

The present paper reviews the operation of a 0.6 ton/day process development unit (PDU) for continuous liquefaction of lignite at 1500-2500 psig and 700-950°F in an atmosphere of synthesis gas in the presence of a hydrogen donor solvent. Solvent recovery and solvent recycle capabilities are provided as well as process gas clean up and recycle. Ash is removed by dilution of vacuum flash bottoms with toluene followed by settling to remove ash and unreacted lignite. The deashed solvent refined lignite (SRL) is recovered by distilling the toluene for recycle from the extract. The SRL, a solid at room temperature, is low in ash, has a reduced sulfur content, a high calorific value, and would be environmentally acceptable for particulate and SO_x emissions upon combustion.

Hydrogenating sufficiently to produce SRL at high conversion can be done in an atmosphere of synthesis gas by making use of the natural catalyst activity of the lignite without the use of very high pressures. The SRL is a superior raw material for additional hydrogenation to liquids for fuels or as a source of organic chemicals. While the natural catalytic activity favors initial hydrogenation solution, increased hydrogenation can be better accomplished using a specific hydrogenation catalyst. Removal of ash constituents which could poison the hydrogenation catalyst makes the concept of two stage hydrogenation attractive in that both steps can be optimized and more effective overall hydrogenation can be achieved.

PROCESS DEVELOPMENT UNIT

The Project Lignite Process Development Unit has a nominal design capacity of 50 pounds of raw lignite feed per hour and produces approximately 15 pounds per hour of solvent refined lignite (SRL) with a melting point of 300-400°F as well as additional quantities of lighter liquids and gases.

Pulverized lignite of full moisture content is slurried with solvent. The slurry is pressurized, preheated and reacted at selected temperature and pressure in a reducing gas atmosphere. The products are then separated as gases, liquids, and SRL from the unreacted coal and mineral matter. A simplified block flowsheet of the process is presented in Figure 1.

Asreceived lignite is first split into representative samples using a rail car-size sample splitter and stored in nitrogen-purged bins until needed. The lignite is crushed and pulverized to 100 percent passing 60 mesh and 90 percent passing 200 mesh Tyler screens. After pulverization, the lignite is stored in 55-gallon drums prior to slurry preparation.

The pulverized lignite is blended in a slurry mixing tank

with recycle liquefaction solvent. For startup, a petroleum-derived solvent having a boiling range of 212° to 446°F at 1.6 mm Hg absolute is used. The slurry is continuously pumped through a recirculating loop to maintain suspension. A side stream is pumped to high pressure, mixed with hydrogen and/or carbon monoxide gas, and is heated to reaction temperature in an electrically heated, fluidized-solids preheater. Most operations are in the range of 700°F to 850°F at 1500-2500 psi. Upon leaving the preheater, the mixture enters the dissolvers (R-1A and R-1B) where reaction temperatures are maintained and sufficient residence time provided for solvation. The two dissolvers can be used in series or separately. Operations are usually with a single dissolver. The undissolved material consists of unreacted lignite and inorganic mineral matter. Upon leaving the dissolvers, the non-condensable gases and light hydrocarbon vapors are separated from the slurry in a series of five separation vessels in which pressure and temperature are decreased in stages.

The PDU is designed so that solvent can be recycled without operating the solid-liquid separation area. In this mode of operation, the slurry after gas separation and pressure letdown is heated to approximately 600°F in a Dowtherm preheater followed by vacuum flash distillation. Volatile liquids and solvent are separated from the vacuum bottoms which contain the mineral residue, undissolved lignite and solvent refined lignite. The overhead stream is condensed, combined with another light liquid stream from the primary separators, and fed to a second Dowtherm preheater preceding the main solvent recovery fractionators. In the fractionator, light ends, liquefaction solvent and heavy ends are separated. The liquefaction solvent is recycled.

The non-condensable gases from the primary high pressure separators are channelled through the gas recovery area; carbon dioxide and hydrogen sulfide are removed, and a portion of the hydrogen and carbon monoxide gas is recycled.

When the solid-liquid separation system is in use, an initial partial vacuum flash vessel (F-0) is used to remove part of the liquefaction solvent from the separator effluent. The bottoms are pumped to a mixing-surge vessel and mixed with toluene (2:1 by volume). The mixture is pumped to a gravity settling tower (V-8) operating above the critical pressure but slightly below the critical temperature of toluene. Additional wash toluene is fed counter-currently to the direction of settling. The dissolved SRL, the remainder of the liquefaction solvent and toluene are withdrawn from the top of the settler and passed into a vessel (V-9) in which the toluene is removed by flash distillation. The toluene is recycled and the SRL plus liquefaction solvent recovered. The SRL solvent stream is passed through Dowtherm preheater E-11 and into the vacuum flash drum F-1. The SRL is recovered as the bottoms from this vessel and the overhead liquefaction solvent stream is combined with the partial flash (F-0) overhead as feed to the light ends column (F-2). The F-2 bottoms are sent back to the slurrymix tank as recycle solvent.

TYPICAL OPERATING CONDITIONS AND RESULTS

During steady operation of the PDU, which is normally operated continuously for a five-day week, material balances are made over yield periods of twelve hours duration. Fresh containers are used for the products during a yield period, and samples and operating data are collected throughout the period.

In the following discussions, material balance and yield data are presented for Run Period M-17A, flows being expressed in pounds per hour averaged over the twelve hour yield period. Test conditions for this run are close to the optimum obtained thus far. The solid-liquid separation unit using toluene as deashing solvent was also in operation during this run.

Operating rates and stream ratios are given in Table 1. Lignite at 45 lb/hr was slurried with 81 lb/hr recycle solvent and mixed with 531 SCFH of feed gas containing 76 percent hydrogen and 24 percent carbon monoxide. In the solid-liquid separations area, 112 lb/hr of toluene was used to extract product SRL and liquefaction solvent from unconverted solids.

Operating temperatures and pressures for various process vessels, identified as coded in the flow sheet (Figure 1), are presented in Table 2. Operating pressure in preheater and dissolver was 2500 psig, and slurry left the preheater at 747°F and reached 802°F in the dissolver. There is usually a 50 to 100°F temperature rise in the dissolver due to the exothermic overall reaction. A single reactor (R-1A) having a reaction volume of 1.42 cu ft was used. In ash separation, the toluene settling tower was operated at 378°F and 300 psig, and the vacuum flash unit maintained at 626°F and 15 Torr.

Source and characteristics of lignite, reacting gases and starting solvent are given in Table 3. The lignite was charged at 31.5 percent moisture. The as-mined moisture was probably around 37 percent, and transportation, crushing and handling were done in such a way as to minimize moisture loss. Feed gas was supplied from tank trailers of pure hydrogen and carbon monoxide and mixed just before entering the compressor. The startup solvent was Gulf FS-120, a petroleum derived catalytic cracker recycle stock. Comparison of its boiling ranges fractions with those of recycle solvent during the run shows a tendency of the solvent to lighten on continued recycle.

The gross input and output material balances are shown in Figure 2, in which the magnitude of the two recycle streams are compared with the coal and gas processed. For each pound of input lignite, 0.26 lb of syngas, 1.8 lb of liquefaction solvent and 2.5 lb of toluene deashing solvent were circulated.

The net material balance based on lignite and syngas fed is shown in Figure 3. In this run, per 100 pounds of MAF lignite charged, 54 pounds of SRL and 13 pounds of light organic liquids were produced.

Table 4 gives net yields of products both in lb/hr and in lb/100 pounds MAF lignite. There is a net consumption of 5.9 percent of water, and 67.3 percent of the MAF lignite is converted to SRL and light organic liquids. Conversion was 89.4 percent although conversions as high as 95 percent have been obtained in some runs.

Table 5 presents gas amounts in and out as volume percent gas composition and as lb/hr. Note that 38 percent of the entering CO was consumed as compared to only 14 percent of the entering hydrogen, and that 0.67 moles of CO₂ appear in the exit gas per mole of CO charged, or 1.77 moles of CO₂ per mole of CO consumed.

Some of the characteristics of the product SRL are given in Table 6. In Run 17-A, flow rates were satisfactory in the solid-liquid separations system, but problems with the solids removal system in the settling tower V-8 prevented removal of the unconverted coal and mineral matter, which therefore carried over to some extent into the SRL product. Thus in Table 6 actual vacuum bottoms composition and properties are shown, along with the analyses that would have been obtained had the solids been properly removed.

Ultimate analysis of lignite, starting and recycle solvent, and SRL product are shown in Table 7. The coal-derived material contains a substantial amount of oxygen, 5.7 percent in the SRL and 5.1 percent in the recycle solvent. The startup solvent has built up from 0.5 percent oxygen to 5.1 percent in the recycle solvent, showing that compounds formed from the coal have replaced much of the starting solvent. A large amount of this oxygen is present as phenolic compounds.

CORRELATIONS OF OPERATING VARIABLES

Data from 18 runs, from the series of runs M-1 through M-23 that resulted in usable yield periods, are summarized in Tables 8, 9, and 10. Correlations and curves developed from these data are presented in Figures 4 through 11, using symbols identified in the tables.

The influence of maximum dissolver temperature on yield characteristics is presented in Figure 4 at a 1/1 H₂/CO ratio, 2500 psi, and for a limited range of liquid hourly space velocities and solvent to lignite ratios. Conversion, as to be expected, increased with increased temperature. However, yield of light oil plus SRL decreased slightly over the temperature range investigated. The major change was in increased production of gases indicating additional degradation of the lighter fractions. Little change was evident in utilization of moisture by reaction.

The slopes of the lines of Figure 4 were used to establish temperature coefficients which were then used to adjust all yield data to the same maximum dissolver temperature of 826°F. These adjusted data are plotted in Figure 5 versus liquid hourly space velocity. Increased liquid space velocity corresponds to reduced residence time of the lignite-solvent slurry in the dissolvers. Consequently, conversion is reduced, although only slightly, for about a 40 percent increase in space velocity. Less degradation of liquefaction products is indicated by an increase in oil yields and a decrease in gaseous products. Water consumption was nearly constant for the conditions tested.

The data of Figure 5 were further adjusted to a constant LHSV of 1.425, then plotted as a function of solvent/coal ratio in Figure 6. Yields increased uniformly with increasing solvent/lignite ratio from 1.6 to 2.3. Consumption of water as a "hydrogenation" source also increased. Over the range of gas charge rates used, there appeared to be no significant effect of either gas rate or gas to coal ratio.

Sufficient data have been accumulated at 2500 psig and a 1/1 H₂/CO ratio to utilize regression analysis to obtain equations relating yields to the operating variables of maximum reactor temperature, liquid hourly space velocity, and solvent/MAF lignite weight ratio. These equations should be quite useful in visualization of the effects of process variables, and are summarized on the following page.

Percent Net Gas = $204.971X_1 - 15.110X_2 + 8.835X_3 - 228.615$

Percent Net Oil = $-26.505X_1 - +8.450X_2 + 8.272X_3 + 71.759$

Percent Net H₂O = $-37.225X_1 - -4.461X_2 - 9.724X_3 + 63.617$

$X_1 = \text{Temperature Variable, } \frac{\text{Temp., } ^\circ\text{F} + 460}{1000}$

$X_2 = \text{LHSV, Hr}^{-1}$

$X_3 = \text{Solvent/MAF Coal Weight Ratio}$

Solvent recovery is critical to the liquefaction process. Suitable solvent must be generated from the coal substance during liquefaction and recovered to replace losses. Information concerning solvent recovery is given in Figure 7 as a function of maximum dissolver temperature. Shown in the lower half of Figure 7 is the total solvent accounted for. The line represent the average of the calculated total solvent production at 2500 psig. The total is over 100 percent, indicating that a net production of solvent range material is possible. However, the effective solvent, i.e., that boiling from about 550°F to 850°F equivalent temperature at atmospheric pressure, seems to reach a maximum with a slight net production around 820°F maximum dissolver temperature, but falls below 100 percent at temperatures below 800°F or above 835°F, as shown in the upper half of Figure 7.

The runs made at lower pressures, either 1500 or 2000 psig, regardless of H₂/CO ratio, result both in lower total oil and in lower effective solvent production. In all cases the yields were less than 100 percent of the solvent charged which indicates a net solvent consumption, probably because of higher conversion to gas. With other ratios of H₂/CO at 2500 psig the yields were not greatly different from those with a 1/1 H₂/CO ratio. With 1/3 H₂/CO ratio, the effective solvent recovered was about the same as with 1/1, though the total oil was slightly less, because of relatively less light oils. With 3/1 H₂/CO the total oil recovery was greater than with 1/1, though the effective solvent recovery was less because of increased production of light oils.

The data for runs at 3/1 H₂/CO ratio, adjusted to a constant dissolver temperature, LHSV and solvent to coal ratio are presented in Figure 8. The pressure has little effect on gas yield, but increasing pressure results in increasing conversion and even more rapidly increasing oil yields. At lower pressures there is a net make of water, but at higher pressures water is consumed, the consumption increasing rapidly with pressure. This results in increasing yields of light oil and SRL.

The solvent recovery data for the 3/1 H₂/CO runs are presented in Figure 9. At this ratio effective solvent recovery is less than 100 percent even at 2500 psig, though the total oil recovery is over 100 percent because of the relatively

higher light oil yield obtained at higher hydrogen concentration.

The yields resulting from several runs made at 2500 psig and varying H₂/CO ratio were adjusted to constant parameter values as indicated in Figure 10 and plotted against mole percent hydrogen in the charge gas. The data indicate an optimum at about 50 to 60 percent hydrogen. One run made with pure hydrogen (M-20) resulted in relatively low conversion and oil yield. However, this run was made at such a high temperature that coking and/or repolymerization may have significantly affected the results.

Solvent recovery data are similarly plotted versus mole percent hydrogen in Figure 11. The optimum also appears to be near 60 percent hydrogen.

The use of lignite containing its natural moisture (about 30 weight percent as charged to the PDU) and synthesis gas containing carbon monoxide results in substantial production of carbon dioxide. The product gas also contains light hydrocarbons resulting from the breakdown of the coal substance and of the solvent. The yields of carbon dioxide and of C1 to C3 hydrocarbons seem to be essentially linear functions of maximum dissolver temperature for the 2500 psig 1/1 H₂/CO operations. Variables other than temperature seem to have little effect. The yields of C1 to C3 hydrocarbons range from almost zero at 760°F to about 12 weight percent of the MAF lignite at 880°F, while CO₂ yields range from about 12 weight percent of the MAF lignite to 65 percent over the same temperature interval. Lower pressures and 3/1 or 1/3 H₂/CO ratios have little effect on C1 to C3 hydrocarbon yields. At the higher temperatures decreasing the amount of CO decreases CO₂ yields, while increasing CO increases CO₂ yields. One run made with pure hydrogen resulted in a very low CO₂ yield and a high gas yield, but again this was at such a high temperature level that other effects may also have become significant.

It now appears that for this particular reactor configuration and limitations, optimum results will be obtained at 2500 psig with 50 to 60 percent hydrogen in the charge gas using one reactor corresponding to a liquid (slurry) hourly space velocity of about 1.4. The effects of solvent/coal ratio have not been evaluated completely, but it appears that the lower the ratio the better the solvent recovery, which means that the thickest slurry that can be handled should be charged.

PLANS FOR FUTURE WORK

The Project Lignite work has been extended for an additional year to continue the development of the process. The effort will be concentrated on proving the operability of the unit in runs of longer duration and in evaluating the solid-liquid separations system.

A series of three one-month long runs is planned over a 6-months period to establish the effects of continuing to recycle the lignite-derived process solvent and demonstrate the self-sufficiency of the process with respect to liquefaction solvent. The gas cleanup and recycle section will be operated on at least one extended run to determine makeup gas requirements and the effect of any impurities remaining in the recycle gas. The solid-liquid separations system will be operated integrally with the PDU to produce clean SRL.

As a part of this program, prestressed test coupons will be placed in process streams to determine their susceptibility to corrosion in the process environment. An attempt will also be made to study the buildup of solids in the dissolver, a problem that has serious implications for long-term steady operation of the PDU. The rate of buildup and composition will be determined and means of alleviating the problem will be considered.

The long runs should also provide a means of producing substantial amounts of clean SRL for evaluation by other ERDA contracts for various end uses.

A longer range goal of the investigation, not included in the current extension, is the hydrotreating of SRL to hydrocarbon liquids. This has been carried out in the laboratory but removed from the PDU program. Certain other programs should also be carried out including modified dissolver design, hydrotreating of recycle solvent, and testing of other coals in the process. In general, the PDU is a useful developmental tool that could test many innovations in process technology in the upgrading of lignite.

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TABLE 1 - OPERATING RATES AND RATIOS

Lignite (31.5% Moisture)	44.75 lb/hr
Gas (24% CO, 76% H ₂)	11.54 lb/hr
	531 SCFH
Liquefaction Solvent	81.3 lb/hr
Deashing Solvent (Toluene)	111.9 lb/hr
Ratios and Space Velocities	
Slurry rate, LHSV	1.34 hr ⁻¹
Gas Rate (in SCF)	374 hr ⁻¹
Solvent/coal ratio	1.82
Toluene/slurry ratio	3.6 (approx.)

TABLE 2 - OPERATING TEMPERATURES AND PRESSURES

	Temp ^o F	Pressure, psig
Dissolver Preheater E-1 Inlet	289	2500
Outlet	747	2500
Dissolver R-1A from Bottom 3 ft	752	2500
7 ft	779	2500
11 ft	793	2500
15 ft	802	2500
Separators S-1	574	2500
S-2	76	2500
S-3	479	250
S-4A	78	60
Partial Flash F-0 Overhead	471	15 Torr
Bottoms	340	15 Torr
Vacuum Flash F-1 Vapor	554	15 Torr
Upper Wall	626	15 Torr
Solids-Liquids Settling Tower V-8	378	300 psig
Toluene Recovery Tower V-9	444	2 psig

TABLE 3 - FEED MATERIALS

Lignite

North American Coal Co., Zap, N.D.
 Screen size 90%-200 mesh, 100%-60 mesh
 Moisture: 31.5%

Ultimate Analysis (% Wt)	
C	46.21
H	6.42
N	0.66
S	0.44
O (diff)	40.54
Ash	5.73

Feed Gas (mixed from tank trailers)

	% Vol.	% Wt.
CO	24.0	81.4
H ₂	76.0	18.6

Startup liquefaction solvent

Gulf Oil Co. FS-120 cat. cracker recycle stock	FS-120	Recycle
Boiling range (1.6 Torr)	Startup	Solvent
0-100°C	5.0%	30.0%
100-230°C	83.6	57.7
230-255°C	11.4	11.8
Specific Gravity	1.043	0.988

TABLE 4 - NET YIELD OF PRODUCTS

	<u>lb/hr</u>	<u>lb/100 lb MAF Lignite</u>
Gas	7.87	28.0
Light Oil (0-100°C at 1.6 Torr)	1.42	5.1
Liquefaction Solvent (100-230°C at 1.6 Torr)	1.71	6.1
Heavy Oil (230°-255°C at 1.6 Torr)	0.56	2.0
SRL	15.21	54.1
Unreacted Lignite	2.99	10.2
Water Consumed	1.67	5.9

TABLE 5 - GAS MATERIAL BALANCE FOR GAS COMPONENTS

	<u>Vol % In</u>	<u>Vol % Out</u>	<u>lb/hr In</u>	<u>lb/hr Out</u>
H ₂	76.2	66.2	2.15	1.84
CO	23.8	14.7	9.39	5.79
CO ₂	-	13.8	-	9.93
H ₂ S	-	0.1	-	0.13
CH ₄	-	3.8	-	0.93
C ₂ H ₆	-	1.0	-	0.52
C ₃ H ₈	-	0.2	-	0.22
NH ₃	-	0.03	-	0.04

TABLE 6 - ANALYSIS OF PRODUCT SRL

	F-1 Vacuum Bottoms (<u>measured</u>)	SRL (<u>calculated</u>)
Boiling Range Fraction, wt % 255°C (1.6 Torr)	0	0
SRL	77.22	100.0
Unconverted Coal, wt %	15.30	0
Ash, wt %	7.48	0
Gradient Bar Melting Point °F	327°	-
Pyridine Solubles, wt% ash-free	83.5	100
Specific Gravity	1.28	1.25
Heat of Combustion, Btu/lb	14,330	15,990

TABLE 7 - ULTIMATE ANALYSIS OF MATERIALS

	<u>Lignite Charged</u>	<u>Starting Solvent</u>	<u>Recycle Solvent</u>	<u>Vacuum Bottoms</u>	<u>Deashed SRL</u>
C	46.21	89.05	83.56	80.09	86.56
H	6.42	8.12	9.84	5.26	5.69
N	0.66	0.14	0.21	0.95	1.03
S	0.44	2.19	1.27	0.94	1.02
O*	40.54	0.50	5.12	5.28	5.70
Ash	5.73	0	0	7.48	0

*By difference

Table 8 - Summary of Liquefaction Tests (1/1 H₂/CO)

Yield Period Symbols	M-1C		M-2A		M-2C		M-3B		M-5A		M-5B		M-5C		M-5D		M-8C		M-9C			
	●	☉	☉	☉	☉	☉	☉	☉	☉	☉	☉	☉	☉	☉	☉	☉	☉	☉	☉	☉		
Hours on Coal at End of Y.P.	62	25	73	38	19	43	55	67	39	43	39	43	39	43	39	43	39	43	39	43	43	
During Run	62	87	135	173	227	251	263	275	358	227	251	263	275	358	227	251	263	275	358	227	251	
Cumulative	2	2	2	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	
No. of Reactors	53	53	61	50	58	47	46	51	54	58	47	46	51	54	58	47	46	51	54	58	47	
Coal Charged, lbs/hr	471	458	454	414	399	426	483	379	449	399	426	483	379	449	399	426	483	379	449	399	426	
Gas Charged, SCF/hr	2.30	2.18	2.06	1.89	1.59	1.80	1.92	1.97	1.62	1.59	1.80	1.92	1.97	1.62	1.59	1.80	1.92	1.97	1.62	1.59	1.80	
Solvent/Coal Ratio	0.90	0.86	0.95	1.48	1.33	1.33	1.38	1.51	1.40	1.33	1.33	1.38	1.51	1.40	1.33	1.33	1.38	1.51	1.40	1.33	1.33	
LHSV, Hr ⁻¹	164	160	159	289	279	299	334	265	314	279	299	334	265	314	279	299	334	265	314	279	299	
GHSV, Hr ⁻¹	18.6	18.5	21.2	35.2	40.8	32.6	32.4	36.0	37.5	40.8	32.6	32.4	36.0	37.5	40.8	32.6	32.4	36.0	37.5	40.8	32.6	
Coal Rate, Lbs/Hr/CF	8.8	8.6	7.5	8.2	6.8	9.2	10.4	7.4	8.4	6.8	9.2	10.4	7.4	8.4	6.8	9.2	10.4	7.4	8.4	6.8	9.2	
Gas Rate, SCF/Lb Coal	2.9	2.9	1.2	2.4	0.5	0.8	1.5	1.9	1.3	0.5	0.8	1.5	1.9	1.3	0.5	0.8	1.5	1.9	1.3	0.5	0.8	
Gas Consumed, SCF/Lb Coal																						
Yields, Wt % MAF Coal																						
Net Gas	10.4	12.4	9.7	25.2	35.6	35.5	36.7	32.9	28.2	35.6	35.5	36.7	32.9	28.2	35.6	35.5	36.7	32.9	28.2	35.6	35.5	
Net Oil	58.1	69.1	50.2	58.3	55.9	62.0	68.0	65.1	61.5	55.9	62.0	68.0	65.1	61.5	55.9	62.0	68.0	65.1	61.5	55.9	62.0	
(Light Oil)					(8.9)		(9.6)	(11.0)	(4.9)	(8.9)		(9.6)	(11.0)	(4.9)	(8.9)		(9.6)	(11.0)	(4.9)	(8.9)		
(SRL)	(58.1)	(69.1)	(50.2)	(58.3)	(47.0)	(62.0)	(58.4)	(54.1)	(56.6)	(47.0)	(62.0)	(58.4)	(54.1)	(56.6)	(47.0)	(62.0)	(58.4)	(54.1)	(56.6)	(47.0)	(62.0)	
Net H ₂ + Ash	3.7	-0.6	2.7	-2.6	-7.5	-8.8	-14.1	-12.0	-4.4	-7.5	-8.8	-14.1	-12.0	-4.4	-7.5	-8.8	-14.1	-12.0	-4.4	-7.5	-8.8	
Unconv. MAF Coal	27.8	19.1	37.4	19.1	16.0	11.3	9.4	14.0	14.6	16.0	11.3	9.4	14.0	14.6	16.0	11.3	9.4	14.0	14.6	16.0	11.3	
Solvent Recycle, %																						
Actual	85.9	64.1	87.2	83.1	90.2	93.5	94.7	95.1	99.9	90.2	93.5	94.7	95.1	99.9	90.2	93.5	94.7	95.1	99.9	90.2	93.5	
Possible (Calc.)	98.6	97.2	96.4	98.0	103.7	99.6	103.3	103.7	102.0	103.7	99.6	103.3	103.7	102.0	103.7	99.6	103.3	103.7	102.0	103.7	99.6	
Material Balance, %	100.0	103.4	97.7	98.8	94.7	97.8	95.5	97.8	98.1	94.7	97.8	95.5	97.8	98.1	94.7	97.8	95.5	97.8	98.1	94.7	97.8	
Conditions																						
Temperatures, °F																						
Preheater Outlet	702	733	698	746	754	748	750	742	730	754	748	750	742	730	754	748	750	742	730	754	748	
Dissolver Inlet	-	-	-	708	714	663	738	708	731	768	731	768	731	768	731	768	731	768	731	768	731	768
R-1A 3 ft	708	714	663	738	714	663	738	714	663	738	714	663	738	714	663	738	714	663	738	714	663	
R-1A 15 ft	748	760	699	797	748	760	699	797	748	760	699	797	748	760	699	797	748	760	699	797	748	
R-1B 3 ft	716	735	675	786	716	735	675	786	716	735	675	786	716	735	675	786	716	735	675	786	716	
R-1B 15 ft	735	752	690	804	735	752	690	804	735	752	690	804	735	752	690	804	735	752	690	804	735	
Dissolver Outlet	-	-	-	804	886	826	845	850	-	886	826	845	850	-	886	826	845	850	-	886	826	
Flash Preheat	594	583	552	626	603	627	626	627	608	603	627	626	627	608	603	627	626	627	608	603	627	
Flash Vapor	572	538	528	568	574	544	538	537	575	574	544	538	537	575	574	544	538	537	575	574	544	
Pressures, psig																						
Dissolver	1500	1500	1500	2000	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500	2500
Inter-Sep.	350	350	312	300	250	250	250	250	250	250	250	250	250	250	250	250	250	250	250	250	250	250
Vac. Flash, Torr	10	6	6	14	12	10	10-15	11	10	12	10	10-15	11	10	12	10	10-15	11	10	12	10	
S-LS System in Use?	No	Yes																				

Table 10 - Summary of Liquefaction Tests

Yield Period	M-16A	M-17A	M-17B	M-20A	M-21A	M-21B	M-21D	M-21E	M-22A	M-23A	M-23B
Hours on Coal at End of TP	19	29	41	19	24	36	76	88	24	27	47
During Run	914	950	962	1044	1071	1083	1123	1135	1159	1199	1219
Cumulative	1	1	1	1	1	1	1	1	1	1	1
No. of Reactors	48	45	37	48	47	46	48	45	48	49	49
Coal Charged, Lbs/Hr	435	535	537	478	630	605	612	608	558	554	559
Gas Charged, SCF/Hr	1.76	1.81	2.16	1.84	1.94	1.87	1.87	1.98	1.89	1.80	1.76
Solvent/Coal Ratio	1.43	1.34	1.27	1.41	1.34	1.39	1.40	1.38	1.44	1.41	1.40
LHSV, Hr ⁻¹	305	374	376	334	440	424	429	425	390	387	390
GHSV, Hr ⁻¹	33.9	31.3	25.7	33.8	32.7	32.4	33.5	31.6	33.8	34.1	34.1
Coal Rate, Lbs/Hr/CF	9.0	12.0	14.6	9.9	13.5	13.1	12.8	13.5	11.6	11.4	11.5
Gas Rate, SCF/lb Coal	3.5	2.4	2.6	4.5	3.6	3.0	3.7	3.2	1.2	1.6	1.1
Gas Consumed, SCF/lb Coal											
Yields, Wt % MAF Coal											
Net Gas	39.2	28.0	35.7	41.8	28.7	35.2	30.8	36.5	25.5	27.5	30.3
Net Oil	55.5	67.3	63.7	14.9	51.1	58.4	57.4	60.7	42.3	34.8	36.4
(Light Oil)	(2.3)	(13.1)	(13.3)	(-29.8)	(-12.5)	(-0.9)	(-3.0)	(1.2)	(-14.7)	(-23.1)	(-11.4)
(SRL)	(53.2)	(54.2)	(50.4)	(44.7)	(63.6)	(59.3)	(60.4)	(59.5)	(57.0)	(57.9)	(47.8)
Net H ₂ O and Ash	-10.2	-5.9	-5.6	5.1	5.7	5.5	-1.4	-7.8	6.8	-0.2	2.6
Unconv. MAF Coal	15.5	10.6	6.2	38.2	14.7	12.1	13.2	10.6	25.4	37.9	30.7
Liquid Recycle, %											
Actual	98.8	103.5*	100.6*	80.1	88.4	72.5	67.2	68.3	93.1	86.7	92.2
Possible (Calc.)	100.9	104.6	103.8	88.7	95.8	99.7	99.0	100.4	94.8	92.0	95.9
Solvent (Ex. Lt. Oil) Possible	94.6	104.0	94.6	82.5	89.1	92.0	95.4	94.4	90.5	86.4	93.1
Material Balance, %	96.7	100.1	97.9	98.1	101.5	95.2	101.9	99.6	97.8	99.6	97.0
Conditions											
Temperatures, °F											
Preheater Outlet	744	747	745	787	741	743	746	743	752	764	774
Dissolver Inlet											
R-1A 3 ft	NIU	752	774	830	NIU	740	746	740	748	741	746
R-1A 15 ft	NIU	802	826	877	NIU	NIU	NIU	NIU	740	NIU	NIU
R-1B 3 ft	784	NIU	NIU	NIU	NIU	NIU	NIU	NIU	787	NIU	NIU
R-1B 15 ft	858	NIU	NIU	NIU	NIU	NIU	NIU	NIU	NIU	791	791
Dissolver Outlet	849	NIU	NIU	876	NIU	832	828	822	765	831	830
Flash Preheat	633	639	660	644	639	646	646	649	625	646	637
Flash Vapor	584	554	550	472	515	513	525	525	504	524	522
Pressures											
Dissolver, psig	2500	2500	2500	2500	2000	2000	2000	2000	1500	1500	1500
Inter. Sep. psig	250	250	250	250	250	250	260	250	230	230	230
Vac. Flash, Torr	15	15	15	15	15	15	15	15	15	15	15
S-L/S System in Use ?	No	Yes	Yes	No	No	No	Yes	Yes	No	No	No
H ₂ /CO Ratio	1/3	3/1	3/1	Pure H ₂	3/1	3/1	3/1	3/1	3/1	3/1	3/1
CO ₂ Yield, Hr % MAF Coal	67.8	35.4	43.8	16.9	41.1	45.0	41.8	47.0	25.2	30.7	32.1
Cl-C ₃ Yield, Hr % MAF Coal	15.7	5.9	10.6	27.3	6.9	7.4	6.7	7.4	7.2	8.6	8.9
*From SLS Solvent, %	-	3.9	6.3	-	-	-	-	-	-	-	-

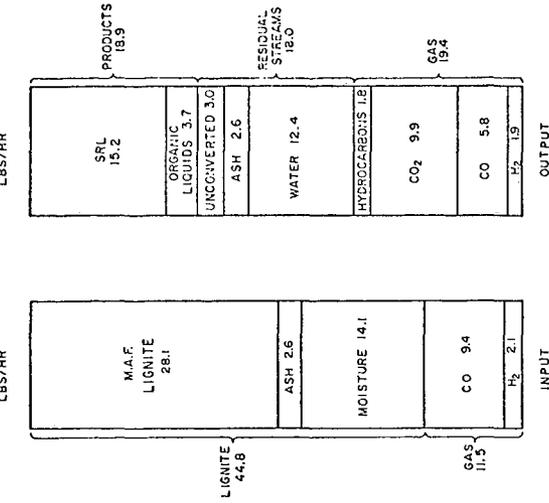


FIGURE 3-NET MATERIAL BALANCE, RUN 17A

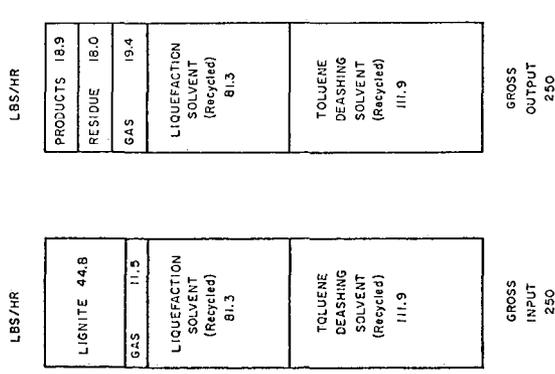


FIGURE 2-GROSS MATERIAL BALANCE, RUN 17A

FIGURE 4

CONVERSION AND YIELDS VS. MAXIMUM DISSOLVER TEMPERATURE
 (1.0 H₂/CO RATIO, 2500 psig, 1.27-1.60 LHSV, 1.59-2.26
 SOLVENT/COAL RATIO)

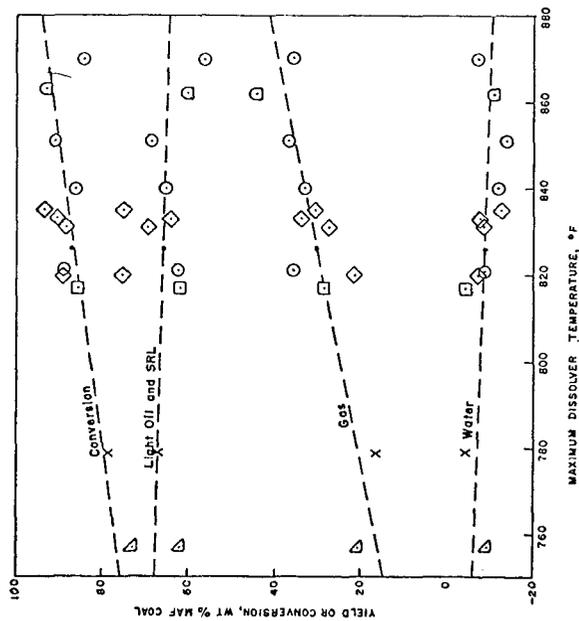


FIGURE 5

CONVERSION AND YIELDS ADJUSTED TO 826° F
 MAXIMUM DISSOLVER TEMPERATURE VS. LHSV
 (1.0 H₂/CO RATIO, 2500 psig, 826° F, 1.59-2.26
 SOLVENT/COAL RATIO)

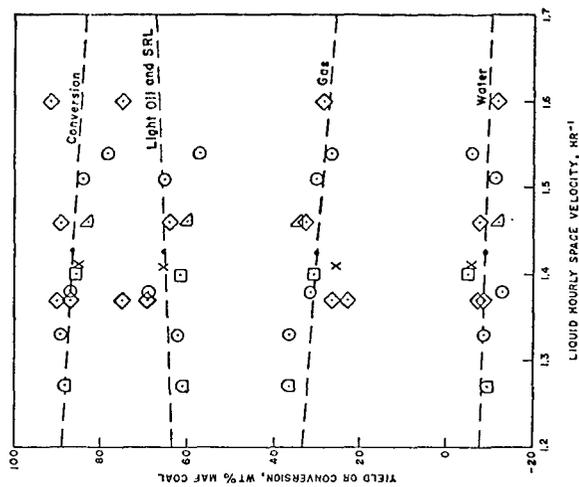


FIGURE 6

CONVERSION AND YIELDS ADJUSTED TO 826° F
 MAXIMUM DISSOLVER TEMPERATURE AND 1.425 LHSV
 VS. SOLVENT/COAL RATIO

(1.0 H₂/CO RATIO, 2500 psig, 826°F, 1.425 LHSV)

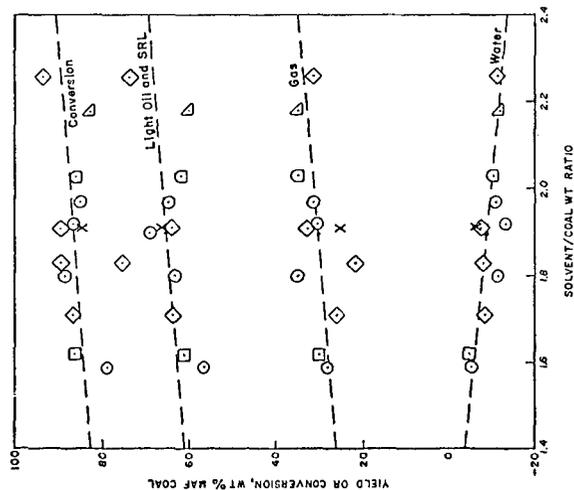


FIGURE 7

SOLVENT RECOVERY vs. MAXIMUM DISSOLVER TEMPERATURE
 2500 psig, 1/1 H₂/CO Ratio, ~ 1.4 LHSV, ~ 1.9 Solvent/Coal
 Ratio

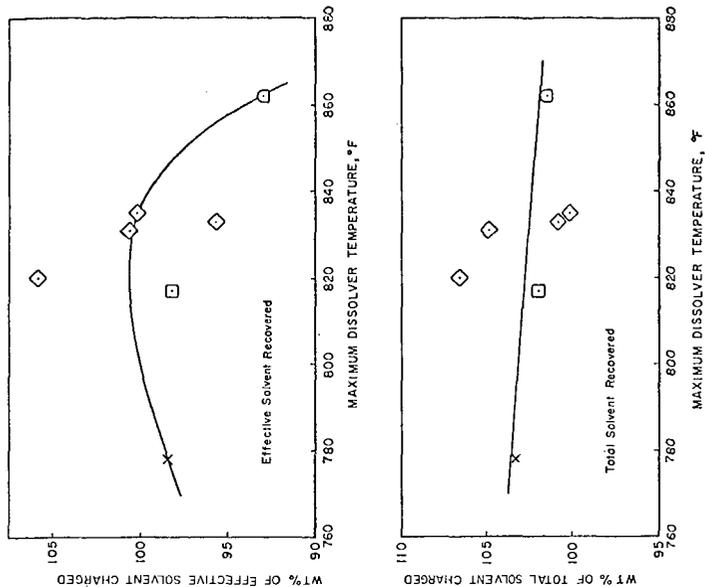


FIGURE 8

CONVERSION AND YIELDS vs. DISSOLVER PRESSURE
 (Adjusted to 820° F Maximum Dissolver Temperature,
 1.37 LHSV, and 1.91 Solvent to Coal Ratio)

3/1 H₂/CO Ratio

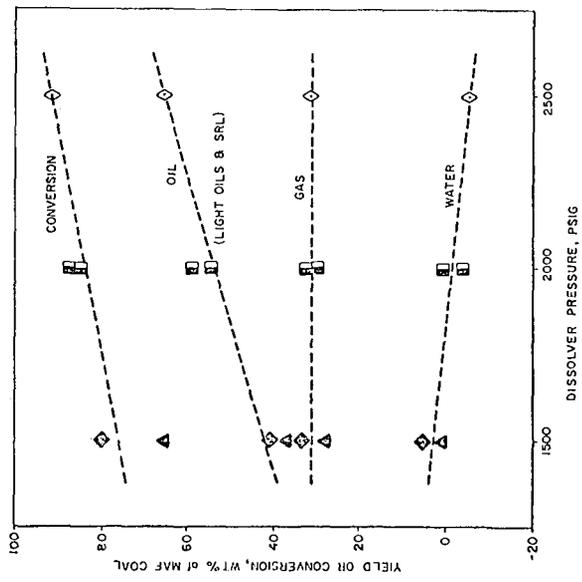


FIGURE 9

SOLVENT RECOVERY vs. DISSOLVER PRESSURE
 (Adjusted to 820° F Maximum Dissolver Temperature, 1.37 LHSV,
 and 1.91 Solvent/Coal Ratio)

3/1 H₂/CO Ratio

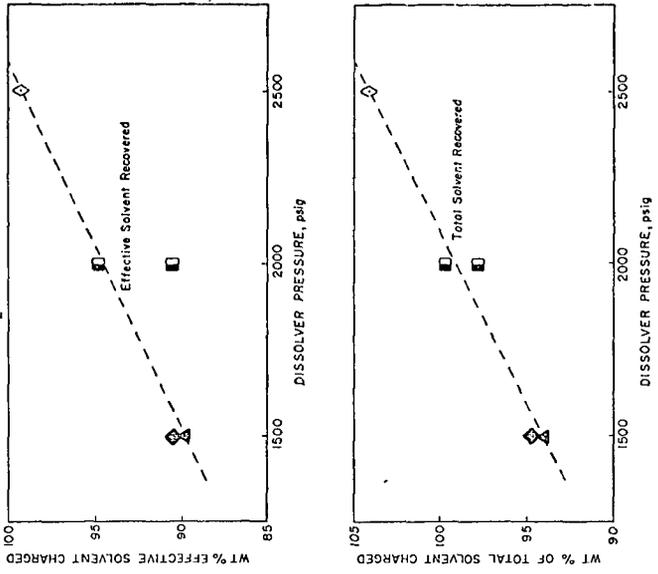


FIGURE 11
 SOLVENT RECOVERY vs MOL % H₂ IN CHARGE GAS
 (Adjusted to 835° F Maximum Dissolver Temperature,
 1.41 LHSV, and 1.91 Solvent/Coal Ratio)
 2500 psig Dissolver Pressure

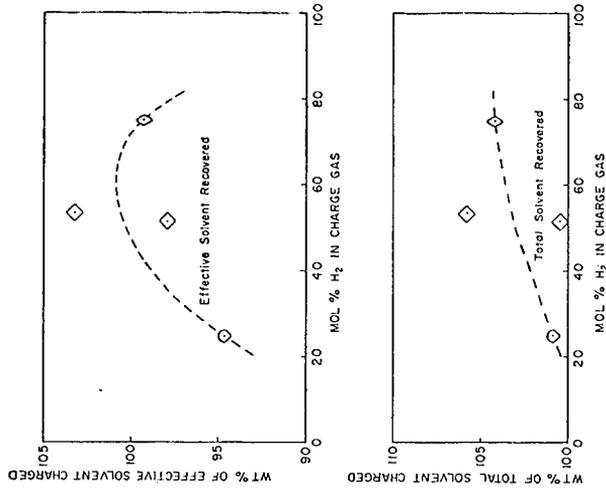


FIGURE 10
 CONVERSION AND YIELDS vs MOL % H₂ IN CHARGE GAS
 (Adjusted to 835° F Maximum Dissolver Temperature,
 1.41 LHSV, and 1.91 Solvent/Coal Ratio)
 2500 psig Dissolver Pressure

