c)	2.7	1.6	0.8
Material Balance Data, wt %			
C	101	100	95
H	99	105	103
N	97	96	98
S	100 (d)	86	60
O	89	177	101
ash	162	61	113
Total	102	100	96

- (a) Includes hydrogen associated with the decomposition of DMDS
(b) Excludes hydrogen gas calculated as transient in reactor
(c) Fixed carbon in non-distillate product/fixed carbon in feed
(d) Forced sulfur balance, based on DMDS addition

TABLE 7
MICROAUTOCLAVE HYDROTREATING RESULTS
FEEDSTOCK: 15OA-LA (a, b, c)

Second Stage Run No.	Hydrotreat Time, min	566 °C⁺ Resid Conversion, wt % MAF resid	C₁-C₃ Gas Yield, wt % Feed	C₁-C₃ Gas Yield per g Resid Converted	H₂ Uptake, mg/g MAF Feed	H₂ Uptake per g Resid Converted
15oa-3, 50 MA [R7-097-1]	30	38.7	6.6	18.2	38	104
15oa-1, 50 MA [R7-064-1]	60	44.1	8.7	21.2	43	105
15oa-4, 50 MA [R7-099-1]	90	49.7	10.6	22.8	56	120
15oa-2, 50 MA [R7-091-1]	120	51.0	12.1	25.3	67	140

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

(b) 93.5 wt % (MAF feed basis) 566 °C⁺ to the hydrotreater, each run.

(c) Source: First-stage tests Run 15-LA and 15a-LA, Black Thunder Mine Coal, Reilly Industries anthracene oil, 375 °C, 60 min

Filtration test NCF 57 FDR

High-Temp SIMDIS Analysis with Aromatic Correction

High-Temp SIMDIS Analysis with Paraffin Calibrant

High-Temp SIMDIS Analysis with Aromatic Correction

High-Temp SIMDIS Analysis with Aromatic Correction

Note: found recovery increased by 6.23% for retained THF

Figure 1. Simulated Distillation Curves.

High-Temp SIMDIS Analysis with Paraffin Calibrant

High-Temp SIMDIS Analysis with Aromatic Correction

High-Temp SIMDIS Analysis with Aromatic Correction

High-Temp SIMDIS Analysis with Aromatic Correction

Figure 1. Simulated Distillation Curves (Continued).

High-Temp SIMDIS Analysis with Paraffin Calibrant

High-Temp SIMDIS Analysis with Aromatic Correction

High-Temp SIMDIS Analysis with Aromatic Correction

High-Temp SIMDIS Analysis with Aromatic Correction

Figure 1. Simulated Distillation Curves (Continued).

Resid Conversion Kinetics

440 C, 2% H₂S, 10.1 MPa (1450 psig) total charge pressure

*3g DAR with 0.1% Mo in Molyvan L

**2g NCF 57 FDR (15La) resid with 1% Mo in Molyvan L

rka 4-30-97

Figure 2. Resid Conversion Kinetics.

Hydrogen Requirements

440 C, 2% H₂S, 10.1 MPa (1450 psig) total charge pressure

2g NCF 57 FDR (15La) resid with 1% Mo in Molyvan L

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Figure 3. Hydrogen Requirements.