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SOLVENT REFINED LIGNITE PROCESS DEVELOPMENT

Quarterly Technical Progress Report No. 3, January—March 1978

By
Donald E. Severson

July 12, 1978

Work Performed Under Contract No. EF-77-C-02-4189

Engineering Experiment Station
University of North Dakota
Grand Forks, North Dakota

U. S. DEPARTMENT OF ENERGY

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QUARTERLY TECHNICAL PROGRESS REPORT NO. 3
JANUARY, FEBRUARY, MARCH 1978

Donald E. Severson, Principal Investigator
Engineering Experiment Station
University of North Dakota

July 12, 1978

PREPARED FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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I. OBJECTIVES AND SCOPE

Project Lignite was established in 1972 in the Department of Chemical Engineering at the University of North Dakota for the purpose of determining the appropriate technological approach to the conversion of Northern Great Plains lignite to premium solid, liquid and gaseous fuels. The ultimate goal of the work was to make recommendations for a refinery based on lignite as feedstock. The experimental portion of the program was intended to develop data and know-how to carry out technological steps in the refinery processes for which information for scaleup was insufficient.

In keeping with this objective, laboratory and benchscale work were carried out and a continuous process development unit (PDU) was constructed and operated to demonstrate the feasibility of continuous liquefaction of lignite. Economic studies were made, and under a project subcontract, the Department of Chemistry of UND developed information on catalytic hydrogenation of solvent refined lignite.

The continuous process originally envisioned was the two stage conversion of lignite to fuel liquids, with solvent refined lignite as an intermediate and useful fuel product. The first stage is a unit converting 50 pounds per hour of raw lignite into solvent refined lignite by reaction with synthesis gas (carbon monoxide and hydrogen) in the presence of a hydrogen donor solvent that is continually regenerated from the lignite during processing. The mineral separations system is a countercurrent decantation process carried out at elevated temperature and pressure. The second stage was to be the catalytic hydrogenation to premium liquid fuels of the solvent refined lignite produced in the first stage. However, this latter step was not implemented, and the program has been directed at developing the liquefaction and solid-liquid separation stages.

Project Lignite was originally undertaken in accordance with Office of Coal Research Contract No. 14-32-0001-1224, later changed to ERDA Contract No. E(49-18)-1224, for the period March 28, 1972, through March 27, 1977. The period was later extended to June 15, 1977. In early 1977, a supplemental agreement was made under ERDA Contract No. EF-77-C-02-4189 to provide for the continuation of Project Lignite for the period June 1, 1977, through June 15, 1978.

The continuation basically provides for extended runs in the liquefaction section of the Process Development Unit, together

with some operation of the gas clean-up and recycle sections, the evaluation of the solid/liquid separation section, corrosion coupon testing, the determination of solids accumulation on the dissolvers, the production of clean solvent refined lignite, and data collection and analysis.

This report is the third and last of the required quarterly progress reports provided for in the supplementary agreement.

II. SUMMARY

Six runs, each nominally at one week's duration, M-34 through M-39, were completed; this concludes the PDU experimental program for Project Lignite. The first three runs concluded the series to study the effects of different lignites on plant operability and product yields. Runs M-37 and M-38 were made using two Montana subbituminous coals. It was concluded that coals with the highest ash content, both lignite and subbituminous coals, gave greater overall conversion to gaseous and liquid organic products. The subbituminous coals gave lower gas yields and lower overall conversion while producing similar liquid yields when compared with lignite. Reactor solids build up rapidly during initial operations, then level off and remain constant at 29 pounds per cubic foot of reactor volume as a run proceeds. Reactor solids evidently are composed of clay minerals and large particles of pyridine insoluble organic material when processing either lignites or subbituminous coals; in addition calcium/magnesium carbonate crystals (calcite/dolomite) are present in the case of lignite.

Long term, trouble free operation of the deashing unit was not accomplished. Integration of the unit for several hours of combined operation of the process was achieved, and the product from the deashing unit contained as low as 0.6 percent ash and pyridine insoluble material. Solids buildup in the solids settling tower was a continuing problem.

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III. TECHNICAL PROGRESS

A. PDU OPERATIONS

1. General

Six runs, M-34 through M-39, of one week's duration each, were completed during this quarter. This concluded the test program on the Project Lignite PDU, and three series of experiments were completed. Runs M-34, M-35, and M-36 were the last three runs in a series to determine differences in plant operability and in liquefaction yields for four different North Dakota lignites. Run M-37 and M-38 were run using two different Montana subbituminous coals, and differences in operability and in product yields for the two different ranks of coal were established. Run M-39 was made using Zap lignite and a pure hydrogen feed gas.

Plant operability was established in all of the runs including Run M-39 when pure hydrogen was fed to the unit. Mechanical problems persisted in every run, and long term integration of the liquid-solid separation (deashing) system was not achieved.

Process conditions together with complete work-ups showing overall conversions and product yields for each of the runs are given in Appendices A through F under individual run headings. The results of the runs are summarized in Table 1. Each run is briefly reviewed and results discussed in the following sections.

The laboratory facilities were completely dedicated to analytical support of PDU operations; no autoclave experiments were conducted.

2. Run M-34

Run M-34 was made using lignite from Gascoyne, North Dakota. Nominal operating conditions were the same as those used in Run M-33: 820-830°F reaction temperature; 2500 psig dissolver pressure; liquid and gas hourly space velocities of 1.43 and 333, respectively; equimolar mixture of carbon monoxide and hydrogen in the feed gas; and a solvent-to-MAF coal ratio of about 3. Reactor R-1B was used during the run and all recoverable organic distillates boiling above about 300°F at atmospheric pressure were recycled. Gaseous and liquid products from the reactor were cooled to 500-600°F and separated. The slurry was then depressured and vacuum concentrated. The vacuum bottoms were charged to the deashing unit, and diluted with toluene. The mineral residue and unconverted coal were removed following sedimentation.

Yield Period Coal From Symbols	M-34A ← Gascoyne → ◁ →	M-34D	M-35A ← → ◁ →	M-35C	M-36A ← Velva → ◁ →	M-36C	M-36D	M-38A Decker ◊ →	M-38D Rosebud	M-39A Zap ◊
Hours on Coal @ End of YP										
Since Reactor Cleaning	41.4	72.9	28.5	96.7	38.0	76.0	99.0	26.3	79.3	51.1
During Run	41.4	72.9	28.5	96.7	38.0	76.0	99.0	26.3	79.3	51.1
Cumulative	3456.5	3488.0	3516.5	3584.7	3622.7	3660.7	3683.7	3714.4	3767.4	3818.5
Reactor Used	R-1B	R-1B	R-1B	R-1B	R-1B	R-1B	R-1B	R-1B	R-1B	R-1A
Coal Charged, lbs/hr	45	44	46	48	46	50	46	42	42	46
Gas Charged, SCF/hr	482	467	506	515	483	469	476	432	438	570
Solvent/Coal Ratio	2.19	2.08	2.02	1.85	1.84	1.68	1.84	2.05	1.99	1.91
LHSV, Hr ⁻¹	1.47	1.40	1.44	1.39	1.35	1.36	1.34	1.30	1.31	1.39
GHSV, Hr ⁻¹	337	327	354	360	338	328	333	302	306	399
Coal Rate, lbs/hr/CF Reactor	31.2	30.9	32.1	33.7	32.1	34.9	32.3	29.2	29.4	32.3
Gas Rate, SCF/lb Coal	10.8	10.6	11.0	10.7	10.5	9.4	10.3	10.3	10.4	12.3
Gas Consumed, SCF/lb Coal	2.4	2.7	1.5	3.2	2.2	2.0	1.9	1.8	2.2	2.3
Mole Percent H ₂ in Gas Consumed	24.9	25.7	31.0	39.4	35.0	30.4	23.8	7.7	31.0	102.7
Yields, Wt % MAF Coal										
Net Gas	36.2	34.1	31.7	29.0	27.7	28.5	32.2	20.9	16.0	16.8
Net Oil	63.1	69.5	55.0	64.7	66.1	62.8	57.2	61.8	70.6	54.2
(Dist Oil)	(10.6)	(15.3)	(1.3)	(13.8)	(26.3)	(27.2)	(19.4)	(13.6)	(19.8)	(8.4)
(SRL)	(52.5)	(54.2)	(53.7)	(50.9)	(39.8)	(35.6)	(37.8)	(48.2)	(50.8)	(45.8)
Net H ₂ O & Ash	-8.0	-10.3	0.6	-5.2	-3.4	-4.4	-1.1	-2.8	-3.6	11.1
Unconverted MAF Coal	8.7	6.7	12.7	11.5	9.6	13.1	11.7	20.1	17.0	17.9
Solvent Recycle, %										
Actual	92.6	100.7	94.4	95.8	93.7	97.8	97.4	97.7	97.8	95.6
Potential (Calc)	102.6	103.9	100.4	104.4	108.3	109.4	106.1	105.1	106.7	102.8
Potential (Ex Lt Oil)	103.0	102.6	106.4	96.2	93.8	104.7	98.5	108.0	103.0	98.9
Material Balance, %	100.3	101.8	99.5	100.9	97.8	99.9	99.1	99.8	101.4	98.5
Conditions										
Temperatures, °F										
Slurry Preheater Outlet	735	739	760	730	743	736	750	750	742	766
Dissolver Inlet	718	700	629	614	630	678	646	726	718	741
R-1A 3 ft	NIU	NIU	NIU	NIU	NIU	NIU	NIU	NIU	NIU	774
R-1A 15 ft	NIU	NIU	NIU	NIU	NIU	NIU	NIU	NIU	NIU	804
R-1B 3 ft	762	747	755	744	722	719	753	738	737	NIU
R-1B 15 ft	824	807	820	823	816	820	829	813	806	NIU
Dissolver Outlet	801	806	794	804	771	782	792	777	718	711
Flash Preheat	610	620	630	633	594	639	644	614	624	637
Flash Vapor	525	532	519	519	512	530	530	497	504	507
Pressures										
Dissolver, Psig	2450	2450	2500	2500	2500	2500	2500	2500	2500	2500
Inter Sep, Psig	240	253	234	240	243	227	237	241	200	200
Vacuum Flash, Torr	15	14	15	14	27	18	19	20	20	17
SLS System in Use?	Yes	No	Yes	No	Yes	No	No	Yes	No	Yes
Mole % H ₂ in Feed Gas	52.1	53.5	52.7	52.5	51.0	50.0	50.8	51.9	52.0	99.6
Mole % CO in Feed Gas	47.5	46.1	47.0	47.2	48.6	49.4	48.8	47.6	47.6	0.0
CO ₂ Yield, Wt % MAF Coal	53.9	55.3	38.6	45.5	40.6	40.4	44.1	31.7	28.8	10.5
C ₁ -C ₃ Yield, Wt % MAF Coal	5.9	5.8	5.5	7.7	5.8	6.2	6.6	4.6	3.4	7.2
Gas Consumed (H ₂ Equiv), Wt % MAF Coal	2.34	2.68	1.31	2.85	2.04	1.55	1.75	1.20	1.73	1.85

A number of operational difficulties occurred during the run with temperature control of the reactor being the first. This resulted from faulty thermocouple splices and cross connections of heater and power supplies during rewiring of the reactor. The remaining problems were of a more routine nature, but they required considerable attention during the run and interrupted two yield periods. The deashing unit was operated early in the run and of the two yield periods completed, one was made after the deashing unit was shut down.

Material balance closures were 100.3 percent and 101.8 percent for yield periods M-34A and M-34D, respectively. Adjustment of the material balance to 100 percent for each of the yield periods is discussed in the detailed write-up of the run (see Appendix A). Net gas yields averaged 35.2 weight percent while the net organic liquid yield was 66.3 percent of the MAF lignite fed. These values vary only slightly from those of Run M-33. Overall conversion was 91 percent compared with 92 percent of the MAF lignite fed in Run M-33. Averaging the results obtained for the six yield periods in both Runs M-33 and M-34 indicate that the net gas and net organic liquid yields when processing Gascoyne lignite are 36 and 64 weight percent of the MAF lignite charged, respectively, while the average overall conversion for the two runs was 91.5 percent.

Comparing the ash and pyridine insolubles in the F-1 vacuum bottoms for yield period M-34D in which the deashing unit was not used with the sum of the pyridine insolubles and the ash in the SRL after deashing in Run M-34A showed that the product deashing was successful. In the former instance the insoluble material plus ash totaled nearly 27 percent of the vacuum bottoms while deashing reduced those materials to 0.6 weight percent of the final product. Only about 3 percent of the SRL charged was lost to the residue during the deashing process.

Following Run M-34 approximately 85 pounds of oily solids and 23 pounds of solvent were removed from the reactor by blow-down. Samples of the solid material were analyzed for pyridine insolubles both before and after extraction with toluene. Additionally, sieve analysis showed 29 percent of the reactor solids larger than 21 microns and nearly 72 percent larger than 15 microns. This reflects an increase in particle size compared with the feed material and may indicate growth of particles of minerals such as calcium carbonate. Dried reactor solids were analyzed by X-Ray diffraction and by X-Ray fluorescence. The former analysis showed calcite to be the most abundant mineral with quartz, anhydrite and feldspar showing decreasing abundance.

3. Run M-35

Run M-35 was another in the series of runs to evaluate the effects of lignite variation on PDU operability and liquefaction yields. Lignite mined by the Consolidation Coal Company near Velva, North Dakota was used in this run. Velva lignite is similar in chemical makeup to Zap lignite even though slight variations exist in the chemical composition of the two ashes.

Nominal operating conditions were similar to those of previous runs: 820-830°F maximum reaction temperature, 2500 psig dissolver operating pressure, solvent-to-coal ratio of 1.8, and gas and slurry hourly space velocities of 1.43 and 330, respectively. One reactor was used, and all recoverable organic liquids boiling above 300°F at atmospheric pressure were recycled. The product deashing system operated during part of the run.

Liquefaction operations during Run M-35 were smooth and largely trouble-free; however, difficulties in charging and transferring slurry in the deashing section prevented extended integration of the deashing operation during the run. About 64 hours of deashing operation with only one 12-hour interruption were achieved during the run. Of the three yield periods started during Run M-35, two were completed; M-35A with the deashing section operating and M-35C with no deashing. Data period M-35B was terminated because of difficulties in the liquid-solid separation system.

Material balance closures were 99.5 and 100.9 percent for the two completed yield periods. Adjustment to 100 percent recovery for data period M-35A, made during product deashing, was made to account for the approximately 305 pounds of solids that had accumulated in the solids precipitation tower during 64 hours of operation.

Net gas and organic liquid yields averaged 30.4 and 59.8 weight percent of the MAF lignite charged, respectively. Distillable oils averaged nearly 13 percent of the total net organic liquid yield. Overall conversion of MAF lignite to gas, oil and SRL averaged about 88 percent. Actual solvent recovery for recycle averaged 95 percent while the calculated potential solvent recovery was 102 percent. Deashing, during yield period M-35A, was successful; the pyridine insoluble organic material plus ash totaled only about 0.8 weight percent of the final product.

Following Run M-35, about 66 pounds of oily solids and 27 pounds of solvent were recovered from the liquefaction reactor. The wet and toluene-washed solids were analyzed for pyridine solubles. Dried, toluene-washed solids were analyzed further by

sieving and X-Ray diffraction, and the ash of these solids were also analyzed. Results of these tests are shown in the detailed write-up of Run M-35 (Appendix B).

4. Run M-36

Run M-36, the last of six runs made using three different North Dakota lignites, was a replicate of Run M-35 using Velva lignite. Nominal run conditions were identical with those of the previous five runs: 820-830°F reactor temperature, 2500 psig reactor pressure, solvent-to-coal ratio of 1.8, and liquid and gas hourly space velocities of 1.43 and 330, respectively. The feed gas was an equimolar mixture of carbon monoxide and hydrogen. One reactor was used, and all recoverable organics distilling above 300°F at one atmosphere were recycled. The product deashing unit operated at 450°F and 300 psig.

Operating problems were limited to the vacuum flash recovery equipment and to the mineral residue settler. The vacuum flash tower operated at 25 to 30 Torr during most of the run because of a leak in the system that could not be located and repaired. In the mineral residue settler, plugging in the line upstream of the overflow discharge line apparently resulted from solids carried in the overflow liquid stream. A large part of the liquid flashes when depressured across the control valve, leaving solids to accumulate in the throat and upstream of the valve.

Four yield periods were started during Run M-36, three were completed. Data period M-36B was terminated before completion when plugging occurred in the liquid-solid separation system. The last two yield periods were made without deashing.

Average material balance closure for the three yield periods was nearly 99 percent. The net gas and the net liquid yields were 29.5 and 62 percent of the MAF lignite fed, respectively. Distillable oil yield averaged 24 percent of the MAF lignite for the run, and the overall conversion to gaseous and liquid products was 88.5 percent.

A striking difference between the SRL yields in Run M-35 compared with that of Run M-36 is evident. The variation is perhaps attributable to differences in the vacuum distillation conditions or to variance in the distillation test rather than to differences in the run conditions.

At the end of Run M-36, the reactor was blown down resulting in recovery of about 76 pounds of oily solids and 30 pounds of liquids. The solids were analyzed both before and after toluene extraction. The results were similar to those obtained in Run M-35, and are discussed in Appendix C.

Results obtained in the coal variety study indicate operating problems are much the same regardless of the type of lignite processed. In addition, liquefaction yields obtained with the four lignites, though different, do not vary sufficiently to warrant redesign of the plant. Establishing different "optimum" operating conditions depending on the lignite fed may be worthwhile, however.

5. Run M-37

Run M-37 was the first run in a short study involving processing subbituminous coals in the PDU. The first coal used was from Decker, Montana, and the nominal operating conditions were those determined to be optimum when processing Zap lignite in the presence of a hydrogen-carbon monoxide reducing gas atmosphere: 820-830°F maximum reactor temperature, 2500 psig reactor pressure, solvent-to-coal ratio of 1.8, and feed gas mixture of hydrogen and carbon monoxide containing equimolar quantities of the two gases. The gas was fed at a rate of about 8 SCFM, while the slurry feed rate was 126 pounds per hour, compared with 140 hours per hour when feeding lignite. The lower slurry feed rate results in the same MAF coal feed rate (32 pounds per hour) because of the lower moisture content of the subbituminous coal. The deashing system was operating during the run with the settling tower conditions being 450°F and 300 psig.

Run M-37 suffered through a number of operating problems including minor mechanical problems during startup, and no yield periods were completed. Following resolution of startup problems, the unit responded well when coal was first introduced. However, a malfunctioning pressure control valve caused a buildup of pressure in the liquefaction section, and the rupture disc on the high pressure condensate accumulator burst. The rapid depressuring resulted in flashing of the solvent leaving the reactor partially plugged. Efforts to resume operations following replacement of the rupture disc were unsuccessful because the deposits could not be removed from the reactor. Consequently, the run was terminated.

During Run M-37, about 199 pounds of coal were charged, and it was observed that the temperature rise through the reactor was greater than that usually observed when processing lignite. Analysis of the reactor solids removed following termination of the run indicated that quartz followed by calcite were the predominant mineral species present in the toluene-washed material.

6. Run M-38

Run M-38 was made to continue the series using subbituminous coals as feed to the PDU. Nominal run conditions were the same as those of Run M-37; 820-830°F maximum reaction temperature, 2500 psig pressure, solvent-to-raw coal ratio of 1.8, and the slurry feed rate was about 126 pounds per hour. The MAF coal feed rate was approximately 32 pounds per hour, one reactor volume was

used, and all recoverable organic distillates boiling above about 300°F were recycled. An equimolar mixture of hydrogen and carbon monoxide was fed to the reaction. The deashing unit operated during the first half of the run at a temperature and pressure of 450°F and 300 psig, respectively.

An inadvertant mix-up in the feed coal resulted in initially feeding Decker subbituminous coal followed by alternately feeding coal from the Rosebud, Montana mine and Decker coal in the middle of the run. Four yield periods were completed during Run M-38; the first was made with Decker subbituminous coal, the fourth was made with Rosebud coal, and the middle two yield periods were made when alternate drums of Decker and Rosebud coals were being charged. Thus, only yield periods M-38A and M-38D were worked up and reported, and the results were used to compare the effects of the two subbituminous coals.

Material balance closure was 99.8 percent and 101.4 percent for yield periods M-38A and M-38D, respectively. Yield data and selected analyses of the coals are summarized below:

Yield Period	M-38A	M-38D
Coal Charged	Decker	Rosebud
Proximate Analysis, Wt%		
Moisture	21.7	25.7
Ash	3.9	11.2
Volatile Matter	32.4	26.6
Fixed Carbon	42.0	36.5
Product Yields, Wt% MAF Coal		
Net Gas	20.9	16.0
Net Liquid	61.8	70.6
(Distilled Oil)	(13.6)	(19.8)
(SRC)	(48.2)	(50.8)
Overall Conversion	79.9	83.0

It appears that the higher ash content of the Rosebud coal catalyzed or otherwise affected the liquefaction reaction resulting in lower net gas yields and higher net liquid yields. The SRC yield, however, remained nearly the same for the two coals. Rosebud coal produced a higher yield of distillable oil than did Decker, the lower ash containing coal. Overall conversion for both coals ranged from 80 to 83 percent of the MAF coal charged.

The reactor was blowdown at the end of Run M-38. Analysis of the recovered solids showed quartz, followed by calcite, to be the predominant minerals remaining in the reactor. This is consistent with the results obtained following Run M-37 when Decker coal was processed throughout the run, and it is just the reverse of what was found when processing lignite. This may be because the higher oxygen content in lignite promotes the formation of calcium carbonate. It was also found that lignite tends to form greater quantities of reactor deposits than do subbituminous coals.

7. Run M-39

Run M-39 was the last run in the 1977-78 PDU test program. It was scheduled for 96 hours instead of the normal 120 hours because of a Good Friday holiday.

The run was made using Zap lignite at operating conditions found to be optimum with this lignite. In this run, however, pure hydrogen rather than the usual equimolar mixture of carbon monoxide and hydrogen was fed to the system. Other nominal run conditions were: 820-830°F maximum dissolver temperature, 2500 psig pressure, solvent-to-coal ratio of 1.8, 140 pounds per hour total slurry feed rate, and gas feed rate of 8 SCF per minute. The deashing section was operated at the usual conditions: the solids settling tower at 450°F and 300 psig and the toluene recovery tower at 500°F reboiler temperature and 2-5 psig pressure.

Only one yield period was completed because of various leaks and mechanical problems. The worst leak occurred at the bottom closure of the reactor, and it was necessary to switch to the standby reactor. The material balance closure for the completed yield period, M-39A, was 98.5 percent. Results of Run M-39 showed the overall conversion of MAF lignite to gas, distillable oil, and SRL was about 82 percent while the net gas and organic liquid yields were 17 and 54 percent, respectively. Hydrogen consumption was about 1.91 weight percent of the MAF lignite fed.

Results of deashing were not as good as in the past few runs; the total of ash and pyridine insolubles in the final product was reduced to 1.3 weight percent.

When it was necessary to shut down reactor R-1B because of the bottom closure leak about 75 pounds of reactor solids were removed by reactor blowdown. This followed 32 hours of processing coal. After 19 hours of processing coal in reactor R-1A, approximately 81 pounds of material, of which 19 pounds were oily solids, were recovered.

8. Discussion of Results

Run M-36 completed the series of runs made to evaluate the effects of lignite variety on PDU operations and liquefaction yields. Detailed figures and tables are presented in the discussion of Run M-36 (Appendix C). Results typical for each of the four lignites, Zap, Gascoyne, Larson, and Velva were averaged and several general observations are evident. A summary of the information is presented on the next page.

Lignite type	Larson	Zap	Gascoyne	Velva
Solvent Recycle, %				
Actual	85.7	92.6	93.7	95.8
Potential	101.4	104.0	103.2	105.7
Potential (540°F +)	96.6	96.0	101.1	99.9
Yields, Wt% MAF Coal				
Net Gas	33.1	31.0	35.6	29.9
Net Liquid	54.7	63.4	64.4	61.1
(Dist Oil)	(4.1)	(13.0)	(15.1)	(17.6)
(SRL)	(50.6)	(50.4)	(49.3)	(43.5)
Overall Conversion	80.2	89.9	91.3	88.3

The highest conversion and net liquid and gas yields were obtained using Gascoyne lignite. Larson lignite was the least reactive with both low overall conversion and low net liquid yield. The fraction of net liquids in the distillable oil range was lowest for Larson lignite and highest for Velva lignite.

Liquefaction results for the four lignites can be correlated with their ash contents, and consequently with the content of minerals which may have a catalytic effect. Gascoyne lignite, the highest in ash content, produced the highest overall conversion to gaseous and liquid products. Larson lignite had the lowest ash content and the lowest overall conversion. Evidently conversion increases with increased ash content up to about 6.3 percent ash at which point conversion remains constant at about 91.5 percent.

A summary of the results obtained when processing North Dakota lignite indicates that overall conversion ranges from 80 to 91 percent of the MAF coal while the net gas yield ranged from 30 to 36 percent and the net liquid yields were 54 to 65 percent. It is also evident that the potential for generating adequate process derived recycle solvent is good, and that varying process conditions may be necessary as different lignites are fed, but redesigning the process is not required.

Run 38 completed the run series using subbituminous coals in the PDU. A summary of the results comparing subbituminous coal with North Dakota lignite is on the following page.

Coal Type	Montana Bituminous Decker	Rosebud	ND Lignite Average
Solvent Recycle, %			
Actual	97.7	97.8	92.0
Potential	105.1	106.7	103.6
Yields, Wt% MAF Coal			
Net Gas	20.9	16.0	32.4
Net Liquid	61.8	70.6	60.9
(Dist Oil)	(13.6)	(19.8)	(12.4)
(SRC)	(48.2)	(50.8)	(48.5)
Total Conversion, %	79.9	83.0	87.4

Some variations in operating conditions exist for the various runs, but lower net gas yields are evident when processing subbituminous coal than those obtained when processing lignite. The net liquid yields for the two types of coal were similar, in the range of 60-70 weight percent of the MAF coal fed. Overall conversion of MAF coal was generally lower (about 82 percent) for the subbituminous coals than for the average for the North Dakota lignites (about 87 percent). The lower conversion may be due, in part, to the slightly lower reaction temperature when processing the subbituminous coals (810°F as opposed to 825°F for lignites).

It is interesting to note that coals containins higher ash contents, Rosebud subbituminous coal and Gascoyne lignite, appeared to yield higher overall conversions when compared with other coals of the same rank. Additionally, reducing gas consumption, on a hydrogen equivalent basis, averaged 2.1 weight percent of the MAF coal for lignite compared to approximately 1.5 weight percent for the Montana subbituminous coals studied. This difference in gas consumption may be the result of the water gas shift reaction being more important with the higher moisture content lignite. This is also indicated by comparing the water consumption of the two types of coals; water consumption averaged 4.9 weight percent of the MAF coal fed for lignite compared with 2.1 weight percent for subbituminous coal.

Data obtained on reactor solids buildup for the two ranks of coal indicates that lignites have a greater tendency to form reactor deposits either by particle agglomeration or by mineral growth than do the subbituminous coals. This may be because of the identity of the minerals present in the coal or because of the difference in ash concentration in the two ranks. Lignites generally contain a higher ash content and there is a predominance of calcite in the reactor solids while the predominant mineral in reactor deposits when processing subbituminous coals appears to be quartz.

Run 39 completed a series of runs made to determine the effects of feed gas composition on operability and product yields. A summary follows showing results obtained when using Zap lignite and similar operating conditions but with varying fractions of hydrogen in the feed gas.

Varying Feed Gas Composition			
Mole % H ₂ in Feed Gas	52.5	62.9	99.6
Yield Period(s)	L-1K	L-2D	M-39A
Solvent Recycle, %			
Actual	86.4	93.1	95.6
Potential	107.6	100.0	102.8
Yields, Wt% MAF Lignite			
Net Gas	26.9	23.6	16.8
Net Liquid	65.8	58.6	54.2
(Dist Oil)	(22.2)	(-0.1)	(8.4)
(SRL)	(43.6)	(58.7)	(45.8)
Overall Conversion, %	89.2	82.9	82.1

It appears that as the reducing gas atmosphere is enriched in hydrogen, the overall conversion of MAF lignite to gas, oil and SRL decreases. Net gas and organic liquid yields also decrease with increasing fractions of hydrogen.

In addition to concluding the PDU test program, Run M-39 also completed the study of reactor solids buildup. A complete summary of the data is given in Table 3 - Appendix F. No conclusive results can be drawn; however, the following observations can be made:

- 1) Reactor solids apparently result from the settling of clay minerals such as feldspar and quartz along with large particles of pyridine insoluble organic materials, and/or formation and settling of calcium/magnesium carbonate crystals - calcite and dolomite - when processing lignite;
- 2) the level of total solids remaining in the reactor following process shutdown increases rapidly, then levels off, and remains constant at about 29 or 30 pounds per cubic foot of reactor volume.

With regard to (1) above, it appears that little calcium carbonate is formed when processing Montana subbituminous coal and that most reactor solids are deposits of clay minerals and unconverted coal. When processing North Dakota lignite, calcium carbonate, as well as the clay minerals and unconverted coal, is present in the reactor deposits.

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IV. CONCLUSIONS

A. PDU OPERATIONS

1. Six runs, M-34 through M-39, were completed. Yield periods were completed, for all runs except Run M-37, with good material balance closures.
2. Run M-39 completed the PDU test program on Project Lignite.
3. Gascoyne lignite, of the four North Dakota lignites processed, gave the highest overall conversion to gaseous and liquid products, while Larson lignite resulted in the lowest conversions. Gascoyne and Larson lignites also had the highest and lowest ash contents, respectively, indicating that ash content and any resultant catalytic activity may affect overall conversion and product distribution.
4. Lower net gas yields were evident when processing two Montana subbituminous coals compared with that obtained for lignites. Net liquid yields for the two types of coal are comparable; therefore, the overall conversion was somewhat lower for the subbituminous coals.
5. As the reducing gas atmosphere is enriched in hydrogen the overall conversion and the net gas and net organic liquid yields decrease when processing Zap lignite.
6. Reactor solids evidently result from agglomeration and settling of clay minerals and large particles of pyridine insoluble organic materials, and/or the formation and settling of calcium/magnesium carbonate crystals. The constituents of the solids present in the reactor depend upon the identity of the coal processed.
7. Reactor solids build up rapidly then level off and remain constant at 29-30 pounds per cubic foot of reactor volume.
8. Long term integration of the deashing section into the overall process was not achieved, but the contents of insoluble organic material and ash in the final product was satisfactorily low when it was operating.

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APPENDIX A

Process Development Unit Run M-34. January 8-13, 1978

Run M-34 was a replicate of Run M-33, using lignite from Gascoyne, North Dakota. Operating conditions were 820-830°F nominal reaction temperature and 2500 psig dissolver pressure with liquid and gas hourly space velocities of 1.43 and 333 respectively. Reactor R-1B was on stream throughout the run and all recoverable organic distillates boiling above about 300°F were recycled. On leaving the liquefaction reactor, the gaseous and liquid products were cooled to 500 or 600°F and separated. The slurry was then depressured and vacuum concentrated. The vacuum bottoms were withdrawn from the vacuum system and charged to the deashing unit where they were diluted with toluene and where the mineral residue and unconverted coal were removed after sedimentation.

A number of problems were encountered during Run M-34, with temperature control difficulties predominant during the first 48 hours of operation. These difficulties were the result of rewiring Reactor R-1B, which was completed just prior to the start of the run. Several faulty thermocouple splices and some cross connections of heaters and power supplies resulted in temperature control problems with both the reactor wall and adiabatic heaters.

Later in the run, and after the electrical problems with reactor R-1B had been rectified, several other problems were encountered. The lower vacuum lockout valve under the vacuum flash tower and the solids removal valve below the solids precipitation tower started leaking and finally required the installation of new Rulon seats to obtain satisfactory sealing and operation. The run then progressed smoothly until about four hours into yield period M-34B when the shear pin protecting the F-0 vacuum bottoms pump sheared. The pin was replaced and product deashing continued for another hour before the shear pin again failed. This ended efforts to deash the product, and the deashing unit was isolated, cooled, and shutdown.

Data period M-34C was started utilizing only the liquefaction unit. However, it was terminated early when the timer actuated cams controlling the sequencing of the F-1 vacuum lockout valves malfunctioned, causing a significant vacuum loss during each dumping cycle. This problem took some time to diagnose and correct.

During Run M-34, four data periods were started but, because of the various problems mentioned, only two were completed, one with the deashing unit on stream and one without. The two completed yield periods, M-34A and M-34D, were worked up in entirety including elemental balances and oil-phase ASTM D86 and D1160 distillations. The completed workups including run conditions are attached.

Material balance closure was 100.3 percent in data period M-34A and 101.8 percent in M-34D. Yield period M-34A was adjusted to 100 percent recovery by slightly decreasing the V-8 bottoms rate. The V-8 bottoms composition was then calculated from the compositions and flow rates of the other streams entering and leaving the product deashing sections. During Run M-34, 184 pounds of solids accumulated in the settling tower while it was in service. As a result, the V-8 bottoms stream is relatively higher in solvent and toluene than it actually would have been if the solids had been removed proportionately from the settling tower.

Yield period M-34D was adjusted by normalizing the outgoing product streams for 100 percent recovery and then a forced carbon balance was made by adjusting the oil-water ratio of the S-8 bottoms stream. A comparison of the two yield periods after adjustment is shown on the following page.

Net gas yields during Run M-34 averaged 35.2 weight percent of the MAF lignite fed. This compares favorably with the 36.8 weight percent average obtained from the yield periods in Run M-33 which were also made with Gascoyne lignite.

The average net organic liquid yield during Run M-34 was 66.3 weight percent of the MAF coal charged. This is slightly higher than the average 62.5 weight percent reported in Run M-33 but may be the result of normal variance in the yields and in the pyridine extraction tests on the dissolver slurry products or the F-1 vacuum bottoms. The pyridine extractables in Run M-33 indicate MAF lignite conversions of about 91.1 percent while in Run M-34 where liquefaction conditions were almost identical to those of Run M-33, the conversion was 92.3 percent.

A comparison of the average results of Runs M-33 and M-34 is shown in Table 2. Also included is an average representing the results of the six yield periods involved. This six yield period average is perhaps more indicative of the actual yields to be obtained when processing Gascoyne lignite than any of the individual yield periods. This average then indicates that the net gas and organic liquid yields obtained when processing Gascoyne lignite would be 36 and 64 weight percent of the MAF lignite charged, respectively. Net distillable oil yields would average 8 weight percent of the MAF coal while SRL yields would run about 56 weight percent. Conversion of MAF lignite to gas, oil and SRL would average 91.5 percent, and the calculated potential solvent recovery on a total oil basis would be 102.1 percent.

The results of product deashing during yield period M-34A were quite favorable with the sum of the pyridine insoluble material and mineral residue (ash) reduced to 0.6 weight percent of the finished product. A substantial reduction when compared

Table 1
Process Development Unit Run M-34

Yield Period	M-34A	M-34D
No. of Reactors	1	1
LHSV	1.47	1.40
GHSV	337	327
Solvent/MAF Coal Ratio	4.06	3.93
H ₂ /CO Mol Ratio	1.10	1.16
Temperatures, °F		
Preheater Outlet, E-1	735	739
Reactor 3 ft	762	747
Reactor 7 ft	785	779
Reactor 11 ft	796	781
Reactor 15 ft	824	807
Max. Reaction	824	807
Vac. Flash Ovhd	525	532
Dissolver Pressure, psig	2450	2450
Solvent Recycle, %		
Actual	92.6	100.7
Potential	102.8	103.9
Yields, Wt % MAF Coal		
Net Gas	36.2	34.1
Net Liquid	63.1	69.5
(Dist Oil)	(10.6)	(15.3)
(SRL)	(52.5)	(54.2)
Net H ₂ O and Ash	-8.0	-10.3
Unconverted Coal	8.7	6.7
Overall Material Balance, %	100.3	101.8

Table 2
Process Development Unit Runs M-33 & M-34

Run	M-33	M-34	Average
No. of Yield Periods Averaged	4	2	6
No. of Reactors	1	1	1
LHSV	1.41	1.44	1.42
GHSV	299	332	310
Solvent/MAF Coal Ratio	3.85	4.00	3.90
H ₂ /CO Mol Ratio	1.0	1.1	1.0
Temperatures, °F			
Preheater Outlet, E-1	751	737	746
Reactor 3 ft	766	754	762
Reactor 7 ft	799	782	793
Reactor 11 ft	799	788	795
Reactor 15 ft	822	816	820
Max. Reaction	822	816	820
Vac. Flash Ovhd	516	528	520
Dissolver Pressure, psig	2500	2450	2500
Solvent Recovery, %			
Actual	92.2	96.6	93.7
Potential	101.3	103.6	102.1
Yields, Wt % MAF Coal			
Net Gas	36.8	35.2	36.2
Net Liquid	62.5	66.3	63.8
(Dist. Oil)	(5.1)	(12.9)	(7.7)
(SRL)	(57.4)	(53.4)	(56.1)
Net H ₂ O and Ash	-8.2	-9.2	-8.5
Unconverted Coal	8.9	7.7	8.5
Overall Material Balance, %	98.0	101.0	99.0

with yield period M-34D which was made without deashing and for which the sum of these materials totaled 26.7 weight percent of the F-1 vacuum bottoms.

Loss of SRL during the product deashing step was approximately 3 percent of that charged. This material in theory should have left the solids settling tower with the mineral residue and pyridine insoluble material, though in practice a large part of it accumulated in the tower.

At the end of Run M-34 the reactor solids blowdown system was utilized for the first time. The procedure employed was first to cool the reactor by charging either raw or once distilled solvent alone while maintaining 2500 psig H_2 pressure. When the temperature was reduced to 400°F, gas and solvent flows were stopped and the inlet and outlet valves to the reactor closed. Slurry preheat coil E-1 was then depressured and flushed with solvent until sand bath temperatures were below 300°F. With the reactor isolated at 400°F and 2500 psig pressure, the reactor solids accumulator was connected to the reactor blowdown valve located near the reactor inlet. The blowdown valve, which had been fitted with tungsten carbide trim for this service, was then opened and the pressure allowed to equalize. In this manner, the reactor solids were transported to the collection vessel and then later removed for analysis. The sketch on the following page illustrates the apparatus and piping involved. To check the effectiveness of the blowdown method, the cooled reactor was disassembled and inspected. Only a small amount of wall scale, too small to recover or measure, remained.

Approximately 85 pounds of oily solids and 23 pounds of solvent were removed from the reactor by blowdown at the end of Run M-34. Prior to blowdown, the reactor had been flushed with solvent for about 5-1/2 hours. The recovered solids and liquids were separated by decantation and then the solids were coned and quartered twice to yield about a 5 pound sample. On a portion of this sample, the ash and pyridine extractables were determined:

Run M-34 Raw Reactor Solids

	Wt %
Pyridine Solubles	36.8
Pyridine Insolubles	23.4
Ash	39.8
Total	100.0

In addition another portion of this 5 pound sample was mixed with an equal volume of toluene, heated to near the boiling point of toluene with stirring and then allowed to stand until the solids settled, after which the hot liquor was decanted. The remaining solids were dried at 105°C overnight and then pyridine solubles and ash content were determined on the dried material:

PROJECT LIGNITE REACTOR BLOWDOWN AND SOLIDS COLLECTION SYSTEM

Run M-34 Toluene Washed Reactor Solids

	Wt %
Pyridine Solubles	3.3
Pyridine Insolubles	45.8
Ash	50.9
Total	100.0

Sieve analysis of the dried solids indicated that approximately 29 percent of the material was larger than about 21 microns in diameter and 71.5 percent was larger than 15 microns. The sieve analyses for the dried, toluene washed, reactor solids and for the M-34 feed coal are shown below:

U.S. Screen Sieve Mesh	Total Percentages			
	On		Passing	
	Feed Coal	Reactor Solids	Feed Coal	Reactor Solids
65	1.6	29.3	98.4	70.7
100	10.8	71.5	89.2	28.5
140	29.6	89.3	70.4	10.7
170	52.2	95.2	47.8	4.8
230	71.8	99.3	28.2	0.7
325	88.4	99.8	11.6	0.2
Pan	100.0	100.0	0.0	0.0

An increase in particle size occurs in the reactor during liquefaction, perhaps because of growth of minerals such as calcuim carbonate.

An aliquot of the dried reactor solids was analyzed at the Grand Forks Energy Research Center by X-Ray diffraction. Identifiabile minerals present were:

Calcite - most abundant, with some magnesium carbonate
Quartz - About 1/3 as abundant of calcite
Anhydrite - 1/6 as abundant as calcite
Feldspar - small amount-1/20 the amount of calcite

In addition, an ash sample of the toluene washed reactor solids was analyzed by X-Ray fluorescence. The results, normalized to 100 percent, are shown on the following page along with a similar analysis for the feed coal ash and the M-34D F-1 bottoms ash. The analyses for the feed coal and the F-1 bottoms ash were obtained by atomic absorption and colorametric methods.

Process Development Unit Run M-34
Ash Analysis

Wt %	Ash		
	Feed Coal	F-1 Btms (34D)	Toluene-washed Reactor Solids
Loss on Ignition @ 800°C	0.3	2.1	-
SiO ₂	18.5	26.3	18.7
Al ₂ O ₃	13.4	18.0	10.9
Fe ₂ O ₃	6.5	10.6	6.0
TiO ₂	0.7	0.8	0.3
P ₂ O ₅	0.1	0.1	0.6
CaO	35.9	23.4	46.0
MgO	11.5	9.7	8.4
Na ₂ O	3.9	2.9	1.7
K ₂ O	0.7	1.4	0.4
SO ₃	8.5	4.7	7.0
Total	100.0	100.0	100.0

RUN CONDITIONS

RUN NO. M-34A
DATE MADE 1/11-12/78

TEMPERATURE °F

PREHEATER E-I
SAND BATH 753
INLET 246
OUTLET 735

DISSOLVERS

R-1A (1) _____
(2) _____
(3) _____
(4) _____
R-1B (1) 762
(2) 785
(3) 796
(4) 824

REACTOR PRODUCT SEPARATORS

S-1 537
S-2 45
S-3 476
S-4A 66
S-4B 71

PREHEATER E-II

DOWTHERM 649
INLET 357
OUTLET 610

VACUUM FLASH F-I

OVERHEAD VAPOR 525
UPPER WALL 632
LOWER WALL 598
DOWNCOMER 385
CONDENSATE
ACCUMULATOR, S-7 75

PREHEATER E-13

DOWTHERM 467
INLET 68
OUTLET 207

LIGHT ENDS COLUMN, F-2

REBOILER 428
LOWER SECTION 225
FEED SECTION 222
OVERHEAD -
S-8 72
S-9 _____

SOLVENT COLUMN, F-3

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

HEAVY ENDS COLUMN, F-4

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

VACUUM FLASH, F-O

OVERHEAD 381
BOTTOMS -

PRECIPITATION TOWER, V-8

ZONE (1) 319
(2) 405
(3) 519

RECOVERY TOWER V-9

REBOILER 383
OVERHEAD 342

PRESSURE, psig

SEPARATORS S-1 and S-2
2450

SEPARATOR S-3
240

SEPARATORS S-4A and S-4B
60

VACUUM FLASH, F-I
15 Torr

LIGHT ENDS COLUMN, F-2
Atm

SOLVENT COLUMN, F-3
NIU

HEAVY ENDS COLUMN, F-4
NIU

VACUUM FLASH, F-O
15 Torr

SETTLING TOWER, V-8
273

RECOVERY TOWER, V-7
4

	Start	End	Run No: M-34A
Date of Run	1/11/78	1/12/78	Units: lbs/hr, °F, psig
Time of Run	1900	0700	Prep By: BCL & GGB
Length of Run	12	hours	Updated: 6/7/78
Hours on Coal at End of Y. P.	41.4		

MASS RATES AND RUN CONDITIONS

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

V-8 Btms Adjusted
Revised: 6/7/78

Run No: M-34A
Date: 1/11-12/78
Time: 1900-0700

SUMMARY

Temperature: 735°F
Pressure: 2450 psig
LHSV: 1.47
GHSV: 337

	lbs/hr.		Net Yield		Wt % MAF Coal	
	Total In	Total Out				
STREAM DESCRIPTION						
SAMPLE POINT						
Hydrogen - H ₂	1.32	1.18	-0.14			
Carbon Monoxide - CO	16.90	11.00	-5.90			
Carbon Dioxide - CO ₂		12.91	12.91			
Hydrogen Sulfide - H ₂ S		0.30	0.30			
Methane - CH ₄		0.87	0.87	8.68	36.24	
Ethane - C ₂ H ₆		0.37	0.37			
Propane - C ₃ H ₈		0.18	0.18			
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.14	0.18	0.04			
Ammonia - NH ₃		0.05	0.05			
Light Oil - (0-100°C @ 1.6 Torr)	112.12	112.32	0.20			
Solvent - (100-230°C @ 1.6 Torr)	78.63	81.02	2.39	15.11	63.09	
Heavy Oil - (230-255°C @ 1.6 Torr)	1.96	1.91	-0.05			
SRL		12.57	12.57			
Coal (MAF)	23.95	2.08	-21.87		-91.32	
Ash	3.99	3.30	-0.69		-2.88	
Water	17.41	16.18	-1.23		-5.13	
Phenol						
TOTAL	256.42	256.42	-0-			

Carbon	195.45	193.85	-1.60			
Hydrogen	20.59	20.71	0.12			
Nitrogen	0.46	0.65	0.19			
Sulfur	1.66	1.57	-0.09			
Oxygen (by diff)	34.27	36.34	2.07			
Ash	3.99	3.30	-0.69			
TOTAL	256.42	256.42	-0-			

PROJECT LIGNITE

MEASURED & ADJUSTED MATERIAL BALANCE

Revised: 6/7/78

Run No: M-34A
Date: 1/11-12/78
Time: 1900-0700

MATERIAL IN

lbs/hr.

Temperature: 735°F
Pressure: 2450 psig
LHSV: 1.47
GHSV: 337

STREAM DESCRIPTION	Feed Gas	Lignite	Feed Solvent	Deashing Solvent		Total In
SAMPLE POINT	Sx-21	Sx-1	Sx-14	Sx-40		
Hydrogen - H ₂	1.32					1.32
Carbon Monoxide-CO	16.90					16.90
Carbon Dioxide - CO ₂						
Hydrogen Sulfide-H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen-N ₂	0.14					0.14
Ammonia-NH ₃						
Light Oil-(0-100°C @ 1.6 Torr)			18.82	93.30		112.12
Solvent-(100-230°C @ 1.6 Torr)			76.53	2.10		78.63
Heavy Oil-(230-255°C @1.6 Torr)			1.96			1.96
SRL						
Cool (MAF)		23.95				23.95
Ash		3.99				3.99
Water		16.73	0.68			17.41
Phenol						
TOTAL	18.36	44.67	97.99	95.40		256.42

Elemental	Carbon	7.24	17.45	85.06	85.70		195.45
	Hydrogen	1.32	2.99	8.02	8.26		20.59
	Nitrogen	0.14	0.13	0.13	0.06		0.46
	Sulfur		0.42	1.23	0.01		1.66
	Oxygen (by diff)	9.66	19.69	3.55	1.37		34.27
	Ash		3.99				3.99
	TOTAL	18.36	44.67	97.99	95.40		256.42

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Adjusted
Revised: 6/7/78

Run No: M-34A
Date: 1/11-12/78
Time: 1900-0700

MATERIAL OUT

Temperature: 735°F
Pressure: 2450 psig
LHSV: 1.47
GHSV: 337

lbs/hr.

Quantitative	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
	Hydrogen - H ₂	1.11	0.06				0.01
	Carbon Monoxide - CO	10.01	0.91				0.08
	Carbon Dioxide - CO ₂	8.96	3.54				0.41
	Hydrogen Sulfide - H ₂ S	0.14	0.16				
	Methane - CH ₄	0.74	0.12				0.01
	Ethane - C ₂ H ₆	0.25	0.10				0.02
	Propane - C ₃ H ₈	0.05	0.11				0.02
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.17	0.01				
	Ammonia - NH ₃	0.01	0.02	0.02			
	Light Oil - (0-100°C @ 1.6 Torr)			0.18		5.98	
	Solvent - (100-230°C @ 1.6 Torr)			0.07		0.24	
	Heavy Oil - (230-255°C @1.6 Torr)						
	SRL						
	Coal (MAF)						
	Ash						
	Water			15.29		0.35	
	Phenol						
	TOTAL	21.44	5.03	15.56	Negl.	6.57	0.55

Elemental	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)	Ash
	7.53	1.37	0.17	0.13	12.24	
	1.61	0.14	0.03	0.15	3.10	
	0.21	1.73	0.01		13.61	
		0.66		0.01	0.46	
		0.02			0.34	
	TOTAL	21.44	5.03	15.56	Negl.	6.57
						0.55

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

V-8 Btms Adjusted
Revised: 6/7/78

Temperature: 735°F
Pressure: 2450 psig
LHSV: 1.47
GHSV: 337

Run No: M-34A
Date: 1/11-12/78
Time: 1900-0700

MATERIAL OUT

lbs/hr.

	STREAM DESCRIPTION	Cold Trap	V-8 Btms	F-1 Btms	V-9 Ovhd	← Recycle S-7 Btms	Solvent → LEC Btms
	SAMPLE POINT	Sx-33	Calc	Sx-2	Sx-28	Sx-39	Sx-12
	Hydrogen - H ₂						
	Carbon Monoxide - CO						
	Carbon Dioxide - CO ₂						
	Hydrogen Sulfide - H ₂ S						
	Methane - CH ₄						
	Ethane - C ₂ H ₆						
	Propane - C ₃ H ₈						
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂						
	Ammonia - NH ₃						
Quantitative	Light Oil - (0-100°C @ 1.6 Torr)	0.09	5.35		84.14	11.31	5.27
	Solvent - (100-230°C @ 1.6 Torr)	0.13	2.03	5.35	1.46	65.14	6.60
	Heavy Oil - (230-255°C @ 1.6 Torr)					1.69	0.22
	SRL		0.36	12.21			
	Coal (MAF)		2.08				
	Ash		3.19	0.11			
	Water	0.05				0.49	
	Phenol						
	TOTAL	0.27	13.01	17.67	85.60	78.63	12.09
Elemental	Carbon	0.17	8.48	15.38	76.50	68.33	10.01
	Hydrogen	0.03	0.75	1.10	7.38	6.38	1.15
	Nitrogen		0.02	0.12	0.05	0.24	0.01
	Sulfur		0.06	0.18		0.94	0.10
	Oxygen (by diff)	0.07	0.51	0.78	1.67	2.74	0.82
	Ash		3.19	0.11			
	TOTAL	0.27	13.01	17.67	85.60	78.63	12.09

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Adjusted
Revised: 6/7/78

Run No: M-34A
Date: 1/11-12/78
Time: 1900-0700

MATERIAL OUT
lbs/hr.

Temperature: 735°F
Pressure: 2450 psig
LHSV: 1.47
GHSV: 337

Quantitative	STREAM DESCRIPTION					Total
	SAMPLE POINT					Out
	Hydrogen - H ₂					1.18
	Carbon Monoxide - CO					11.00
	Carbon Dioxide - CO ₂					12.91
	Hydrogen Sulfide - H ₂ S					0.30
	Methane - CH ₄					0.87
	Ethane - C ₂ H ₆					0.37
	Propane - C ₃ H ₈					0.18
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.18
	Ammonia - NH ₃					0.05
	Light Oil - (0-100°C @ 1.6 Torr)					112.32
	Solvent - (100-230°C @ 1.6 Torr)					81.02
	Heavy Oil - (230-255°C @1.6 Torr)					1.91
	SRL					12.57
	Coal (MAF)					2.08
	Ash					3.30
	Water					16.18
	Phenol					
	TOTAL					256.42

Elemental	Carbon					193.85
	Hydrogen					20.71
	Nitrogen					0.65
	Sulfur					1.57
	Oxygen (by diff)					36.34
	Ash					3.30
	TOTAL					256.42

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 6/7/78

Run No: M-34A

Date: 1/11-12/78

Time: 1900-0700

WEIGHT PERCENT

	WEIGHT PERCENT					
	Lignite	F-1 Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
SAMPLE DESCRIPTION						
SAMPLE POINT	Sx-1	Sx-2	Sx-12	Sx-14	Sx-16	Sx-16
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)						
Light Oils - (0-100°C @ 1.6 Torr)			43.60	19.21	96.10	
Solvent - (100-230°C @ 1.6 Torr)		30.30	54.60	78.10	3.90	
Heavy Oil - (230-255°C @1.6 Torr)			1.80	2.00		
SRL		69.10				
Coal (MAF)	53.61					
Ash	8.93	0.60				
Water	37.46			0.69		100
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)		100				
Melting Point °F		144				
Specific Gravity 60/60			0.964	1.005		
Viscosity, cp						

	WEIGHT PERCENT					
	Lignite	F-1 Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
Carbon	39.07	87.06	82.81	86.80	87.43	
Hydrogen	6.69	6.23	9.49	8.19	9.92	11.11
Nitrogen	0.30	0.68	0.10	0.13	0.06	
Sulfur	0.93	1.03	0.78	1.26	0.18	
Oxygen (by diff)	44.08	4.40	6.82	3.62	2.41	88.89
Ash	8.93	0.60				
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	2	2	2	2	2	-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 6/7/78

Run No: M-34A

Date: 1/11-12/78

Time: 1900-0700

WEIGHT PERCENT

SAMPLE DESCRIPTION	S-8 Total	Product Gas	Feed Gas	S-4B Oil	S-4B Water	S-4B Total
	Sx-16	Sx-19	Sx-21	*Sx-23	Sx-23	Sx-23
Hydrogen - H ₂		5.19	7.20			
Carbon Monoxide - CO		46.69	92.02			
Carbon Dioxide - CO ₂		41.79				
Hydrogen Sulfide - H ₂ S		0.66				
Methane - CH ₄		3.45				
Ethane - C ₂ H ₆		1.16				
Propane - C ₃ H ₈		0.24				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂		0.77	0.78			
Ammonia - NH ₃ (PPM)		0.05			0.10	0.10
Light Oils - (0-100°C @ 1.6 Torr)	91.05			72.63		1.18
Solvent - (100-230°C @ 1.6 Torr)	3.69			27.00		0.44
Heavy Oil - (230-255°C @1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water	5.26			0.37	99.90	98.28
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

ELEMENTAL	WEIGHT PERCENT					
	S-8 Total	Product Gas	Feed Gas	S-4B Oil	S-4B Water	S-4B Total
Carbon	82.83	35.13	39.44	81.32		1.32
Hydrogen	9.98	6.37	7.20	8.93	11.12	11.09
Nitrogen	0.06	0.81	0.78	0.13	0.08	0.08
Sulfur	0.17	0.62		0.59		0.01
Oxygen (by diff)	6.96	57.07	52.58	9.03	88.80	87.50
Ash						
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	-	2	2	2	2	-

*Insufficient oil sample for analysis. Assumed to be the same as the S-4A oil.

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 6/7/78.

Run No: M-34A

Date: 1/11-12/78

Time: 1900-0700

WEIGHT PERCENT

SAMPLE DESCRIPTION	V-8 Btms	V-9 Ovhd	S-4A Ovhd	J-1 Discharge	Cold Trap	S-7 Btms
	Sx-27	Sx-28	Sx-30	Sx-32	Sx-33	Sx-34/39
Hydrogen - H ₂			1.23	1.28		
Carbon Monoxide - CO			18.16	14.26		
Carbon Dioxide - CO ₂			70.36	73.96		
Hydrogen Sulfide - H ₂ S			3.23			
Methane - CH ₄			2.33	2.83		
Ethane - C ₂ H ₆			1.92	3.03		
Propane - C ₃ H ₈			2.16	4.64		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂			0.20			
Ammonia - NH ₃ (PPM)			0.41			
Light Oils - (0-100° C @ 1.6 Torr)	40.69	98.30			34.60	14.38
Solvent - (100-230° C @ 1.6 Torr)	24.69	1.70			46.10	82.85
Heavy Oil - (230-255° C @ 1.6 Torr)						2.15
SRL	15.72					
Coal (MAF)	6.32					
Ash	12.58					
Water					19.30	0.62
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)	92.77					
Melting Point °F						
Specific Gravity 60/60						1.008
Viscosity, cp						

Carbon	77.01	89.37	32.03	34.62	63.50	86.90
Hydrogen	6.71	8.62	2.84	3.44	8.66	8.12
Nitrogen	0.21	0.06	0.54		0.30	0.30
Sulfur	0.57		3.04		0.59	1.20
Oxygen (by diff)	2.92	1.95	61.55	61.94	26.95	3.48
Ash	12.58					
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	1	2	2	2	2	4

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 6/7/78

Run No: M-34A

Date: 1/11-12/78

Time: 1900-0700

WEIGHT PERCENT

SAMPLE DESCRIPTION	S-4A Oil	S-4A Water	S-4A Total	S-7 Btms	Deashing Solvent	V-8 Btms
	Sx-37	Sx-37	Sx-37	Sx-34/39	Sx-40	Calc
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)		0.10				
Light Oils - (0-100°C @ 1.6 Torr)	72.63		71.54	14.38	97.80	41.12
Solvent - (100-230°C @ 1.6 Torr)	27.00		26.60	82.85	2.20	15.60
Heavy Oil - (230-255°C @1.6 Torr)				2.15		
SRL						2.77
Coal (MAF)						15.99
Ash						24.52
Water	0.37	99.90	1.86	0.62		
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

Carbon	81.32		80.10	86.90	89.83	65.16
Hydrogen	8.93	11.12	8.97	8.12	8.66	5.76
Nitrogen	0.13	0.08	0.13	0.30	0.06	0.15
Sulfur	0.59		0.58	1.20	0.01	0.51
Oxygen (by diff)	9.03	88.80	10.22	3.48	1.44	3.90
Ash						24.52
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	2	-	-	4	2	-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 6/7/78

Run No: M-34A

Date: 1/11-12/78

Time: 1900-0700

MOLE PERCENT

SAMPLE DESCRIPTION	Product Gas	Feed Gas	S-4A Ovhd	J-1 Discharge		
	Sx-19	Sx-21	Sx-30	Sx-32		
SAMPLE POINT						
Hydrogen - H ₂	47.00	52.08	18.97	19.97		
Carbon Monoxide - CO	30.20	47.52	19.96	15.84		
Carbon Dioxide - CO ₂	17.20		49.22	52.28		
Hydrogen Sulfide - H ₂ S	0.35		2.92			
Methane - CH ₄	3.90		4.48	5.49		
Ethane - C ₂ H ₆	0.70		1.97	3.14		
Propane - C ₃ H ₈	0.10		1.51	3.28		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.50	0.40	0.22			
Ammonia - NH ₃ (PPM)	0.05		0.75			
Light Oils - (0-100° C @ 1.6 Torr)						
Solvent - (100-230° C @ 1.6 Torr)						
Heavy Oil - (230-255° C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water						
Phenol						
TOTAL	100	100	100	100		
Calc Mole Wt.	18.11	14.46	30.78	31.10		
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	2	2		

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-34A
DATE: 1/11-12/78
TIME: 1900-0700

SAMPLE DESCRIPTION	F-1 Btms	F-3 Btms	Feed Solv.	S-8 Btms	V-9 Ovhd
SAMPLE POINT	Sx-2	Sx-12	Sx-14	Sx-16	Sx-28
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5	5		
IBP		31.2	27.4		
5%		55.6	49.7		
10%		65.6	73.0		
20%		81.6	121.2		
30%		97.3	152.2		
40%		111.8	177.0		
50%		134.0	180.5		
60%		145.8	192.8		
70%		164.0	198.5		
80%		-	205.3		
90%		196.4	222.8		
95%		208.0	247.3		
DRY PT.		216.5	251.0		
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR				758	746
IBP				52	112
5%				93	113
10%				101	113
20%				108	113
30%				111	113
40%				113	113
50%				114	113
60%				115	113
70%				117	113
80%				120	114
90%				129	114
95%				196	116
DRY PT.				222	120
ASPHALTENE TEST					
WT % ASH	0.60				
WT % UNCONVERTED COAL	-				
WT % PRE-ASPHALTENES	25.90				
WT % ASPHALTENES	27.16				
WT % MALTENES & DIST. OIL	46.34				
TOTAL	100.00				
SAYBOLT VISCOSITY, SSU					
100° F		9.2	10.1		
210° F		8.1	8.4		
MISCELLANEOUS					
KF H ₂ O WT % (Raw Sx)			0.69		
INFRARED RATIO		0.16	0.26		
SP. GRAV. 60/60 F		0.964	1.005		

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES - BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES - N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

**PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-34A
DATE: 1/11-12/78
TIME: 1900-0700**

SAMPLE DESCRIPTION	V-9 Btms	Cold Trap Liq	S-7 Btms	S-4A Btms	Recycle Solv.
SAMPLE POINT	Sx-29	Sx-31/33	Sx-34	Sx-37	Sx-39
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR	5		5		5
IBP	100.5		25.4		69.8
5%	155.0		50.0		101.6
10%	171.0		75.0		121.0
20%	183.5		127.0		153.6
30%	192.0		155.0		172.8
40%	199.5		171.3		181.3
50%	205.0		183.7		189.2
60%	215.5		189.7		195.0
70%	236.0		196.0		203.6
80%	262.3		206.0		222.2
90%	269.0		230.0		252.8
95%	-		248.0		-
DRY PT.	-		-		252.8
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR		744		753.3	
IBP		73		67	
5%		108		107	
10%		112		114	
20%		117		121	
30%		124		130	
40%		134		143	
50%		244		178	
60%		292		226	
70%		324		262	
80%		-		303	
90%		-		339	
95%		-		-	
DRY PT.		350		344	
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F					10.5
210° F					8.6
MISCELLANEOUS					
KF H ₂ O WT% (Raw Sx)		19.30	0.62	0.37	
INFRARED RATIO			0.29		
SP. GRAV. 60/60 F			1.008		1.008

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
 ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
 MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
 DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-34A
DATE: 1/11-12/78
TIME: 1900-0700

SAMPLE DESCRIPTION		Deashing Solv.			
SAMPLE POINT		Sx-40			
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR					
VOLUME PERCENT IBP 5% 10% 20% 30% 40% 50% 60% 70% 80% 90% 95% DRY PT.	TEMPERATURE, °C				
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR		747.1			
VOLUME PERCENT IBP 5% 10% 20% 30% 40% 50% 60% 70% 80% 90% 95% DRY PT.	TEMPERATURE, °C	111			
		112			
		112			
		113			
		113			
		113			
		114			
		114			
		114			
		114			
		118			
DRY PT.		120			
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F					
210° F					
MISCELLANEOUS					
KF H ₂ O WT % (Raw Sx)					
INFRARED RATIO					
SP. GRAV. 60/60 F					

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 6/7/78

Run No: M-34A
Date: 1/11-12/78
Time: 1900-0700

MATERIAL OUT

Temperature: 735°F
Pressure: 2450 psig
LHSV: 1.47
GHSV: 337

lbs/hr.

STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
Hydrogen - H ₂	1.11	0.06				0.01
Carbon Monoxide - CO	10.01	0.91				0.08
Carbon Dioxide - CO ₂	8.96	3.54				0.41
Hydrogen Sulfide - H ₂ S	0.14	0.16				
Methane - CH ₄	0.74	0.12				0.01
Ethane - C ₂ H ₆	0.25	0.10				0.02
Propane - C ₃ H ₈	0.05	0.11				0.02
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.17	0.01				
Ammonia - NH ₃	0.01	0.02	0.02			
Light Oil - (0-100°C @ 1.6 Torr)			0.18		5.98	
Solvent - (100-230°C @ 1.6 Torr)			0.07		0.24	
Heavy Oil - (230-255°C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water			15.29		0.35	
Phenol						
TOTAL	21.44	5.03	15.56	Negl.	6.57	0.55

ELEMENTAL						
	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)	Ash
	7.53	1.37	0.17	0.13	12.24	
	1.61	0.14	0.03	0.15	3.10	
	0.21	1.73	0.01		13.61	
		0.66		0.01	0.46	
TOTAL	21.44	5.03	15.56	Negl.	6.57	0.55

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 6/7/78

Run No: M-34A
Date: 1/11-12/78
Time: 1900-0700

MATERIAL OUT

Temperature: 735°F
Pressure: 2450 psig
LHSV: 1.47
GHSV: 337

	lbs/hr.					
	Cold Trap	V-8 Btms	F-1 Btms	V-9 Ovhd	← Recycle S-7 Btms	Solvent → LEC Btms
SAMPLE POINT	Sx-32	Sx-27	Sx-2	Sx-28	Sx-39	Sx-12
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃						
Light Oil - (0-100°C @ 1.6 Torr)	0.09	5.56		84.14	11.31	5.27
Solvent - (100-230°C @ 1.6 Torr)	0.13	3.38	5.35	1.46	65.14	6.60
Heavy Oil - (230-255°C @ 1.6 Torr)					1.69	0.22
SRL		2.15	12.21			
Coal (MAF)		0.86				
Ash		1.72	0.11			
Water	0.05				0.49	
Phenol						
TOTAL	0.27	13.67	17.67	85.60	78.63	12.09

Carbon	0.17	10.52	15.38	76.50	68.33	10.01
Hydrogen	0.03	0.92	1.10	7.38	6.38	1.15
Nitrogen		0.03	0.12	0.05	0.24	0.01
Sulfur		0.08	0.18		0.94	0.10
Oxygen (by diff.)	0.07	0.40	0.78	1.67	2.74	0.82
Ash		1.72	0.11			
TOTAL	0.27	13.67	17.67	85.60	78.63	12.09

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 6/7/78

Run No: M-34A
Date: 1/11-12/78
Time: 1900-0700

MATERIAL OUT

lbs/hr.

Temperature: 735°F
Pressure: 2450 psig
LHSV: 1.47
GHSV: 337

Quantitative	STREAM DESCRIPTION					Total Out
	SAMPLE POINT					
	Hydrogen - H ₂					1.18
	Carbon Monoxide - CO					11.00
	Carbon Dioxide - CO ₂					12.91
	Hydrogen Sulfide - H ₂ S					0.30
	Methane - CH ₄					0.87
	Ethane - C ₂ H ₆					0.37
	Propane - C ₃ H ₈					0.18
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.18
	Ammonia - NH ₃					0.05
	Light Oil - (0-100°C @ 1.6 Torr)					112.53
	Solvent - (100-230°C @ 1.6 Torr)					82.37
	Heavy Oil - (230-255°C @ 1.6 Torr)					1.91
	SRL					14.36
	Cool (MAF)					0.86
	Ash					1.83
	Water					16.18
	Phenol					
	TOTAL					257.08

Elemental	Carbon					195.89
	Hydrogen					20.88
	Nitrogen					0.66
	Sulfur					1.59
	Oxygen (by diff)					36.23
	Ash					1.83
	TOTAL					257.08

RUN CONDITIONS

RUN NO. M-34D
DATE MADE 1/13/78

TEMPERATURE °F

PREHEATER E-I
SAND BATH 763
INLET 240
OUTLET 739

DISSOLVERS

R-1A (1) NIU
(2) _____
(3) _____
(4) _____
R-1B (1) 747
(2) 779
(3) 781
(4) 807

REACTOR PRODUCT SEPARATORS

S-1 524
S-2 50
S-3 474
S-4A 71
S-4B 74

PREHEATER E-II

DOWTHERM 658
INLET 428
OUTLET 620

VACUUM FLASH F-1

OVERHEAD VAPOR 532
UPPER WALL 602
LOWER WALL 584
DOWNCOMER 406
CONDENSATE
ACCUMULATOR, S-7 80

PREHEATER E-13

DOWTHERM 467
INLET 70
OUTLET 202

LIGHT ENDS COLUMN, F-2

REBOILER 410
LOWER SECTION 218
FEED SECTION 213
OVERHEAD _____
S-8 72
S-9 _____

SOLVENT COLUMN, F-3

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

HEAVY ENDS COLUMN, F-4

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

VACUUM FLASH, F-0

OVERHEAD NIU
BOTTOMS _____

PRECIPITATION TOWER, V-8

ZONE (1) NIU
(2) _____
(3) _____

RECOVERY TOWER V-9

REBOILER NIU
OVERHEAD _____

PRESSURE, psig

SEPARATORS S-1 and S-2
2450

SEPARATOR S-3
253

SEPARATORS S-4A and S-4B
61

VACUUM FLASH, F-1
14 Torr

LIGHT ENDS COLUMN, F-2
Atm

SOLVENT COLUMN, F-3
NIU

HEAVY ENDS COLUMN, F-4
NIU

VACUUM FLASH, F-0
NIU

SETTLING TOWER, V-8
NIU

RECOVERY TOWER, V-9
NIU

ADJUSTED

MASS RATES AND RUN
CONDITIONS

	Start	End	Run No: M-34D
Date of Run	1/13/78	1/13/78	Units: lb/hrs, °F, psig
Time of Run	0300	1500	Prep. By: FDN
Length of Run	12	hrs.	Updated: 4/26/78
Hours on Coal at End of Y.P.	72.9		Out streams, except recycle solvent normalized.

153.48 TOTAL

 $\square$ OPEN $\blacksquare$ CLOSED

TOTAL 153.48

Numbers in parentheses are not included in the totals.

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
Normalized with Forced Carbon Balance
Revised: 4/26/78

Run No: M-34D
Date: 1/13/78
Time: 0300-1500

SUMMARY

Temperature: 806°F
Pressure: 2450 psig
LHSV: 1.40
GHSV: 327

lbs/hr.

	STREAM DESCRIPTION	Total In	Total Out	Net Yield		
					Wt % MAF Coal	
Quantitative	SAMPLE POINT					
	Hydrogen - H ₂	1.32	1.16	-0.16		
	Carbon Monoxide - CO	15.91	9.44	-6.47		
	Carbon Dioxide - CO ₂		12.86	12.86		
	Hydrogen Sulfide - H ₂ S		0.29	0.29		
	Methane - CH ₄		0.76	0.76		
	Ethane - C ₂ H ₆		0.33	0.33		
	Propane - C ₃ H ₈		0.26	0.26	7.92	34.08
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂	0.15	0.13	-0.02		
	Ammonia - NH ₃		0.07	0.07		
	Light Oil - (0-100°C @ 1.6 Torr)	18.36	20.05	1.69		
	Solvent - (100-230°C @ 1.6 Torr)	70.01	71.25	1.24		
	Heavy Oil - (230-255°C @ 1.6 Torr)	2.85	3.48	0.63	16.15	69.49
	SRL		12.59	12.59		
	Coal (MAF)	23.24	1.56	-21.68		-93.29
	Ash	3.93	3.26	-0.67		-2.88
	Water	17.71	15.99	-1.72		-7.40
	Phenol					
	TOTAL	153.48	153.48	-0-		
Elemental	Carbon	103.30	103.30	-0-		
	Hydrogen	11.91	11.73	-0.18		
	Nitrogen	0.48	0.60	+0.12		
	Sulfur	1.49	1.52	+0.03		
	Oxygen (by diff)	32.37	33.07	0.70		
	Ash	3.93	3.26	-0.67		
	TOTAL	153.48	153.48	-0-		

PROJECT LIGNITE
MEASURED & ADJUSTED MATERIAL BALANCE

Revised: 4/26/78

Run No: M-34D
Date: 1/13/78
Time: 0300-1500

MATERIAL IN

Temperature: 806°F
Pressure: 2450 psig
LHSV: 1.40
GHSV: 327

lbs/hr

STREAM DESCRIPTION	Feed Gas	Lignite	Feed Solvent			Total In
	Sx-21	Sx-1	Sx-14			
Hydrogen - H ₂	1.32					1.32
Carbon Monoxide - CO	15.91					15.91
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.15					0.15
Ammonia - NH ₃						
Light Oil - (0-100°C @ 1.6 Torr)			18.36			18.36
Solvent - (100-230°C @ 1.6 Torr)			70.01			70.01
Heavy Oil - (230-255°C @ 1.6 Torr)			2.85			2.85
SRL						
Coal (MAF)		23.24				23.24
Ash		3.93				3.93
Water		17.05	0.66			17.71
Phenol						
TOTAL	17.38	44.22	91.88			153.48

Carbon	6.82	16.93	79.55			103.30
Hydrogen	1.32	2.99	7.60			11.91
Nitrogen	0.15	0.13	0.20			0.48
Sulfur		0.41	1.08			1.49
Oxygen (by diff)	9.09	19.83	3.45			32.37
Ash		3.93				3.93
TOTAL	17.38	44.22	91.88			153.48

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
Normalized with Forced Carbon Balance
Revised: 4/26/78

Run No: M-34D
Date: 1/13/78
Time: 0300-1500

MATERIAL OUT

Temperature: 806°F
Pressure: 2450 psig
LHSV: 1.40
GHSV: 327

lbs/hr.

Quantitative	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Sx-23	Sx-44	Calc	Sx-32
	Hydrogen - H ₂	1.06	0.09				0.01
	Carbon Monoxide - CO	8.53	0.82				0.09
	Carbon Dioxide - CO ₂	9.12	3.36				0.38
	Hydrogen Sulfide - H ₂ S	0.13	0.16				
	Methane - CH ₄	0.65	0.10				0.01
	Ethane - C ₂ H ₆	0.23	0.09				0.01
	Propane - C ₃ H ₈	0.12	0.12				0.02
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.11	0.02				
	Ammonia - NH ₃	0.03	0.03	0.01			
	Light Oil - (0-100°C @ 1.6 Torr)					2.04	
	Solvent - (100-230°C @ 1.6 Torr)					0.02	
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Coal (MAF)						
	Ash						
	Water			14.33		0.89	
	Phenol						
	TOTAL	19.98	4.79	14.34	Negl.	2.95	0.52

Elemental	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)	Ash
	6.92	1.30	0.13	0.12	11.51	
	1.52	0.17	0.04	0.15	2.91	
		1.59	0.01		12.74	
		0.31			0.92	
	1.72	0.02				0.33
	TOTAL	19.98	4.79	14.34	Negl.	2.95
						0.52

PROJECT LIGNITE
ADJUSTED MATERAIL BALANCE
Normalized with Forced Carbon Balance
Revised: 4/26/78

Run No: M-34D
Date: 1/13/78
Time: 0300-1500

MATERIAL OUT
lbs/hr.

Temperature: 806°F
Pressure: 2450 psig
LHSV : 1.40
GHSV: 327

Quantitative	STREAM DESCRIPTION	Cold Trap	F-1 Btms	← Recycle S-7 Btms	Solvent → LEC Btms	Total Out
	SAMPLE POINT	Sx-33	Sx-2	Sx-39	Sx-12	
	Hydrogen - H ₂					1.16
	Carbon Monoxide - CO					9.44
	Carbon Dioxide - CO ₂					12.86
	Hydrogen Sulfide - H ₂ S					0.29
	Methane - CH ₄					0.76
	Ethane - C ₂ H ₆					0.33
	Propane - C ₃ H ₈					0.26
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.13
	Ammonia - NH ₃					0.07
	Light Oil - (0-100°C @ 1.6 Torr)	0.30		14.19	3.52	20.05
	Solvent - (100-230°C @ 1.6 Torr)		0.65	68.67	1.91	71.25
	Heavy Oil - (230-255°C @ 1.6 Torr)			3.30	0.18	3.48
	SRL		12.59			12.59
	Cool (MAF)		1.56			1.56
	Ash		3.26			3.26
	Water			0.77		15.99
	Phenol					
	TOTAL	0.30	18.06	86.93	5.61	153.48

Elemental	Carbon	0.26	12.66	75.30	4.75	103.30
	Hydrogen	0.03	0.83	6.94	0.54	11.73
	Nitrogen	Negl.	0.15	0.26	0.01	0.60
	Sulfur	Negl.	0.16	1.06	0.03	1.52
	Oxygen (by diff)	0.01	1.00	3.37	0.28	33.07
	Ash		3.26			3.26
	TOTAL	0.30	18.06	86.93	5.61	153.48

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 4/26/78

Run No: M-34D

Date: 1/13/78

Time: 0300-1500

WEIGHT PERCENT

SAMPLE DESCRIPTION	WEIGHT PERCENT					
	Lignite	F-1 Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
SAMPLE POINT	Sx-1	Sx-2	Sx-12	Sx-14	Sx-16	Sx-16
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)						
Light Oils - (0-100° C @ 1.6 Torr)			62.80	19.98	98.90	
Solvent - (100-230° C @ 1.6 Torr)		3.60	34.10	76.20	1.10	
Heavy Oil - (230-255° C @1.6 Torr)			3.10	3.10		
SRL		69.68				
Coal (MAF)	52.55	8.66				
Ash	8.88	18.06				
Water	38.57			0.72		100
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)		89.43				
Melting Point °F		258				
Specific Gravity 60/60			0.949	1.010		
Viscosity, cp						

Carbon	38.30	70.11	84.64	86.58	83.59	
Hydrogen	6.77	4.58	9.60	8.27	10.18	11.11
Nitrogen	0.29	0.81	0.22	0.22	0.00	
Sulfur	0.92	0.90	0.56	1.18	0.06	
Oxygen (by diff)	44.84	5.54	4.98	3.75	6.17	88.89
Ash	8.88	18.06				
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	2	2	2	2	2	-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 4/26/78

Run No: M-34D

Date: 1/13/78

Time: 0300-1500

WEIGHT PERCENT

SAMPLE DESCRIPTION	S-8	Product	Feed	S-4B	S-4B	S-4B
	Total	Gas	Gas	Oil	Water	Total
SAMPLE POINT	Sx-16	Sx-19	Sx-21	*Sx-23	Sx-23	Sx-23
Hydrogen - H ₂		5.28	7.59			
Carbon Monoxide - CO		42.70	91.52			
Carbon Dioxide - CO ₂		45.66				
Hydrogen Sulfide - H ₂ S		0.65				
Methane - CH ₄		3.26				
Ethane - C ₂ H ₆		1.16				
Propane - C ₃ H ₈		0.61				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂		0.54	0.89			
Ammonia - NH ₃ (PPM)		0.14			0.06	0.06
Light Oils - (0-100°C @ 1.6 Torr)	98.90			71.42		
Solvent - (100-230°C @ 1.6 Torr)	1.10			28.00		
Heavy Oil - (230-255°C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water				0.58	99.94	99.94
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

Carbon	83.59	34.62	39.22	82.92		
Hydrogen	10.18	6.50	7.59	9.44	11.11	11.11
Nitrogen	0.00	0.66	0.89	0.16	0.05	0.05
Sulfur	0.06	0.61		0.53		
Oxygen (by diff)	6.17	57.61	52.30	6.95	88.84	88.84
Ash						
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	-	2	2	2	2	-

*Insufficient oil sample for analysis. Assumed to be the same as the S-4A oil.

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 4/26/78

Run No: M-34D

Date: 1/13/78

Time: 0300-1500

WEIGHT PERCENT

SAMPLE DESCRIPTION	S-4A Ovhd	J-1 Discharge	Cold Trap	S-7 Btms	S-4A Oil	S-4A Water
	Sx-30	Sx-32	*Sx-33	Sx-34/39	Sx-37	Sx-37
Hydrogen - H ₂	1.84	1.60				
Carbon Monoxide - CO	17.06	16.66				
Carbon Dioxide - CO ₂	70.13	73.86				
Hydrogen Sulfide - H ₂ S	3.35					
Methane - CH ₄	2.11	2.37				
Ethane - C ₂ H ₆	1.96	2.63				
Propane - C ₃ H ₈	2.63	2.88				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.35					
Ammonia - NH ₃ (PPM)	0.57					0.07
Light Oils - (0-100°C @ 1.6 Torr)			99.76	16.32	71.42	
Solvent - (100-230°C @ 1.6 Torr)				79.00	28.00	
Heavy Oil - (230-255°C @1.6 Torr)				3.80		
SRL						
Coal (MAF)						
Ash						
Water			0.24	0.88	0.58	99.93
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60				1.022		
Viscosity, cp						

Carbon	31.74	33.52	86.46	86.62	82.92	
Hydrogen	3.54	3.24	8.24	7.98	9.44	11.11
Nitrogen	0.82		0.16	0.30	0.16	0.06
Sulfur	3.15		1.25	1.22	0.53	
Oxygen (by diff)	60.75	63.24	3.89	3.88	6.95	88.83
Ash						
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	2	2	2	4	2	2

*Insufficient sample for distillation. Assumed to be water and light oil.

PROJECT LIGNITE

SAMPLE ANALYSIS

Updated: 4/26/78

Run No: M-34D

Date: 1/13/78

Time: 0300-1500

WEIGHT PERCENT

Quantitative	SAMPLE DESCRIPTION	S-4A Total	S-7 Btms		S-8 Btms		
	SAMPLE POINT	Sx-37	Sx-34/39		Calc.		
	Hydrogen - H ₂						
	Carbon Monoxide - CO						
	Carbon Dioxide - CO ₂						
	Hydrogen Sulfide - H ₂ S						
	Methane - CH ₄						
	Ethane - C ₂ H ₆						
	Propane - C ₃ H ₈						
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂						
	Ammonia - NH ₃ (PPM)	0.01					
	Light Oils - (0-100°C @ 1.6 Torr)	64.33	16.32		69.06		
	Solvent - (100-230°C @ 1.6 Torr)	25.22	79.00		0.77		
	Heavy Oil - (230-255°C @1.6 Torr)		3.80				
	SRL						
	Coal (MAF)						
	Ash						
	Water	10.44	0.88		30.17		
	Phenol						
	TOTAL	100	100		100		
	Pyridine Solubles (Wt % Ash free)						
	Melting Point °F						
	Specific Gravity 60/60		1.022				
	Viscosity, cp						

Elemental	Carbon	74.69	86.62		58.37		
	Hydrogen	9.60	7.98		10.46		
	Nitrogen	0.15	0.30		-		
	Sulfur	0.48	1.22		0.04		
	Oxygen (by diff)	15.08	3.88		31.13		
	Ash						
	TOTAL	100	100		100		
Elemental	Number of Sample Avg.	-	4		-		

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 4/26/78

Run No: M-34D

Date: 1/13/78

Time: 0300-1500

MOLE PERCENT

SAMPLE DESCRIPTION	Product Gas	Feed Gas	S-4A Ovhd	J-1 Exhaust		
SAMPLE POINT	Sx-19	Sx-21	Sx-30	Sx-32		
Hydrogen - H ₂	47.95	53.48	26.05	23.68		
Carbon Monoxide - CO	27.70	46.07	17.30	17.64		
Carbon Dioxide - CO ₂	18.85		45.25	49.76		
Hydrogen Sulfide - H ₂ S	0.35		2.80			
Methane - CH ₄	3.70		3.75	4.38		
Ethane - C ₂ H ₆	0.70		1.85	2.60		
Propane - C ₃ H ₈	0.25		1.70	1.94		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.35	0.45	0.35			
Ammonia - NH ₃ (PPM)	0.15		0.95			
Light Oils - (0-100°C @ 1.6 Torr)						
Solvent - (100-230°C @ 1.6 Torr)						
Heavy Oil - (230-255°C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water						
Phenol						
TOTAL	100	100	100	100		
Calc Mole Wt.	18.16	14.10	28.39	29.64		
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

ELEMENTAL						
Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	2	2		

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-34D
DATE: 1/13/78
TIME: 0300-1500

SAMPLE DESCRIPTION	F-1 Btms	F-3 Btms	Feed Solv.	S-8 Btms	S-7 Btms
SAMPLE POINT	Sx-2	Sx-12	Sx-14	Sx-16	Sx-34
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5	5		5
IBP		37.0	23.0		24.6
5%		53.6	80.4		86.0
10%		60.8	92.3		101.6
20%		71.6	121.0		128.6
30%		80.2	153.0		156.6
40%		89.5	171.6		173.0
50%		99.0	181.0		182.8
60%		-	188.5		190.0
70%		136.2	196.4		198.2
80%		163.0	207.8		209.6
90%		184.3	243.2		243.3
95%		199.3	260.0		260.0
DRY PT.		207.5	-		-
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR				744.8	
IBP				50	
5%				84	
10%				92	
20%				101	
30%				106	
40%				110	
50%				112	
60%				114	
70%				115	
80%				118	
90%				121	
95%				136	
DRY PT.				136	
ASPHALTENE TEST					
WT % ASH	18.06				
WT % UNCONVERTED COAL	8.66				
WT % PRE-ASPHALTENES	30.21				
WT % ASPHALTENES	21.46				
WT % MALTENES & DIST. OIL	21.61				
TOTAL	100.00				
SAYBOLT VISCOSITY, SSU					
100° F		9.0	11.5		
210° F		7.6	8.4		
MISCELLANEOUS					
KF H ₂ O WT % (Raw Sx)			0.72		0.88
INFRARED RATIO		0.17	0.25		0.26
SP. GRAV. 60/60 F		0.949	1.010		1.022

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-34D
DATE: 1/13/78
TIME: 0300-1500

SAMPLE DESCRIPTION	S-4A Btms	Recycle Solv.		
SAMPLE POINT	Sx-37	Sx-39		
ASTM D-1160 DISTILLATION DATA				
PRESSURE, TORR		5		
IBP		27.4		
5%		90.2		
10%		104.3		
20%		134.0		
30%		162.6		
40%		176.6		
50%		185.2		
60%		191.3		
70%		199.0		
80%		209.3		
90%		-		
95%		-		
DRY PT.		-		
ASTM D-86 DISTILLATION DATA				
BAROMETRIC PRESSURE, TORR	745.7			
IBP	70			
5%	88			
10%	124			
20%	148			
30%	173			
40%	200			
50%	217			
60%	241			
70%	266			
80%	296			
90%	346			
95%	-			
DRY PT.	356			
ASPHALTENE TEST				
WT % ASH				
WT % UNCONVERTED COAL				
WT % PRE-ASPHALTENES				
WT % ASPHALTENES				
WT % MALTENES & DIST. OIL				
TOTAL				
SAYBOLT VISCOSITY, SSU				
100° F				12.3
210° F				8.5
MISCELLANEOUS				
KF H ₂ O WT% (Raw Sx)	0.58	0.88		
INFRARED RATIO	-	0.26		
SP. GRAV. 60/60 F	-	1.022		

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 4/26/78

Run No: M-34D
Date: 1/13/78
Time: 0300-1500

MATERIAL OUT

Temperature: 806°F
Pressure: 2450 psig
LHSV: 1.40
GHSV: 327

	lbs/hr.					
	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
STREAM DESCRIPTION	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
SAMPLE POINT						
Hydrogen - H ₂	1.10	0.09				0.01
Carbon Monoxide - CO	8.93	0.85				0.09
Carbon Dioxide - CO ₂	9.54	3.51				0.40
Hydrogen Sulfide - H ₂ S	0.14	0.17				
Methane - CH ₄	0.68	0.11				0.01
Ethane - C ₂ H ₆	0.24	0.10				0.01
Propane - C ₃ H ₈	0.13	0.13				0.02
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.11	0.02				
Ammonia - NH ₃	0.03	0.03	0.01			
Light Oil - (0-100°C @ 1.6 Torr)					3.05	
Solvent - (100-230°C @ 1.6 Torr)					0.03	
Heavy Oil - (230-255°C @ 1.6 Torr)						
SRL						
Cool (MAF)						
Ash						
Water			14.99			
Phenol						
TOTAL	20.90	5.01	15.00	Negl.	3.08	0.54

	lbs/hr.					
	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
Carbon	7.23	1.59			2.58	0.18
Hydrogen	1.36	0.18	1.67		0.31	0.02
Nitrogen	0.14	0.04	0.01			
Sulfur	0.13	0.16				
Oxygen (by diff)	12.04	3.04	13.32		0.19	0.34
Ash						
TOTAL	20.90	5.01	15.00	Negl.	3.08	0.54

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 4/26/78

Run No: M-34D
Date: 1/13/78
Time: 0300-1500

MATERIAL OUT

Temperature: 806°F
Pressure: 2450 psig
LHSV : 1.40
GHSV: 327

lbs/hr.

STREAM DESCRIPTION	Cold Trap	F-1 Btms	← Recycle S-7 Btms	Solvent → LEC Btms		Total Out
	Sx-33	Sx-2	Sx-39	Sx-12		
Hydrogen - H ₂						1.20
Carbon Monoxide - CO						9.87
Carbon Dioxide - CO ₂						13.45
Hydrogen Sulfide - H ₂ S						0.31
Methane - CH ₄						0.80
Ethane - C ₂ H ₆						0.35
Propane - C ₃ H ₈						0.28
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						0.13
Ammonia - NH ₃						0.07
Light Oil - (0-100°C @ 1.6 Torr)	0.31		14.19	3.52		21.07
Solvent - (100-230°C @ 1.6 Torr)		0.68	68.67	1.91		71.29
Heavy Oil - (230-255°C @ 1.6 Torr)			3.30	0.18		3.48
SRL		13.16				13.16
Coal (MAF)		1.64				1.64
Ash		3.41				3.41
Water			0.77			15.76
Phenol						
TOTAL	0.31	18.89	86.93	5.61		156.27

Carbon	0.27	13.24	75.30	4.75		105.14
Hydrogen	0.03	0.87	6.94	0.54		11.92
Nitrogen		0.15	0.26	0.01		0.61
Sulfur		0.17	1.06	0.03		1.55
Oxygen (by diff)	0.01	1.05	3.37	0.28		33.64
Ash		3.41				3.41
TOTAL	0.31	18.89	86.93	5.61		156.27

APPENDIX B

Process Development Unit Run M-35 January 23-27, 1978

Run M-35 was the fifth in a series of six runs made to evaluate the effects of lignite variation on plant operability and liquefaction yields. Lignite mined by the Consolidation Coal Company near Velva, North Dakota was used. Velva lignite is similar to Zap lignite in chemical makeup as illustrated by the proximate and ultimate analyses listed on the following page. Slight variations, however, do exist in the chemical composition of the ash as determined by X-Ray fluorescence and as shown below.

X-Ray Fluorescence Analysis of Ash, Wt %

North Dakota Lignite	Zap	Velva
Loss on Ignition		
SiO ₂	20.2	28.4
Al ₂ O ₃	10.5	14.1
Fe ₂ O ₃	10.0	4.5
TiO ₂	0.5	0.5
P ₂ O ₅	0.6	0.9
CaO	26.7	31.2
MgO	6.8	9.1
Na ₂ O	6.7	1.8
K ₂ O	0.4	0.6
So ₂	17.2	8.9
Total	100.0	100.0

Nominal operating conditions employed in Run M-35 were similar to those used in previous runs made with lignite from other North Dakota locations and were 820-830°F maximum reaction temperature and 2500 psig dissolver operating pressure with a solvent to coal ratio of 1.9 and gas and slurry hourly space velocities of 1.43 and 330, respectively. One reactor volume was used and all recoverable organic liquids boiling above about 300°F at one atmosphere were recycled. All product gases were sampled, measured and then thermally oxidized. The product deashing system was on stream during part of the run.

Run M-35 was smooth and largely trouble free with regard to liquefaction operations; however, several problems with the slurry deashing transfer and charging system prevented long term integrated product deashing. Approximately 44 hours of continuous solid-liquid unit operations had been logged with one yield period completed before a severe leak developed at the packing gland of the toluene-slurry circulating pump P-5. This leak allowed the toluene in the steam traced transfer loop to escape leaving the loop plugged with thick, heavy F-0 vacuum bottoms material. The deashing unit was isolated, the recirculating pump was repaired, and the toluene slurry loop was cleaned. The deashing unit was

Analysis of North Dakota Lignites

Mine Location	Zap	Velva
---------------	-----	-------

Proximate Analysis, Wt %

Moisture	30.91	29.58
Ash	6.04	5.91
Volatile Matter	37.65	42.45
Fixed Carbon	25.40	22.06
Total	100.00	100.00

Ultimate Analysis, Wt % (Dry Basis)

Carbon	68.82	65.99
Hydrogen	4.10	4.23
Nitrogen	0.88	0.83
Oxygen (By difference)	16.72	20.22
Sulfur	0.74	0.34
Ash	8.74	8.39
Total	100.00	100.00

back on line within 12 hours of the initial upset and was operated another 20 hours before the bottoms line from the partial vacuum flash drum, F-0, plugged. With only 33 hours remaining of the scheduled 120, and with the F-0 bottoms line plugged, the deashing equipment was shutdown. Liquefaction operations were continued and a total of 98 hours of coal processing were logged which amounted to 81.7 percent of the total time scheduled or 94 percent of the scheduled coal processing time. During the last eight hours of the run, the liquefaction reactor was cooled and depressured into the reactor blowdown vessel. The reactor solids were then recovered and separated from the liquids by decantation, weighed and sampled.

Three yield periods were started during Run M-35 but only two were completed. Data period M-35B was cut short and incomplete when the packing in the toluene slurry circulating pump failed and the toluene slurry transfer loop plugged.

The two completed yield periods, M-35A & C, were worked up quantitatively and these data along with the sample analyses, average run conditions, and oil phase ASTM D86 and D1160 distillations are attached.

Material balance closure for the two completed yield periods were 99.5 and 100.9 percent. Data period M-35A, which was made with product deashing, was adjusted to 100 percent recovery by increasing the V-8 bottoms rate to correct for material buildup in the solids precipitation tower, which on cleaning was found to contain approximately 305 pounds of solids that had accumulated over about 64 hours of operation. The V-8 bottoms composition was calculated by making a material balance around the deashing section. This calculated analysis should represent closely the composition of the V-8 bottoms had not some of the solids accumulated in the settling tower.

Yield period M-35C, which involved the liquefaction section only, was adjusted to 100 percent recovery by normalizing the outgoing product streams because no specific source of error was indicated. A comparison of the two material balance periods after adjustment is shown on the following page.

Net gas and organic liquid yields averaged 30.4 and 59.8 weight percent of the MAF lignite charged, respectively. Distillable oils averaged 12.5 percent of the total net organic liquid yield while SRL comprised the remaining 87.5 percent.

Conversion of MAF lignite to gas, oil and SRL averaged 87.9 percent. Reducing gas consumption averaged 2.4 SCF per pound of raw lignite charged or on a hydrogen equivalent basis 2.1 weight percent of the MAF lignite.

Process Development Unit Run M-35

Yield Period	M-35A	M-35C	Average
No of Reactors	1	1	1
LHSV	1.44	1.39	1.42
GHSV	354	360	357
Solvent/MAF Coal Ratio	3.41	3.11	3.26
H ₂ /CO Mole Ratio	1.12	1.11	1.12
Temperatures, °F			
Preheater Outlet, E-1	760	730	745
Reactor 3 ft	755	744	750
Reactor 7 ft	795	802	798
Reactor 11 ft	802	790	796
Reactor 15 ft	820	823	822
Max Reaction	820	823	822
Vacuum Flash Ovhd	519	519	519
Dissolver Pressure, psig	2500	2500	2500
Solvent Recycle, %			
Actual	94.4	95.8	95.1
Potential	100.4	104.4	102.4
Yields, Wt % MAF Lignite			
Net Gas	31.7	29.0	30.4
Net Liquid	55.0	64.7	59.8
(Dist Oil)	(1.3)	(13.8)	(7.5)
(SRL)	(53.7)	(50.9)	(52.3)
Net H ₂ O and Ash	0.6	-5.2	-2.3
Unconverted Coal	12.7	11.5	12.1
Overall Material Balance %	99.5	100.9	100.2

Actual solvent recovery for recycle averaged 95.1 percent with a calculated potential recovery on a total oil basis of 102.4 percent.

Results of the deashing operation during yield period M-35A were good with the product ash reduced to 0.8 weight percent. The concentration of pyridine insoluble material in the finished product after deashing was negligible.

At the end of Run M-35, approximately 66 pounds of oily solids and 27 pounds of solvent were recovered from the liquefaction reactor by blowdown. The wet solids were analyzed for pyridine soluble material and ash and then a representative portion was hot toluene washed, dried and the tests repeated. The results are shown below.

Reactor Solids Analysis, Wt %	Raw Reactor Solids	Toluene-Washed Reactor Solids
Pyridine Solubles	41.5	1.9
Pyridine Insolubles	28.3	40.6
Ash	30.2	57.5
Total	100.0	100.0

The dried toluene washed reactor solids were analyzed further by sieving and X-Ray diffraction. In addition, X-Ray fluorescence was carried out on the ash obtained from the toluene-washed solids. The results of these tests are shown on the following page along with some pertinent information for the M-35 feed coal and M-35C F-1 vacuum bottoms.

Analyses of Run M-35 Feed Coal and Toluene Washed Reactor Solids

Sieve Analysis

U.S. Screen Sieve Mesh	Total Percentages			
	On		Passing	
	Feed Coal	Reactor Solids	Feed Coal	Reactor Solids
65	2.6	1.1	97.4	98.9
100	13.0	17.9	87.0	82.1
140	28.2	61.7	71.8	38.3
170	38.1	81.5	61.9	18.5
230	56.2	97.3	43.8	2.7
325	76.2	99.4	23.8	0.6
Pan	100.0	100.0	0.0	0.0

X-Ray Diffraction Pattern Analysis

"Toluene-Washed Reactor Solids"
Identifiable Minerals

Calcite - most abundant
Dolomite - about 3/4 as abundant as calcite
Quartz - 1/3 as abundant as calcite
Feldspar - 1/3 as abundant as calcite

X-Ray Fluorescence Analysis of Ash, Wt %

	Feed Coal	Reactor Solids	*M-35C F-1 Btms
Loss on Ignition			5.0
SiO ₂	28.4	16.2	17.9
Al ₂ O ₃	14.1	12.0	15.0
Fe ₂ O ₃	4.5	4.2	5.6
TiO ₂	0.5	0.5	0.4
P ₂ O ₅	0.9	0.6	0.2
CaO	31.2	42.2	33.7
MgO	9.1	15.4	11.9
Na ₂ O	1.8	3.5	0.0
K ₂ O	0.6	0.2	1.2
SO ₂	8.9	5.2	9.1
Total	100.0	100.0	100.0

*Ash analysis obtained by atomic absorption and colormetric methods.

RUN CONDITIONS

RUN NO. M-35A
DATE MADE 1/24/78

TEMPERATURE °F

PREHEATER E-I
SAND BATH 780
INLET 241
OUTLET 760

DISSOLVERS

R-1A (1) _____
(2) _____
(3) _____
(4) _____
R-1B (1) 755
(2) 795
(3) 802
(4) 820

REACTOR PRODUCT SEPARATORS

S-1 581
S-2 46
S-3 477
S-4A 68
S-4B 72

PREHEATER E-II

DOWTHERM 539
INLET 355
OUTLET 630

VACUUM FLASH F-I

OVERHEAD VAPOR 519
UPPER WALL 620
LOWER WALL 664
DOWNCOMER 407
CONDENSATE
ACCUMULATOR, S-7 83

PREHEATER E-13
DOWTHERM 466
INLET 69
OUTLET 198

LIGHT ENDS COLUMN, F-2

REBOILER 323
LOWER SECTION 258
FEED SECTION 208
OVERHEAD 188
S-8 67
S-9 -

SOLVENT COLUMN, F-3

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

HEAVY ENDS COLUMN, F-4

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

VACUUM FLASH, F-O

OVERHEAD 451
BOTTOMS -

PRECIPITATION TOWER, V-8

ZONE (1) 277
(2) 430
(3) 440

RECOVERY TOWER V-9

REBOILER 434
OVERHEAD 322

PRESSURE, psig

SEPARATORS S-1 and S-2
2500

SEPARATOR S-3
234

SEPARATORS S-4A and S-4B
60

VACUUM FLASH, F-1
15 Torr

LIGHT ENDS COLUMN, F-2
Atm

SOLVENT COLUMN, F-3
NIU

HEAVY ENDS COLUMN, F-4
NIU

VACUUM FLASH, F-O
15 Torr

SETTLING TOWER, V-8
272

RECOVERY TOWER, V-7
5

	Start	End	Run No: M-35A
Date of Run	1/24/78	1/24/78	Units: lbs/hr, °F, psig
Time of Run	0700	1900	Prep. By: BCL & GGB
Length of Run	12	hrs.	Updated: 5/5/78
Hours on Coal at End of Y.P.	28.5		

MASS RATES AND RUN CONDITIONS

Numbers in parentheses are not included in the totals.

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Rate Adjusted
Revised: 5/5/78

Run No: M-35A
Date: 1/24/78
Time: 0700-1900

SUMMARY

Temperature: 760°F
Pressure: 2500 psig
LHSV: 1.44
GHSV: 354

lbs/hr.

	STREAM DESCRIPTION	Total In	Total Out	Net Yield	Wt % MAF Coal	
Quantitative	SAMPLE POINT					
	Hydrogen - H ₂	1.41	1.30	-0.11		
	Carbon Monoxide - CO	17.56	14.14	-3.42		
	Carbon Dioxide - CO ₂		10.48	10.48		
	Hydrogen Sulfide - H ₂ S		0.10	0.10		
	Methane - CH ₄		0.76	0.76		
	Ethane - C ₂ H ₆		0.44	0.44	8.60	31.71
	Propane - C ₃ H ₈		0.30	0.30		
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂	0.11	0.14	0.03		
	Ammonia - NH ₃		0.02	0.02		
	Light Oil - (0-100°C @ 1.6 Torr)	112.65	108.46	-4.19		
	Solvent - (100-230°C @ 1.6 Torr)	72.11	76.12	4.01	14.92	55.01
	Heavy Oil - (230-255°C @ 1.6 Torr)	3.06	3.59	0.53		
	SRL		14.57	14.57		
	Coal (MAF)	27.12	3.43	-23.69		-87.35
	Ash	2.75	2.66	-0.09		-0.33
	Water	16.22	16.48	0.26		0.96
	Phenol					
	TOTAL	252.99	252.99	-0-		

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					

PROJECT LIGNITE
MEASURED & ADJUSTED MATERIAL BALANCE
Revised: 5/5/78

Run No: M-35A
Date: 1/24/78
Time: 0700-1900

MATERIAL IN

Temperature: 760°F
Pressure: 2500 psig
LHSV: 1.44
GHSV: 354

lbs/hr.

	STREAM DESCRIPTION	Feed Gas	Lignite	Feed Solvent	Deashing Solvent	Total In
		Sx-21	Sx-1	Sx-14	Sx-40	
Quantitative	Hydrogen - H ₂	1.41				1.41
	Carbon Monoxide - CO	17.56				17.56
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂	0.11				0.11
	Ammonia - NH ₃					
	Light Oil - (0-100°C @ 1.6 Torr)			18.97	93.68	112.65
	Solvent - (100-230°C @ 1.6 Torr)			70.59	1.52	72.11
	Heavy Oil - (230-255°C @ 1.6 Torr)			3.06		3.06
	SRL					
	Coal (MAF)		27.12			27.12
	Ash		2.75			2.75
	Water		16.08	0.14		16.22
	Phenol					
	TOTAL	19.08	45.95	92.76	95.20	252.99

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Rate Adjusted
Revised: 5/5/78

Run No: M-35A
Date: 1/24/78
Time: 0700-1900

MATERIAL OUT

Temperature: 760°F
Pressure: 2500 psig
LHSV: 1.44
GHSV: 354

lbs/hr.

	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
Quantitative	Hydrogen - H ₂	1.18	0.09				0.03
	Carbon Monoxide - CO	12.93	1.09				0.12
	Carbon Dioxide - CO ₂	7.28	2.61				0.59
	Hydrogen Sulfide - H ₂ S	0.07	0.03				
	Methane - CH ₄	0.59	0.14				0.03
	Ethane - C ₂ H ₆	0.31	0.12				0.01
	Propane - C ₃ H ₈	0.14	0.11				0.05
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.12	0.02				
	Ammonia - NH ₃			0.02			
	Light Oil - (0-100°C @ 1.6 Torr)			0.14		1.94	
	Solvent - (100-230°C @ 1.6 Torr)			0.07		0.01	
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Coal (MAF)						
	Ash						
	Water			16.27			
	Phenol						
	TOTAL	22.62	4.21	16.50	Negl.	1.95	0.83

Elemental	Carbon						
	Hydrogen						
	Nitrogen						
	Sulfur						
	Oxygen (by diff)						
	Ash						
	TOTAL						

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Rate Adjusted
Revised: 5/5/78

Run No: M-35A
Date: 1/24/78
Time: 0700-1900

MATERIAL OUT

Temperature: 760°F
Pressure: 2500 psig
LHSV: 1.44
GHSV: 354

lbs/hr.

Quantitative	STREAM DESCRIPTION	Cold Trap	V-8 Btms	F-1 Btms	V-9 Ovhd	←Recycle S-7 Btms	Solvent→ LEC Btms
	SAMPLE POINT	Sx-33	Calc	Sx-2	Sx-28	Sx-39	Sx-12
	Hydrogen - H ₂						
	Carbon Monoxide - CO						
	Carbon Dioxide - CO ₂						
	Hydrogen Sulfide - H ₂ S						
	Methane - CH ₄						
	Ethane - C ₂ H ₆						
	Propane - C ₃ H ₈						
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂						
	Ammonia - NH ₃						
	Light Oil - (0-100°C @ 1.6 Torr)	0.05	2.40		83.57	15.80	4.56
	Solvent - (100-230°C @ 1.6 Torr)		2.00	9.59	1.02	60.74	2.69
	Heavy Oil - (230-255°C @ 1.6 Torr)					3.49	0.10
	SRL		4.58	9.99			
	Coal (MAF)		3.43				
	Ash		2.51	0.15			
	Water					0.21	
	Phenol						
	TOTAL	0.05	14.92	19.73	84.59	80.24	7.35

Elemental	Carbon						
	Hydrogen						
	Nitrogen						
	Sulfur						
	Oxygen (by diff)						
	Ash						
	TOTAL						

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Rate Adjusted
Revised: 5/5/78

Run No: M-35A
Date: 1/24/78
Time: 0700-1900

MATERIAL OUT

Temperature: 760°F
Pressure: 2500 psig
LHSV: 1.44
GHSV: 354

lbs/hr.

Quantitative	STREAM DESCRIPTION					Total Out
	SAMPLE POINT					
	Hydrogen - H ₂					1.30
	Carbon Monoxide - CO					14.14
	Carbon Dioxide - CO ₂					10.48
	Hydrogen Sulfide - H ₂ S					0.10
	Methane - CH ₄					0.76
	Ethane - C ₂ H ₆					0.44
	Propane - C ₃ H ₈					0.30
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.14
	Ammonia - NH ₃					0.02
	Light Oil - (0-100°C @ 1.6 Torr)					108.46
	Solvent - (100-230°C @ 1.6 Torr)					76.12
	Heavy Oil - (230-255°C @1.6 Torr)					3.59
	SRL					14.57
	Coal (MAF)					3.43
	Ash					2.66
	Water					16.48
	Phenol					
	TOTAL					252.99

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by dlff)					
	Ash					
	TOTAL					

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/5/78

Run No: M-35A

Date: 1/24/78

Time: 0700-1900

WEIGHT PERCENT

SAMPLE DESCRIPTION	Lignite	F-I Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
	Sx-1	Sx-2	Sx-12	Sx-14	Sx-16	Sx-16
SAMPLE POINT						
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)						
Light Oils - (0-100° C @ 1.6 Torr)			62.00	20.45	99.70	
Solvent - (100-230° C @ 1.6 Torr)		48.60	36.60	76.10	0.30	
Heavy Oil - (230-255° C @1.6 Torr)			1.40	3.30		
SRL		50.62				
Coal (MAF)	59.02	0.00				
Ash	5.98	0.78				
Water	35.00			0.15		100
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)		100				
Melting Point °F		140				
Specific Gravity 60/60			0.924	1.001		
Viscosity, cp						

ELEMENTAL	WEIGHT PERCENT					
	Lignite	F-I Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	2	2	2	2

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/5/78

Run No: M-35A

Date: 1/24/78

Time: 0700-1900

WEIGHT PERCENT

SAMPLE DESCRIPTION	S-8	Product	Feed	S-4B	S-4B	S-4B
	Total	Gas	Gas	Oil	Water	Total
SAMPLE POINT	Sx-16	Sx-19	Sx-21	*Sx-23	Sx-23	Sx-23
Hydrogen - H ₂		5.20	7.37			
Carbon Monoxide - CO		57.17	92.04			
Carbon Dioxide - CO ₂		32.19				
Hydrogen Sulfide - H ₂ S		0.29				
Methane - CH ₄		2.61				
Ethane - C ₂ H ₆		1.35				
Propane - C ₃ H ₈		0.62				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂		0.55	0.59			
Ammonia - NH ₃ (PPM)		0.02			0.10	0.10
Light Oils - (0-100°C @ 1.6 Torr)	99.70			68.12		0.88
Solvent - (100-230°C @ 1.6 Torr)	0.30			31.70		0.41
Heavy Oil - (230-255°C @1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water				0.18	99.90	98.61
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	-	2	2	2	2	-

*Insufficient oil sample. Assumed the same as the S-4A oil.

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/5/78

Run No: M-35A

Date: 1/24/78

Time: 0700-1900

WEIGHT PERCENT

	WEIGHT PERCENT					
	V-8 Btms	V-9 Ovhd	S-4A Ovhd	J-1 Discharge	Cold Trap	S-7 Btms
SAMPLE DESCRIPTION						
SAMPLE POINT	Sx-27	Sx-28	Sx-30	Sx-32	*Sx-33	Sx-34/39
Hydrogen - H ₂			2.14	3.69		
Carbon Monoxide - CO			25.97	14.22		
Carbon Dioxide - CO ₂			61.98	71.53		
Hydrogen Sulfide - H ₂ S			0.78			
Methane - CH ₄			3.22	2.99		
Ethane - C ₂ H ₆			2.79	1.45		
Propane - C ₃ H ₈			2.59	6.12		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂			0.43			
Ammonia - NH ₃ (PPM)			0.10			
Light Oils - (0-100° C @ 1.6 Torr)	37.97	98.80			99.79	19.69
Solvent - (100-230° C @ 1.6 Torr)	6.73	1.20				75.70
Heavy Oil - (230-255° C @ 1.6 Torr)	3.72					4.35
SRL	12.93					
Coal (MAF)	27.13					
Ash	11.52					
Water					0.21	0.26
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60						1.003
Viscosity, cp						

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					
	Number of Sample Avg.	2	2	2	-	4

*Insufficient sample for distillation. Assumed to be light oil and water.

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/5/78

Run No: M-35A

Date: 1/24/78

Time: 0700-1900

WEIGHT PERCENT

SAMPLE DESCRIPTION	S-4A Oil	S-4A Water	S-4A Total	S-7 Btms	Deashing Solvent	V-8 Btms
	Sx-37	Sx-37	Sx-37	Sx-34/39	Sx-40	*Calc .
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)		0.09				
Light Oils - (0-100°C @ 1.6 Torr)	68.12		67.28	19.69	98.40	16.09
Solvent - (100-230°C @ 1.6 Torr)	31.70		31.30	75.70	1.60	13.40
Heavy Oil - (230-255°C @1.6 Torr)				4.35		
SRL						30.70
Coal (MAF)						22.99
Ash						16.82
Water	0.18	99.91	1.42	0.26		
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60				1.003		
Viscosity, cp						

Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	-	4	2	-

*Composition calculated by making material balance around deashing unit.

MOLE PERCENT

Quantitative

Elemental-

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-35A
DATE: 1/24/78
TIME: 0700-1900

SAMPLE DESCRIPTION	F-1 Btms	LEC Btms	Feed Solv.	S-8 Oil	V-9 Ovhd
SAMPLE POINT	Sx-2	Sx-12	Sx-14	Sx-16	Sx-28
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5	5		
IBP		18.5	26.6		
5%		24.9	53.5		
10%		29.5	81.5		
20%		47	120		
30%		70	147		
40%		85	167		
50%		96.2	178.8		
60%		116	186.2		
70%		137	192.5		
80%		167	199.2		
90%		184.8	210.5		
95%		194	225.5		
DRY PT.		202.2	236.3		
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR				758	746
IBP				42	112
5%				62	113
10%				70	113
20%				80	113
30%				89	113
40%				96	113
50%				101	113
60%				106	113
70%				109	113
80%				112	114
90%				114	114
95%				116	116
DRY PT.				129	118
ASPHALTENE TEST					
WT % ASH	0.78				
WT % UNCONVERTED COAL	0.00				
WT % PRE-ASPHALTENES	17.90				
WT % ASPHALTENES	17.34				
WT % MALTENES & DIST. OIL	63.98				
TOTAL	100.00				
SAYBOLT VISCOSITY, SSU					
100° F		8.7	10.4		
210° F		7.5	8.4		
MISCELLANEOUS					
KF H ₂ O WT% (Raw Sx)			0.15		
INFRARED RATIO		0.20	0.22		
SP. GRAV. 60/60 F		0.924	1.001		

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

**PROJECT LIGNITE
SAMPLE ANALYSES**

RUN NO: M-35A

DATE: 1/24/78

TIME: 0700-1900

SAMPLE DESCRIPTION	V-9 Btms	S-7 Btms	S-4A Oil	Deashing Solv.	
SAMPLE POINT	Sx-29	Sx-34/39	Sx-37	Sx-40	
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR	5	5			
IBP	117	24.7			
5%	168	57.5			
10%	179	90.8			
20%	188	122.2			
30%	195	146.8			
40%	201	164.5			
50%	208	175.7			
60%	217.5	183.4			
70%	237	189.9			
80%	271	198.4			
90%		212.5			
95%		233.8			
DRY PT.		233.8			
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR			757	747	
IBP			69	112	
5%			104	113	
10%			119	113	
20%			134	113	
30%			153	113	
40%			193	113	
50%			214	113	
60%			242	113	
70%			277	113	
80%			307	114	
90%			340	114	
95%			350	114	
DRY PT.			350	118	
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F		10.5			
210° F		8.3			
MISCELLANEOUS					
KF H ₂ O WT%(Raw Sx)		0.26			
INFRARED RATIO		0.23			
SP. GRAV. 60/60 F		1.003			

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL

ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL

MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES

DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/5/78

MATERIAL OUT

Run No: M-35A
Date: 1/24/78
Time: 0700-1900

lbs/hr.

Temperature: 760°F
Pressure: 2500 psig
LHSV: 1.44
GHSV: 354

STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
Hydrogen - H ₂	1.18	0.09				0.03
Carbon Monoxide - CO	12.93	1.09				0.12
Carbon Dioxide - CO ₂	7.28	2.61				0.59
Hydrogen Sulfide - H ₂ S	0.07	0.03				
Methane - CH ₄	0.59	0.14				0.03
Ethane - C ₂ H ₆	0.31	0.12				0.01
Propane - C ₃ H ₈	0.14	0.11				0.05
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.12	0.02				
Ammonia - NH ₃			0.02			
Light Oil - (0-100°C @ 1.6 Torr)			0.14		1.94	
Solvent - (100-230°C @ 1.6 Torr)			0.07		0.01	
Heavy Oil - (230-255°C @1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water			16.27			
Phenol						
TOTAL	22.62	4.21	16.50	Negl.	1.95	0.83

ELEMENTAL						
	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)	Ash
TOTAL						

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/5/78

Run No: M-35A
Date: 1/24/78
Time: 0700-1900

MATERIAL OUT

Temperature: 760°F
Pressure: 2500 psig
LHSV : 1.44
GHSV: 354

lbs/hr.

	STREAM DESCRIPTION	Cold Trap	V-8 Btms	F-1 Btms	V-9 Ovhd	Recycle Solvent	
		Sx-33	Sx-27	Sx-2	Sx-28	S-7 Btms	LEC Btms
	SAMPLE POINT					Sx-34/39	Sx-12
	Hydrogen - H ₂						
	Carbon Monoxide - CO						
	Carbon Dioxide - CO ₂						
	Hydrogen Sulfide - H ₂ S						
	Methane - CH ₄						
	Ethane - C ₂ H ₆						
	Propane - C ₃ H ₈						
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂						
	Ammonia - NH ₃						
Quantitative	Light Oil - (0-100°C @ 1.6 Torr)	0.05	5.14		83.57	15.80	4.56
	Solvent - (100-230°C @ 1.6 Torr)		0.91	9.59	1.02	60.74	2.69
	Heavy Oil - (230-255°C @ 1.6 Torr)		0.50			3.49	0.10
	SRL		1.75	9.99			
	Cool (MAF)		3.67				
	Ash		1.56	0.15			
	Water					0.21	
	Phenol						
	TOTAL	0.05	13.53	19.73	84.59	80.24	7.35
Elemental	Carbon						
	Hydrogen						
	Nitrogen						
	Sulfur						
	Oxygen (by diff)						
	Ash						
	TOTAL						

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/5/78

Run No: M-35A
Date: 1/24/78
Time: 0700-1900

MATERIAL OUT

lbs/hr.

Temperature: 760°F
Pressure: 2500 psig
LHSV : 1.44
GHSV: 354

Quantitative	STREAM DESCRIPTION					Total Out
	SAMPLE POINT					
	Hydrogen - H ₂					1.30
	Carbon Monoxide - CO					14.14
	Carbon Dioxide - CO ₂					10.48
	Hydrogen Sulfide - H ₂ S					0.10
	Methane - CH ₄					0.76
	Ethane - C ₂ H ₆					0.44
	Propane - C ₃ H ₈					0.30
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.14
	Ammonia - NH ₃					0.02
	Light Oil - (0-100°C @ 1.6 Torr)					111.20
	Solvent - (100-230°C @ 1.6 Torr)					75.03
	Heavy Oil - (230-255°C @1.6 Torr)					4.09
	SRL					11.74
	Cool (MAF)					3.67
	Ash					1.71
	Water					16.48
	Phenol					
	TOTAL					251.60

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					

RUN CONDITIONS

RUN NO. M-35C

DATE MADE 1/27/78

TEMPERATURE °F

PREHEATER E-1
SAND BATH 756
INLET 216
OUTLET 730

DISSOLVERS

R-1A (1) _____
(2) _____
(3) _____
(4) _____
R-1B (1) 744
(2) 802
(3) 790
(4) 823

REACTOR PRODUCT SEPARATORS

S-1 539
S-2 55
S-3 468
S-4A 70
S-4B 71

PREHEATER E-11

DOWTHERM 664
INLET 425
OUTLET 633

VACUUM FLASH F-1

OVERHEAD VAPOR 519
UPPER WALL 607
LOWER WALL 569
DOWNCOMER 433
CONDENSATE
ACCUMULATOR, S-7 80

PREHEATER E-13

DOWTHERM 466
INLET 71
OUTLET 186

LIGHT ENDS COLUMN, F-2

REBOILER 448
LOWER SECTION 278
FEED SECTION 210
OVERHEAD 180
S-8 66
S-9 _____

SOLVENT COLUMN, F-3

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

HEAVY ENDS COLUMN, F-4

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

VACUUM FLASH, F-0

OVERHEAD NIU
BOTTOMS _____

PRECIPITATION TOWER, V-8

ZONE (1) NIU
(2) _____
(3) _____

RECOVERY TOWER V-9

REBOILER NIU
OVERHEAD _____

PRESSURE, psig

SEPARATORS S-1 and S-2
2500

SEPARATOR S-3
240

SEPARATORS S-4A and S-4B
60

VACUUM FLASH, F-1
14 Torr

LIGHT ENDS COLUMN, F-2
Atm

SOLVENT COLUMN, F-3
NIU

HEAVY ENDS COLUMN, F-4
NIU

VACUUM FLASH, F-0
NIU

SETTLING TOWER, V-8
NIU

RECOVERY TOWER, V-7
NIU

	Start	End	Run No: M-35C
Date of Run	1/27/78	1/27/78	Units: lbs/hr, °F, psig
Time of Run	0400	1600	Prep By: BCL & GGB
Length of Run	12	hrs.	Updated: 5/4/78
Hours on Coal at End of Y.P.	96.7		

MASS RATES AND RUN CONDITIONS

157.00 TOTAL

✕ OPEN

✕ CLOSED

TOTAL 157.00

Numbers in parentheses are not included in the totals.

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
Normalized
Revised: 5/4/78

Run No: M-35C
Date: 1/27/78
Time: 0400- 1600

SUMMARY

Temperature: 730°F
Pressure: 2500 psig
LHSV: 1.39
GHSV: 360

	lbs/hr.				
	STREAM DESCRIPTION	Total In	Total Out	Net Yield.	Wt % MAF Coal
Quantitative	SAMPLE POINT				
	Hydrogen - H ₂	1.43	1.11	-0.32	
	Carbon Monoxide - CO	17.91	11.03	-6.88	
	Carbon Dioxide - CO ₂		12.97	12.97	
	Hydrogen Sulfide - H ₂ S		0.19	0.19	
	Methane - CH ₄		1.20	1.20	8.26
	Ethane - C ₂ H ₆		0.56	0.56	
	Propane - C ₃ H ₈		0.44	0.44	
	Butane - C ₄ H ₁₀				
	Nitrogen - N ₂	0.11	0.18	0.07	18.46
	Ammonia - NH ₃		0.03	0.03	
	Light Oil - (0-100°C @ 1.6 Torr)	24.67	31.04	6.37	
	Solvent - (100-230°C @ 1.6 Torr)	61.73	59.18	-2.55	
	Heavy Oil - (230-255°C @ 1.6 Torr)	2.41	2.52	0.11	64.73
	SRL		14.53	14.53	
	Coal (MAF)	28.52	3.27	-25.25	
	Ash	2.74	2.29	-0.45	
	Water	17.48	16.46	-1.02	-3.58
	Phenol				
	TOTAL	157.00	157.00	-0-	

Elemental	Carbon				
	Hydrogen				
	Nitrogen				
	Sulfur				
	Oxygen (by diff)				
	Ash				
	TOTAL				

PROJECT LIGNITE
MEASURED & ADJUSTED MATERIAL BALANCE
Revised: 5/4/78

Run No: M-35C
Date: 1/27/78
Time: 0400-1600

MATERIAL IN

Temperature: 730°F
Pressure: 2500 psig
LHSV: 1.39
GHSV: 360

lbs/hr.

Quantitative	STREAM DESCRIPTION	Feed Gas	Lignite	Feed Solvent		Total In
	SAMPLE POINT	Sx-21	Sx-1	Sx-14		
	Hydrogen - H ₂	1.43				1.43
	Carbon Monoxide - CO	17.91				17.91
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂	0.11				0.11
	Ammonia - NH ₃					
	Light Oil - (0-100°C @ 1.6 Torr)			24.67		24.67
	Solvent - (100-230°C @ 1.6 Torr)			61.73		61.73
	Heavy Oil - (230-255°C @ 1.6 Torr)			2.41		2.41
	SRL					
	Cool (MAF)		28.52			28.52
	Ash		2.74			2.74
	Water		16.95	0.53		17.48
	Phenol					
	TOTAL	19.45	48.21	89.34		157.00

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

Normalized
Revised: 5/4/78

Temperature: 730°F
Pressure: 2500 psig
LHSV : 1.39
GHSV: 360

Run No: M-35C
Date: 1/27/78
Time: 0400-1600

MATERIAL OUT

lbs/hr.

Quantitative	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
	Hydrogen - H ₂	1.03	0.07				0.01
	Carbon Monoxide - CO	10.13	0.81				0.09
	Carbon Dioxide - CO ₂	9.17	3.03				0.77
	Hydrogen Sulfide - H ₂ S	0.12	0.07				
	Methane - CH ₄	1.02	0.15				0.03
	Ethane - C ₂ H ₆	0.39	0.13				0.04
	Propane - C ₃ H ₈	0.23	0.17				0.04
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.16	0.02				
	Ammonia - NH ₃			0.03			
	Light Oil - (0-100°C @ 1.6 Torr)			2.75		2.79	
	Solvent - (100-230°C @ 1.6 Torr)			0.87		0.30	
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Coal (MAF)						
	Ash						
	Water			15.50		0.18	
	Phenol						
	TOTAL	22.25	4.45	19.15	Negl.	3.27	0.98

Elemental	Carbon						
	Hydrogen						
	Nitrogen						
	Sulfur						
	Oxygen (by diff)						
	Ash						
	TOTAL						

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

Normalized
Revised: 5/4/78

Temperature: 730°F
Pressure: 2500 psig
LHSV: 1.39
GHSV: 360

Run No: M-35C
Date: 1/27/78
Time: 0400-1600

MATERIAL OUT

lbs/hr.

	STREAM DESCRIPTION	Cold Trap	F-1 Btms	←Recycle S-7 Btms	Solvent → LEC Btms	Total Out
	SAMPLE POINT	Sx-33	Sx-2	Sx-39	Sx-12	
	Hydrogen - H ₂					1.11
	Carbon Monoxide - CO					11.03
	Carbon Dioxide - CO ₂					12.97
	Hydrogen Sulfide - H ₂ S					0.19
	Methane - CH ₄					1.20
	Ethane - C ₂ H ₆					0.56
	Propane - C ₃ H ₈					0.44
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.18
	Ammonia - NH ₃					0.03
Quantitative	Light Oil - (0-100°C @ 1.6 Torr)	0.03		19.44	6.03	31.04
	Solvent - (100-230°C @ 1.6 Torr)	0.02	1.11	53.61	3.27	59.18
	Heavy Oil - (230-255°C @ 1.6 Torr)			2.40	0.12	2.52
	SRL		14.53			14.53
	Cool (MAF)		3.27			3.27
	Ash		2.29			2.29
	Water	0.03		0.75		16.46
	Phenol					
	TOTAL	0.08	21.20	76.20	9.42	157.00

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/4/78

Run No: M-35C

Date: 1/27/78

Time: 0400-1600

WEIGHT PERCENT

	WEIGHT PERCENT					
	Lignite	F-1 Brms	LEC Brms	Feed Solvent	S-8 Oil	S-8 Water
SAMPLE POINT	Sx-1	Sx-2	Sx-12	Sx-14	Sx-16	Sx-16
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)						
Light Oils - (0-100°C @ 1.6 Torr)			64.00	27.61	90.20	
Solvent - (100-230°C @ 1.6 Torr)		5.22	34.70	69.10	9.80	
Heavy Oil - (230-255°C @1.6 Torr)			1.30	2.70		
SRL		68.52				
Coal (MAF)	59.16	15.45				
Ash	5.68	10.81				
Water	35.16			0.59		100
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)		82.68				
Melting Point °F		341				
Specific Gravity 60/60			0.966	1.013		
Viscosity, cp						

	WEIGHT PERCENT					
	Lignite	F-1 Brms	LEC Brms	Feed Solvent	S-8 Oil	S-8 Water
Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	2	2	2	-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/4/78

Run No: M-35C

Date: 1/27/78

Time: 0400-1600

WEIGHT PERCENT

SAMPLE DESCRIPTION	S-8	Product	Feed	S-4B	S-4B	S-4B
	Total	Gas	Gas	Oil	Water	Total
SAMPLE POINT	Sx-16	Sx-19	Sx-21	Sx-23	Sx-23	Sx-23
Hydrogen - H ₂		4.61	7.32			
Carbon Monoxide - CO		45.51	92.10			
Carbon Dioxide - CO ₂		41.21				
Hydrogen Sulfide - H ₂ S		0.54				
Methane - CH ₄		4.60				
Ethane - C ₂ H ₆		1.74				
Propane - C ₃ H ₈		1.04				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂		0.74	0.58			
Ammonia - NH ₃ (PPM)		0.01			0.17	0.14
Light Oils - (0-100° C @ 1.6 Torr)	85.32			75.00		14.36
Solvent - (100-230° C @ 1.6 Torr)	9.27			23.80		4.56
Heavy Oil - (230-255° C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water	5.41			1.20	99.83	80.94
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

Quantitative

Elemental

Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	-	2	2	1	2	-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/4/78

Run No: M-35C

Date: 1/27/78

Time: 0400-1600

WEIGHT PERCENT

	WEIGHT PERCENT					
	S-4A Oyhd	J-1 Discharge	Cold Trap	S-7 Btms	S-4A Oil	S-4A Water
SAMPLE DESCRIPTION						
SAMPLE POINT	Sx-30	Sx-32	Sx-33	Sx-34/39	Sx-37	Sx-37
Hydrogen - H ₂	1.60	0.86				
Carbon Monoxide - CO	18.23	9.20				
Carbon Dioxide - CO ₂	68.02	78.41				
Hydrogen Sulfide - H ₂ S	1.59					
Methane - CH ₄	3.34	3.19				
Ethane - C ₂ H ₆	2.90	4.58				
Propane - C ₃ H ₈	3.80	3.76				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.43					
Ammonia - NH ₃ (PPM)	0.09					0.17
Light Oils - (0-100°C @ 1.6 Torr)			41.01	25.52	69.69	
Solvent - (100-230°C @ 1.6 Torr)			24.50	70.35	29.60	
Heavy Oil - (230-255°C @ 1.6 Torr)				3.15		
SRL						
Coal (MAF)						
Ash						
Water			34.49	0.98	0.71	99.93
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60				1.023		
Viscosity, cp						

Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	1	4	2	2

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/4/78

Run No: M-35C

Date: 1/27/78

Time: 0400-1600

WEIGHT PERCENT

Quantitative	SAMPLE DESCRIPTION	S-4A Total	S-7 Brms			
	SAMPLE POINT	Sx-37	Sx-34/39			
	Hydrogen - H ₂					
	Carbon Monoxide - CO					
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					
	Ammonia - NH ₃ (PPM)					
	Light Oils - (0-100°C @ 1.6 Torr)	69.20	25.52			
	Solvent - (100-230°C @ 1.6 Torr)	29.39	70.35			
	Heavy Oil - (230-255°C @ 1.6 Torr)		3.15			
	SRL					
	Coal (MAF)					
	Ash					
	Water	1.41	0.98			
	Phenol					
	TOTAL	100	100			
Elemental	Pyridine Solubles (Wt % Ash free)					
	Melting Point °F					
	Specific Gravity 60/60		1.023			
	Viscosity, cp					

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					
	Number of Sample Avg.	-	4			

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/4/78

Run No: M-35C

Date: 1/27/78

Time: 0400-1600

MOLE PERCENT

SAMPLE DESCRIPTION	Product Gas	Feed Gas	S-4A Ovhd	J-1 Discharge		
	Sx-19	Sx-21	Sx-30	Sx-32		
Hydrogen - H ₂	43.64	52.50	23.10	14.41		
Carbon Monoxide - CO	30.80	47.20	18.85	11.04		
Carbon Dioxide - CO ₂	17.75	-	44.75	59.86		
Hydrogen Sulfide - H ₂ S	0.30		1.35			
Methane - CH ₄	5.45		6.05	6.69		
Ethane - C ₂ H ₆	1.10		2.80	5.13		
Propane - C ₃ H ₈	0.45		2.50	2.87		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.50	0.30	0.45			
Ammonia - NH ₃ (PPM)	0.01		0.15			
Light Oils - (0-100° C @ 1.6 Torr)						
Solvent - (100-230° C @ 1.6 Torr)						
Heavy Oil - (230-255° C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water						
Phenol						
TOTAL	100	100	100	100		
Calc Mole Wt.	18.95	14.35	28.95	33.59		
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	2	2		

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-35C
DATE: 1/27/78
TIME: 0400-1600

SAMPLE DESCRIPTION	F-1 Btms	LEC Btms	Feed Solv.	S-8 Btms	S-4B Oil
SAMPLE POINT	Sx-2	Sx-12	Sx-14	Sx-16	Sx-23
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5	5		
IBP		27.4	25.2		
5%		62.8	79.2		
10%		69.2	89.3		
20%		77.5	107.2		
30%		85.0	128		
40%		93.6	150		
50%		103.2	169		
60%		115.2	178.2		
70%		133.2	188.2		
80%		160.0	200.6		
90%		182.4	231.3		
95%		195.2	260		
DRY PT.		203			
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR				758	740
IBP				57	62
5%				66	87
10%				72	125
20%				89	193
30%				102	203
40%				116	212
50%				131	227
60%				147	241
70%				167	261
80%				203	290
90%				286	334
95%					
DRY PT.				292	338
ASPHALTENE TEST					
WT % ASH	10.81				
WT % UNCONVERTED COAL	15.45				
WT % PRE-ASPHALTENES	37.73				
WT % ASPHALTENES	17.41				
WT % MALTENES & DIST. OIL	18.60				
TOTAL	100.00				
SAYBOLT VISCOSITY, SSU					
100° F		9.7	11.2		
210° F					
MISCELLANEOUS		8.0	8.5		
KF H ₂ O WT % (Raw Sx)			0.59		1.20
INFRARED RATIO			0.20		
SP. GRAV. 60/60 F		0.966	1.013		

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-35C
DATE: 1/27/78
TIME: 0400-1600

SAMPLE DESCRIPTION	Gold Trap Oil	S-7 Btms	S-4A Oil		
SAMPLE POINT	Sx-33	Sx-34/39	Sx-37		
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5			
IBP		22.8			
5%		72.9			
10%		92.2			
20%		109.9			
30%		132.1			
40%		153.4			
50%		170			
60%		179.9			
70%		188.2			
80%		199			
90%		226.1			
95%		260			
DRY PT.					
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR	744		755		
IBP	82		80		
5%	101		92		
10%	117		148		
20%	128		195		
30%	138		208		
40%	149		218		
50%	194		234		
60%	208		253		
70%	225		277		
80%	310		308		
90%			323		
95%					
DRY PT.	332		334		
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F		12.4			
210° F		8.5			
MISCELLANEOUS					
KF H ₂ O WT% (Raw Sx)	34.49	0.98	0.71		
INFRARED RATIO		0.26			
SP. GRAV. 60/60 F		1.023			

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/4/78

Run No: M-35C
Date: 1/27/78
Time: 0400-1600

MATERIAL OUT

Temperature: 730°F
Pressure: 2500 psig
LHSV : 1.39
GHSV: 360

	lbs/hr.					
	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
STREAM DESCRIPTION	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
SAMPLE POINT						
Hydrogen - H ₂	1.05	0.07				0.01
Carbon Monoxide - CO	10.32	0.83				0.09
Carbon Dioxide - CO ₂	9.34	3.09				0.78
Hydrogen Sulfide - H ₂ S	0.12	0.07				
Methane - CH ₄	1.04	0.15				0.03
Ethane - C ₂ H ₆	0.39	0.13				0.05
Propane - C ₃ H ₈	0.24	0.17				0.04
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.17	0.02				
Ammonia - NH ₃		0.01	0.03			
Light Oil - (0-100°C @ 1.6 Torr)			2.80		2.84	
Solvent - (100-230°C @ 1.6 Torr)			0.89		0.31	
Heavy Oil - (230-255°C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water			15.79		0.18	
Phenol						
TOTAL	22.67	4.54	19.51	Negl	3.33	1.00

	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)	Ash
Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/4/78

Run No: M-35C
Date: 1/27/78
Time: 0400-1600

MATERIAL OUT

Temperature: 730°F
Pressure: 2500 psig
LHSV: 1.39
GHSV: 360

lbs/hr.

	STREAM DESCRIPTION	Cold Trap	F-1 Btms	← Recycle Solvent →		Total Out
				S-7 Btms	LEC Btms	
	SAMPLE POINT	Sx-33	Sx-2	Sx-39	Sx-12	
	Hydrogen - H ₂					1.13
	Carbon Monoxide - CO					11.24
	Carbon Dioxide - CO ₂					13.21
	Hydrogen Sulfide - H ₂ S					0.19
	Methane - CH ₄					1.22
	Ethane - C ₂ H ₆					0.57
	Propane - C ₃ H ₈					0.45
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.19
	Ammonia - NH ₃					0.04
Quantitative	Light Oil - (0-100°C @ 1.6 Torr)	0.03		19.44	6.03	31.14
	Solvent - (100-230°C @ 1.6 Torr)	0.02	1.13	53.61	3.27	59.23
	Heavy Oil - (230-255°C @ 1.6 Torr)			2.40	0.12	2.52
	SRL		14.80			14.80
	Cool (MAF)		3.34			3.34
	Ash		2.33			2.33
	Water	0.03		0.75		16.75
	Phenol					
	TOTAL	0.08	21.60	76.20	9.42	158.35

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					

APPENDIX C

Process Development Unit Run M-36 February 6-10, 1978

Run M-36 was a replicate of Run M-35 using lignite mined near Velva, North Dakota. It was also the last of six runs made to determine differences in the liquefaction yields with lignites from various North Dakota locations.

Nominal run conditions were identical to those employed in the previous runs in which Larson and Gascoyne lignites were processed: 820-830°F maximum reaction temperature and 2500 psig dissolver pressure with liquid and gas hourly space velocities of 1.43 and 330, respectively. The solvent to raw coal ratio was approximately 1.8 to 1 and the charge gas was a nominal 50-50 mix of hydrogen and carbon monoxide. One reactor volume was used and all recoverable organic distillates boiling above about 300°F at one atmosphere were recycled. The product deashing unit was on stream and operated at about 450°F and 300 psig. A nominal four to one dilution of vacuum concentrated dissolver slurry products with toluene was employed. All product gases were measured, sampled and oxidized.

Operating problems experienced during Run M-36 were essentially confined to the vacuum flash solvent recovery equipment and the mineral residue settler. Throughout the run, a small leak persisted in the vacuum flash tower increasing its normal operating pressure from 15 to 25 or 30 Torr. Repeated efforts to locate the leak were unsuccessful and most of the run was made with the tower operating at the higher pressure. The second operating problem was plugging upstream of the overflow discharge valve on the ash precipitation tower. This problem has been encountered before and may result when the overflow stream carries over solids. Since most of the overflow flashes when depressured across the control valve, the solids are left to agglomerate and deposit both in the throat of the control valve and in the upstream tubing. Usually disassembly of the tubing reveals that starting at the control valve the upstream tubing is plugged with a gray, sandy, almost dry material.

In Run M-36, four yield periods were started but only three were completed. The second data period M-36B was terminated before completion when the overflow line of the solids settling tower plugged. The last two yield periods M-36C and M-36D were made without deashing.

Material balance closure was 97.8 percent in data period M-36A, 99.9 in 36C and 99.1 in 36D. Yield periods 36C and D were adjusted to 100 percent recovery by normalizing the outgoing product streams and in M-36C, where elemental quantities were known, a forced carbon balance was made by adjusting the oil to waste water ratio.

Yield period M-36A was adjusted to 100 percent recovery by increasing the V-8 bottoms rate and then calculating a composition from material balance data around the deashing unit. This was justified in part by a leaky pressure safety valve on the toluene recovery tower which allowed approximately 13 gallons of toluene to escape before it was detected and in part by 344 pounds of material which accumulated in the settling tower during 56 hours on stream. These adjustments resulted in a reasonably good carbon balance and no modification of the oil to waste water ratio was required.

The workups for the three completed yield periods including sample analyses, average run conditions and ASTM D86 and D1160 distillations are attached and a comparison of the three yield periods is made in Table 1. Temperature during the three M-36 yield periods varied slightly and the results after material balance adjustment are consistent with the temperatures. Thus, in general, the average net gas and liquid yields were respectively 29.5 and 62.0 weight percent of the MAF lignite charged. The distillable oil portion of the net liquid yield was relatively high at 24.3 percent while the SRL fraction was correspondingly low at 37.7 percent.

Comparing the results of Run M-36 with those of Run M-35, also made with Velva lignite, the overall net gas and liquid yields are nearly identical for the two runs as illustrated in Table 2. This is also true for the sum of net water and net ash and for conversion. The most striking difference lies in the distribution of the net liquid yield, particularly with the amounts of SRL generated. In Run M-35, the SRL yield typically amounted to 52 weight percent of the MAF lignite while in Run M-36 it was greatly less at 38 weight percent. Although significant, the disparity in SRL yields is perhaps more the result of variance in the distillation test than of the slight differences in run conditions. The analytical distillation of the F-1 vacuum bottoms at 5 Torr is subject to considerable variance because of the sizeable number of variables entering into the recovery of distillable oils from the product, including overheating, cracking, polymerization, vacuum control and vapor condensation and refluxing. The operating conditions and results of the five yield periods in the two runs were averaged. The averages should be representative of the yields to be expected when processing Velva lignite.

Run M-36 completed the series of runs made to evaluate the effects of lignite variety on PDU operations and liquefaction yields. The results of this evaluation are tabulated in Table 3 and shown graphically in Figures 1 and 2. In Table 3, seventeen yield periods of Runs L-1 and L-2 were averaged to provide a set of results typical of processing Zap lignite. For the Larson lignite, the five yield period of Runs M-31 and M-32 were averaged, and for the Gascoyne lignite, the six yield periods of Runs M-33 and M-34. For Velva lignite, the five yield periods of Runs M-35 and M-36 were averaged.

As shown in Figure 1, the highest conversion and net liquid and gas yields were obtained with Gascoyne lignite. Larson lignite was the least reactive of the lignites tested, generating both low conversion and low net liquid yields. Gas yields with Larson lignite were moderate and perhaps more a result of the water-gas shift reaction than of coal conversion. Dividing the net liquid yield into SRL and distillable oils as shown in Figure 2, graphs C & D, Larson lignite produces the highest yields of SRL and lowest yields of distillable oil while Velva lignite is just the reverse. Solvent recycle on a total oil basis is highest when processing Velva lignite and lowest when Larson lignite is used. Zap lignite produces results intermediate and near the average of the other three lignite varieties.

Liquefaction results for the four lignites studies can be correlated with their ash contents and consequently their contents of catalytic minerals. Gascoyne lignite, highest in ash of the four lignites tested, produced the highest overall MAF lignite conversion while Larson lignite, lowest of the four in ash, yielded the lowest conversion. A plot of MAF lignite conversion versus ash content of the four lignites is shown in Figure 3. It appears that conversion increases rapidly with ash content until approximately 6.3 percent ash after which conversion levels off and remains constant at 91.5 percent.

In summary, the results anticipated when processing North Dakota lignite at the operating conditions of 2500 psig pressure; 820-830°F reaction temperature; 1.4 LHSV; 320 GHSV; 3.2 solvent to MAF coal and 1.1 moles hydrogen to carbon monoxide would be as shown below:

Yields, Wt % MAF Coal		
Net Gas	30 to 36	
Net Liquids	54 to 65	
(Dist Oil)	(4 to 18)	
(SRL)	(44 to 51)	
Net Water and Ash	-3 to -9	
Unconverted Coal	9 to 20	
Solvent Recycle, %		
Potential (total oil basis)		101 to 106
Potential (Lt oil-free basis)		96 to 101
Reducing Gas Consumption		
SCF/lb Raw Coal		2.0 to 2.5
lbs (H ₂ Equiv)/100 lbs MAF Coal		1.6 to 2.4

In addition to the differences in net yields obtained with the different lignites, the quality of the various F-1 vacuum bottom products with regard to solvent retention is illustrated in Figures 4, 5, 6, 7 and 8. The distillable oil content of the F-1 vacuum bottoms, as determined by vacuum distillation at 5 Torr, is plotted versus the gradient bar melting point of the

sample. A family of lines is obtained for the F-1 vacuum bottoms of the various lignites with the product produced from Velva lignite having the greatest retention of distillable oils. Extrapolation of the data indicates the "dry point" or melting point temperature at which no distillable oils remain, is close to 425°F for the four vacuum bottom products. It also appears that the distillable oil content of the F-1 vacuum bottoms is not directly related to the ash or pyridine insolubles content of the sample and seems to be only a function of the gradient bar melting point.

Analyses of the various F-1 vacuum bottoms also included the determination of the particle density by kerosene displacement. Both deashed and undeashed samples were pulverized for this test and the results are shown below along with the results of bulk density determination. Undeashed samples contain greater than 10 percent ash and melt around 320°F. Deashed samples contain less than 2.5 percent ash and melt around 170°F.

Lignite Source of F-1 Vacuum Btms	Particle Density $\frac{\text{lbs}}{\text{ft}^3}$		Bulk Density $\frac{\text{lbs}}{\text{ft}^3}$	
	Undeashed	Deashed	Undeashed	Deashed
Larson	90.1	80.7	39.9	28.8
Gascoyne	97.7	80.1	40.4	33.2
Zap	91.7	87.1	37.5	29.0
Velva	90.7	78.9	36.5	32.9

At the end of Run M-36, the liquefaction reactor was cooled to about 550°F by flushing with solvent for approximately five hours. The reactor was then isolated and blown down into the reactor solids accumulator. Some 76 pounds of oily solids and 30 pounds of liquids were recovered. The solids were analyzed for ash and pyridine insolubles on both an "as received" and toluene-extracted basis. A routine sieve analysis of the toluene washed solids was performed and X-Ray fluorescence analysis carried out on the ash. An X-Ray diffraction analysis to determine identifiable minerals was also completed for the washed reactor solids. The results of these tests, along with some results of other tests on the Velva lignite and F-1 vacuum bottoms is presented below and on the following page.

Sample	Raw Reactor Solids	Toluene-Washed Reactor Solids
Wt % Pyridine Solubles	32.0	2.8
Wt % Pyridine Insolubles	25.6	41.4
Wt % Ash	42.4	55.8
Total	100.0	100.0

To make a broad summary of the results of the coal variety study it may be said that the operating problems encountered while processing the different lignites were not dissimilar to those found in the previous 30 or 50 runs made with Zap lignite. It might also be stated that the liquefaction yields obtained with the different lignites, although different, are not of sufficient magnitude to warrant plant redesign.

Process Development Unit Run M-36

Sieve Analysis of M-36 Feed coal and Toluene-washed Reactor Solids

U.S. Screen Sieve Mesh	Total Percentages			
	On		Passing	
	Lignite	Reactor Solids	Lignite	Reactor Solids
65	3.8	39.2	96.2	60.8
100	16.4	76.4	83.6	23.6
140	33.6	90.4	66.4	9.6
170	47.1	95.5	52.9	4.5
230	62.4	99.1	37.6	0.9
325	89.3	99.6	20.7	0.4
Pan	100.0	100.0	0.0	0.0

X-Ray Diffraction Pattern Analysis of Toluene Washed Reactor Solids

Calcite - most abundant, with some magnesium carbonate
 Feldspar - 1/3 as abundant as Calcite
 Quartz - 1/10 as abundant as Calcite

X-Ray Fluorescence
 Analysis of Ash, Wt %

Sample	Velva Lignite	Toluene Washed Reactor Solids	*M-36C & D F-1 Bottoms
Loss on Ignition @ 800°C	-	-	3.2
SiO ₂	28.4	15.1	20.1
Al ₂ O ₃	14.1	11.5	15.9
Fe ₂ O ₃	4.5	6.0	9.0
TiO ₂	0.5	0.3	0.4
P ₂ O ₅	0.9	0.6	0.1
CaO	31.2	49.5	29.2
MgO	9.1	8.8	12.6
Na ₂ O	1.8	1.4	0.6
K ₂ O	0.6	0.3	1.3
SO ₂	8.9	6.5	7.6
Total	100.0	100.0	100.0

*Determined by Atomic Absorption and Colorimetric Methods.

Table 1
Process Development Unit Run M-36

Yield Period	M-36A	M-36C	M-36D	Average
No. of Reactors	1	1	1	1
LHSV, Hr ⁻¹	1.35	1.36	1.34	1.35
GHSV, Hr ⁻¹	338	328	333	333
Solvent/MAF Coal Ratio	3.16	2.89	3.20	3.08
H ₂ /CO Mol Ratio	1.05	1.01	1.04	1.03
Temperatures, °F				
Preheater Outlet, E-1	743	736	750	743
Reactor 3 ft	722	719	753	731
Reactor 7 ft	786	792	799	792
Reactor 11 ft	765	768	809	781
Reactor 15 ft	816	820	829	822
Max. Reaction	816	820	829	822
Vacuum Flash Ovhd	512	530	530	524
Dissolver Pressure, psig	2500	2500	2500	2500
Solvent Recycle, %				
Actual	93.7	97.8	97.4	96.3
Potential	108.3	109.4	106.1	107.9
Yields, Wt % MAF Coal				
Net Gas	27.7	28.5	32.2	29.5
Net Liquid	66.1	62.8	57.2	62.0
(Dist. Oil)	(26.3)	(27.2)	(19.4)	(24.3)
(SRL)	(39.8)	(35.6)	(37.8)	(37.7)
Net H ₂ O and Ash	-3.4	-4.4	-1.1	-3.0
Unconverted Coal	9.6	13.1	11.7	11.5
Overall Material Balance, %	97.8	99.9	99.1	98.9

Table 2
Process Development Unit Runs M-35 and M-36

Run	M-35 Avg.	M-36 Avg.	Average
No of Y.P.s Averaged	2	3	5
No of Reactors	1	1	1
LHSV, Hr ⁻¹	1.42	1.35	1.38
GHSV, Hr ⁻¹	357	333	343
Solvent/MAF Coal Ratio	3.26	3.08	3.15
H ₂ /CO Mol Ratio	1.12	1.03	1.07
Temperatures, °F			
Preheater Outlet, E-1	745	743	744
Reactor 3 ft	750	731	739
Reactor 7 ft	798	792	794
Reactor 11 ft	796	781	787
Reactor 15 ft	822	822	822
Max. Reaction	822	822	822
Vacuum Flash Ovhd	519	524	522
Dissolver Pressure, psig	2500	2500	2500
Solvent Recycle, %			
Actual	95.1	96.3	95.8
Potential	102.4	107.9	105.7
Yields, Wt % MAF Coal			
Net Gas	30.4	29.5	29.9
Net Liquid	59.8	62.0	61.1
(Dist Oil)	(7.5)	(24.3)	(17.6)
(SRL)	(52.3)	(37.7)	(43.5)
Net H ₂ O and Ash	-2.3	-3.0	-2.7
Unconverted Coal	12.1	11.5	11.7
Overall Material Balance, %	100.2	98.9	99.4

Table 3

Process Development Unit Runs M-31, M-32, M-33, L-1, L-2, M-34, & M-36

Run	M-31 & 32	L-1 & 2	M-33 & 34	M-35 & 36
No of Y.P. Averaged	5	17	6	5
Type of Coal	Larson	Zap	Gascoyne	Velva
No of Reactors	1		1	1
LHSV, Hr ⁻¹	1.42	1.41	1.42	1.38
GHSV, Hr ⁻¹	319	330	310	343
Solvent/MAF Coal Ratio	2.94	3.03	3.90	3.15
Percent H ₂ in Feed Gas	52.2	49.1	51.5	51.4
Temperatures, °F				
Preheater Outlet, E-1	742	734	746	744
Reactor 3 ft	755	752	762	739
Reactor 7 ft	783	792	793	794
Reactor 11 ft	795	793	795	787
Reactor 15 ft	830	816	820	822
Max Reaction	830	816	820	822
Vacuum Flash Ovhd	513	508	520	522
Dissolver Pressure, psig	2500	2500	2500	2500
Solvent Recycle, %				
Actual	85.7	92.6	93.7	95.8
Potential	101.4	104.0	103.2	105.7
Potential (Lt Oil Free)	96.6	96.0	101.1	99.9
Yields, Wt % MAF Coal				
Net Gas	33.1	31.0	35.6	29.9
Net Liquid	54.7	63.4	64.4	61.1
(Dist Oil)	(4.1)	(13.0)	(15.1)	(17.6)
(SRL)	(50.6)	(50.4)	(49.3)	(43.5)
Net H ₂ O and Ash	-7.6	-4.5	-8.7	-2.7
Unconverted Coal	19.8	10.1	8.7	11.7
Overall Material Balance, %	99.1	98.9	99.0	99.4

FIGURE 1
Yields and Conversion With Four North Dakota Lignites

NET YIELDS AND CONVERSION ARE WEIGHT PERCENT OF MAF COAL CHARGED

FIGURE 2
Solvent Recycle and Product Distribution With Four North Dakota Lignites

FIGURE 3
MAF Lignite Conversion,%
Versus
Ash Content of Four Different North Dakota Lignites

FIGURE 4
Distillable Oil Content of F-I Bottoms
Versus
F-I Bottoms Melting Point, °F

FIGURE 5
Velva Lignite
Distillable Oil Content of F-I Bottoms
Versus
F-I Bottoms Melting Point, °F

FIGURE 6
Zap Lignite
Distillable Oil Content of F-I Bottoms
Versus
F-I Bottoms Melting Point, °F

FIGURE 7
Larson Lignite
Distillable Oil Content of F-I Bottoms
Versus
F-I Bottoms Melting Point, °F

FIGURE 8
 Gascoyne Lignite
 Distillable Oil Content of F-I Bottoms
 Versus
 F-I Bottoms Melting Point, °F

RUN CONDITIONS

RUN NO. M-36 A
DATE MADE 2/7-8/78

TEMPERATURE °F

PREHEATER E-I
SAND BATH 765
INLET 228
OUTLET 743

DISSOLVERS
R-1A (1) _____
(2) _____
(3) _____
(4) _____
R-1B (1) 722
(2) 786
(3) 765
(4) 816

REACTOR PRODUCT SEPARATORS
S-1 516
S-2 49
S-3 504
S-4A 71
S-4B 80

PREHEATER E-II
DOWTHERM 666
INLET 356
OUTLET 594

VACUUM FLASH F-1
OVERHEAD VAPOR 512
UPPER WALL 727
LOWER WALL 624
DOWNCOMER 420
CONDENSATE
ACCUMULATOR, S-7 90

PREHEATER E-13
DOWTHERM 426
INLET 81
OUTLET 208

LIGHT ENDS COLUMN, F-2
REBOILER 422
LOWER SECTION 301
FEED SECTION 257
OVERHEAD 213
S-8 80
S-9 -

SOLVENT COLUMN, F-3
REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

HEAVY ENDS COLUMN, F-4
REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

VACUUM FLASH, F-0
OVERHEAD 336
BOTTOMS 455

PRECIPITATION TOWER, V-8
ZONE (1) _____
(2) 278
(3) 453

RECOVERY TOWER V-9
REBOILER 502
OVERHEAD 301

PRESSURE, psig

SEPARATORS S-1 and S-2
2500

SEPARATOR S-3
243

SEPARATORS S-4A and S-4B
61

VACUUM FLASH, F-1
27 Torr

LIGHT ENDS COLUMN, F-2
Atm

SOLVENT COLUMN, F-3
NIU

HEAVY ENDS COLUMN, F-4
NIU

VACUUM FLASH, F-0
27 Torr

SETTLING TOWER, V-8
260

RECOVERY TOWER, V-7
2

	Start	End	Run No: M-36A
Date of Run	2/7/78	2/8/78	Units: lbs/hr, °F, psig
Time of Run	1700	0500	Prep. By: BCL & GGB
Length of Run	12	hrs.	Updated: 5/17/78
Hours on Coal at End of Y.P.	38.0		

MASS RATES AND RUN CONDITIONS

235.66 TOTAL

OPEN

CLOSED

TOTAL 235.66

Numbers in parentheses are not included in the totals.

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Adjusted
Revised: 5/17/78

Run No: M-36A
Date: 2/7-8/78
Time: 1700-0500

SUMMARY

lbs/hr.

Temperature: 743°F
Pressure: 2500 psig
LHSV: 1.35
GHSV: 338

Quantitative	STREAM DESCRIPTION	Total In	Total Out	Net Yield		WE % MAF Coal	
	SAMPLE POINT						
	Hydrogen - H ₂	1.30	1.11	-0.19			
	Carbon Monoxide - CO	17.36	12.41	-4.95			
	Carbon Dioxide - CO ₂		10.80	10.80			
	Hydrogen Sulfide - H ₂ S		0.13	0.13			
	Methane - CH ₄		0.86	0.86	7.36	27.68	
	Ethane - C ₂ H ₆		0.47	0.47			
	Propane - C ₃ H ₈		0.22	0.22			
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.14	0.13	-0.01			
	Ammonia - NH ₃		0.03	0.03			
	Light Oil - (0-100°C @ 1.6 Torr)	96.12	98.16	2.04			
	Solvent - (100-230°C @ 1.6 Torr)	70.92	77.23	6.31	17.58	66.11	
	Heavy Oil - (230-255°C @ 1.6 Torr)	3.79	2.45	-1.34			
	SRL		10.57	10.57			
	Coal (MAF)	26.59	2.56	-24.03		-90.37	
	Ash	2.74	2.16	-0.58		-2.18	
	Water	16.70	16.37	-0.33		-1.24	
	Phenol						
	TOTAL	235.66	235.66	-0-			

Elemental	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)	Ash	
	179.19	179.29	0.10				
	19.02	19.03	0.01				
	0.44	0.51	0.07				
	1.23	1.33	0.10				
	33.04	33.34	0.30				
	2.74	2.16	-0.58				
	TOTAL	235.66	235.66	-0-			

PROJECT LIGNITE
MEASURED & ADJUSTED MATERIAL BALANCE
Revised: 5/17/78

Run No: M-36A
Date: 2/7-8/78
Time: 1700-0500

MATERIAL IN

Temperature: 743°F
Pressure: 2500 psig
LHSV: 1.35
GHSV: 338

lbs/hr.

Quantitative	STREAM DESCRIPTION	Feed Gas	Lignite	Feed Solvent	Deashing Solvent	Total In
	SAMPLE POINT	Sx-21	Sx-1	Sx-14	Sx-40	
	Hydrogen - H ₂	1.30				1.30
	Carbon Monoxide - CO	17.36				17.36
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂	0.14				0.14
	Ammonia - NH ₃					
	Light Oil - (0-100°C @ 1.6 Torr)			10.82	85.30	96.12
	Solvent - (100-230°C @ 1.6 Torr)			69.49	1.43	70.92
	Heavy Oil - (230-255°C @ 1.6 Torr)			3.79		3.79
	SRL					
	Cool (MAF)		26.59			26.59
	Ash		2.74			2.74
	Water		16.57	0.13		16.70
	Phenol					
	TOTAL	18.80	45.90	84.23	86.73	235.66

Elemental						
	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)	Ash
	7.44	1.30	0.14		9.92	
	19.54	3.14	0.17	0.12	20.19	2.74
	73.09	7.04	0.13	1.09	2.88	
	79.12	7.54		0.02	0.05	
	179.19	19.02	0.44	1.23	33.04	2.74
	TOTAL	18.80	45.90	84.23	86.73	235.66

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

V-8 Btms Adjusted

Revised: 5/17/78

Temperature: 743°F

Pressure: 2500 psig

LHSV: 1.35

GHSV: 338

Run No: M-36A

Date: 2/7-8/78

Time: 1700-0500

MATERIAL OUT

lbs/hr.

STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
Hydrogen - H ₂	1.03	0.07				0.01
Carbon Monoxide - CO	11.43	0.85				0.13
Carbon Dioxide - CO ₂	8.07	2.06				0.67
Hydrogen Sulfide - H ₂ S	0.08	0.05				
Methane - CH ₄	0.74	0.10				0.02
Ethane - C ₂ H ₆	0.34	0.09				0.04
Propane - C ₃ H ₈	0.13	0.07				0.02
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.12	0.01				
Ammonia - NH ₃			0.03			
Light Oil - (0-100°C @ 1.6 Torr)			0.61		1.26	
Solvent - (100-230°C @ 1.6 Torr)			0.27		0.03	
Heavy Oil - (230-255°C @ 1.6 Torr)			0.05			
SRL						
Cool (MAF)						
Ash						
Water			15.98			
Phenol						
TOTAL	21.94	3.30	16.94	Neg1	1.29	0.89

Carbon	8.03	1.12	0.78		1.08	0.30
Hydrogen	1.32	0.12	1.87		0.13	0.03
Nitrogen	0.12	0.02	0.03			
Sulfur	0.07	0.05				
Oxygen (by diff)	12.40	1.99	14.26		0.08	0.56
Ash						
TOTAL	21.94	3.30	16.94	Neg1.	1.29	0.89

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

V-8 Btms Adjusted
Revised: 5/17/78

Temperature: 743°F
Pressure: 2500 psig
LHSV : 1.35
GHSV: 338

Run No: M-36A
Date: 2/7-8/78
Time: 1700-0500

MATERIAL OUT

lbs/hr.

STREAM DESCRIPTION	Cold Trap	V-8 Btms	F-1 Btms	V-9 Ovhd	Recycle S-7 Btms	Solvent LEC Btms
	Sx-33	Calc	Sx-2	Sx-28	Sx-39	Sx-12
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃						
Light Oil - (0-100°C @ 1.6 Torr)	0.05	5.33		79.02	8.48	3.41
Solvent - (100-230°C @ 1.6 Torr)	0.11	1.53	9.28	1.53	61.65	2.83
Heavy Oil - (230-255°C @ 1.6 Torr)					2.32	0.08
SRL			10.57			
Cool (MAF)		2.41	0.15			
Ash		1.98	0.18			
Water	0.27				0.12	
Phenol						
TOTAL	0.43	11.25	20.18	80.55	72.57	6.32

Carbon	0.11	7.96	17.51	73.47	63.50	5.43
Hydrogen	0.05	0.71	1.29	7.01	5.86	0.64
Nitrogen		0.02	0.15		0.16	0.01
Sulfur		0.03	0.20	0.02	0.92	0.04
Oxygen (by diff)	0.27	0.55	0.85	0.05	2.13	0.20
Ash		1.98	0.18			
TOTAL	0.43	11.25	20.18	80.55	72.57	6.32

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Adjusted
Revised: 5/17/78

Run No: M-36A
Date: 2/7-8/78
Time: 1700-0500

MATERIAL OUT
lbs/hr.

Temperature: 743°F
Pressure: 2500 psig
LHSV: 1.35
GHSV: 338

Quantitative	STREAM DESCRIPTION					Total Out
	SAMPLE POINT					
	Hydrogen - H ₂					1.11
	Carbon Monoxide - CO					12.41
	Carbon Dioxide - CO ₂					10.80
	Hydrogen Sulfide - H ₂ S					0.13
	Methane - CH ₄					0.86
	Ethane - C ₂ H ₆					0.47
	Propane - C ₃ H ₈					0.22
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.13
	Ammonia - NH ₃					0.03
	Light Oil - (0-100°C @ 1.6 Torr)					98.16
	Solvent - (100-230°C @ 1.6 Torr)					77.23
	Heavy Oil - (230-255°C @ 1.6 Torr)					2.45
	SRL					10.57
	Cool (MAF)					2.56
	Ash					2.16
	Water					16.37
	Phenol					
	TOTAL					235.66

Elemental	Carbon					179.29
	Hydrogen					19.03
	Nitrogen					0.51
	Sulfur					1.33
	Oxygen (by diff)					33.34
	Ash					2.16
	TOTAL					235.66

Time: 1700-0500

WEIGHT PERCENT

Quantitative

Elemental

-125-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/17/78

Run No: M-36A

Date: 2/7-8/78

Time: 1700-0500

WEIGHT PERCENT

SAMPLE DESCRIPTION	WEIGHT PERCENT					
	S-8 Total	Product Gas	Feed Gas	S-4B Oil	S-4B Water	S-4B Total
SAMPLE POINT	Sx-16	Sx-19	Sx-21	*Sx-23	Sx-23	Sx-23
Hydrogen - H ₂		4.72	6.91			
Carbon Monoxide - CO		52.08	92.33			
Carbon Dioxide - CO ₂		36.79				
Hydrogen Sulfide - H ₂ S		0.36				
Methane - CH ₄		3.39				
Ethane - C ₂ H ₆		1.53				
Propane - C ₃ H ₈		0.59				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂		0.53	0.76			
Ammonia - NH ₃ (PPM)		0.01			0.18	0.17
Light Oils - (0-100° C @ 1.6 Torr)	97.50			65.25		3.59
Solvent - (100-230° C @ 1.6 Torr)	2.50			28.80		1.58
Heavy Oil - (230-255° C @ 1.6 Torr)				5.60		0.31
SRL						
Coal (MAF)						
Ash						
Water				0.35	99.82	94.35
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60				0.921		
Viscosity, cp						

Carbon	83.75	36.59	39.57	84.02		4.62
Hydrogen	10.39	6.01	6.91	9.68	11.12	11.04
Nitrogen	0.08	0.54	0.76	0.17	0.15	0.15
Sulfur	0.04	0.34		0.55		0.03
Oxygen (by diff)	5.74	56.52	52.76	5.58	88.73	84.16
Ash						
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	-	2	2	2	2	-

*Insufficient oil sample. Assumed to be the same as the S-4A oil.

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/17/78

Run No: M-36A

Date: 2/7-8/78

Time: 1700-0500

WEIGHT PERCENT

	WEIGHT PERCENT					
	V-8 Btms	V-9 Ovhd	S-4A Ovhd	J-1 Discharge	Cold Trap	S-7 Btms
SAMPLE DESCRIPTION						
SAMPLE POINT	Sx-27	Sx-28	Sx-30	Sx-32	Sx-33	Sx-34/39
Hydrogen - H ₂			2.02	1.42		
Carbon Monoxide - CO			25.87	14.84		
Carbon Dioxide - CO ₂			62.42	75.27		
Hydrogen Sulfide - H ₂ S			1.59			
Methane - CH ₄			2.87	2.67		
Ethane - C ₂ H ₆			2.69	3.85		
Propane - C ₃ H ₈			1.97	1.95		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂			0.42			
Ammonia - NH ₃ (PPM)			0.15			
Light Oils - (0-100° C @ 1.6 Torr)	67.47	98.10			12.19	11.69
Solvent - (100-230° C @ 1.6 Torr)	3.23	1.90			26.40	84.95
Heavy Oil - (230-255° C @ 1.6 Torr)						3.20
SRL	4.36					
Coal (MAF)	19.01					
Ash	5.93					
Water					61.41	0.16
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)	79.79					
Melting Point °F						
Specific Gravity 60/60						1.026
Viscosity, cp						

Elemental	Carbon	84.33	91.21	34.02	33.57	24.82	87.50
	Hydrogen	7.17	8.70	3.76	3.21	11.14	8.08
	Nitrogen	0.24	-	0.54		0.02	0.22
	Sulfur	0.30	0.02	1.50		0.17	1.27
	Oxygen (by diff)	2.03	0.07	60.18	63.22	63.85	2.93
	Ash	5.93					
	TOTAL	100	100	100	100	100	100
	Number of Sample Avg.	2	2	2	2	2	4

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/17/78

Run No: M-36A

Date: 2/7-8/78

Time: 1700-0500

WEIGHT PERCENT

	Quantitative					
	S-4A Oil	S-4A Water	S-4A Total	S-7 Btms	Deashing Solvent	V-8 Btms
SAMPLE DESCRIPTION	Sx-37	Sx-37	Sx-37	Sx-34/39	Sx-40	Calc.
SAMPLE POINT						
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)		0.18				
Light Oils - (0-100°C @ 1.6 Torr)	65.25		65.25	11.69	98.35	47.38
Solvent - (100-230°C @ 1.6 Torr)	28.80		28.80	84.95	1.65	13.60
Heavy Oil - (230-255°C @1.6 Torr)	5.60		5.60	3.20		
SRL						
Coal (MAF)						21.42
Ash						17.60
Water	0.35	99.82	0.35	0.16		
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60	0.921			1.026		
Viscosity, cp						

	Elemental					
Carbon	84.02		84.02	87.50	91.22	70.80
Hydrogen	9.68	11.12	9.68	8.08	8.70	6.31
Nitrogen	0.17	0.15	0.17	0.22	-	0.16
Sulfur	0.55		0.55	1.27	0.02	0.28
Oxygen (by diff)	5.58	88.73	5.58	2.93	0.06	4.85
Ash						17.60
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	2	2	-	4	2	-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/17/78

Run No: M-36A

Date: 2/7-8/78

Time: 1700-0500

MOLE PERCENT

SAMPLE DESCRIPTION	Product Gas	Feed Gas	S-4A Ovhd	J-1 Discharge		
	Sx-19	Sx-21	Sx-30	Sx-32		
Hydrogen - H ₂	43.99	50.95	26.99	21.53		
Carbon Monoxide - CO	34.70	48.65	24.74	16.12		
Carbon Dioxide - CO ₂	15.60		37.98	52.02		
Hydrogen Sulfide - H ₂ S	0.20		1.25			
Methane - CH ₄	3.95		4.80	5.08		
Ethane - C ₂ H ₆	0.95		2.40	3.90		
Propane - C ₃ H ₈	0.25		1.20	1.35		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.35	0.40	0.40			
Ammonia - NH ₃ (PPM)	0.01		0.24			
Light Oils - (0-100° C @ 1.6 Torr)						
Solvent - (100-230° C @ 1.6 Torr)						
Heavy Oil - (230-255° C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water						
Phenol						
TOTAL	100	100	100	100		
Calc Mole Wt.	18.65	14.75	26.77	30.41		
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	2	2		

Velva Lignite
Distillable Oil Content of F-I Bottoms
Versus
F-I Bottoms Melting Point, °F

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-36A
DATE: 2/7-8/78
TIME: 1700-0500

SAMPLE DESCRIPTION	F-1 Btms	LEC Btms	Feed Solv.	S-8 Oil	V-9 Ovhd
SAMPLE POINT	Sx-2	Sx-12	Sx-14	Sx-16	Sx-28
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5	5		
IBP		42.6	23.4		
5%		57.4	89.8		
10%		63.8	110.6		
20%		76	145.2		
30%		88	166.8		
40%		101	177.6		
50%		115.5	185.4		
60%		132.5	192		
70%		153.2	197		
80%		173.4	207.6		
90%		190	245.5		
95%		200.3	260		
DRY PT.		206.6			
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR				743	729
IBP				42	109
5%				68	111
10%				81	111
20%				93	112
30%				101	112
40%				106	112
50%				110	112
60%				112	113
70%				114	113
80%				116	113
90%				118	114
95%				121	118
DRY PT.				130	118
ASPHALTENE TEST					
WT % ASH	0.88				
WT % UNCONVERTED COAL	0.72				
WT % PRE-ASPHALTENES	14.28				
WT % ASPHALTENES	22.02				
WT % MALTENES & DIST. OIL	62.10				
TOTAL	100.00				
SAYBOLT VISCOSITY, SSU					
100° F		9.4	12.1		
210° F		7.7	8.6		
MISCELLANEOUS					
KF H ₂ O WT% (Raw Sx)		-	0.15		
INFRARED RATIO		0.21	0.23		
SP. GRAV. 60/60 F		0.953	1.020		

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-36A
DATE: 2/7-8/78
TIME: 1700-0500

SAMPLE DESCRIPTION	V-9 Btms	Cold Trap Oil	S-7 Btms	S-4A Oil	Deashing Solv.
SAMPLE POINT	Sx-29	Sx-33	Sx-34/39	Sx-37	Sx-40
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR	5		5		
IBP	114.5		25.7		
5%	175		99		
10%	184.8		117.6		
20%	191.2		147.6		
30%	199		168.3		
40%	206.2		180.6		
50%	216		187.3		
60%	231.5		192.8		
70%	260		199.1		
80%	275		207.6		
90%			234		
95%			258.4		
DRY PT.					
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR		744		741	734
IBP		88		66	109
5%		110		107	111
10%		112		128	111
20%		113		147	112
30%		114		188	112
40%		116		209	112
50%		118		230	113
60%		121		257	113
70%		137		284	113
80%				320	114
90%				346	114
95%					117
DRY PT.		232		350	120
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F			12.7		
210° F			8.6		
MISCELLANEOUS					
KF H ₂ O WT% (Row Sx)		61.41	0.16	0.35	
INFRARED RATIO			0.24		
SP. GRAV. 60/60 F			1.026	0.921	

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/17/78

Run No: M-36A
Date: 2/7-8/78
Time: 1700-0500

MATERIAL OUT

Temperature: 743°F
Pressure: 2500 psig
LHSV: 1.35
GHSV: 338

lbs/hr.

STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
Hydrogen - H ₂	1.03	0.07				0.01
Carbon Monoxide - CO	11.43	0.85				0.13
Carbon Dioxide - CO ₂	8.07	2.06				0.67
Hydrogen Sulfide - H ₂ S	0.08	0.05				
Methane - CH ₄	0.74	0.10				0.02
Ethane - C ₂ H ₆	0.34	0.09				0.04
Propane - C ₃ H ₈	0.13	0.07				0.02
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.12	0.01				
Ammonia - NH ₃			0.03			
Light Oil - (0-100°C @ 1.6 Torr)			0.61		1.26	
Solvent - (100-230°C @ 1.6 Torr)			0.27		0.03	
Heavy Oil - (230-255°C @ 1.6 Torr)			0.05			
SRL						
Cool (MAF)						
Ash						
Water			15.98			
Phenol						
TOTAL	21.94	3.30	16.94	Negl.	1.29	0.89

Carbon	8.03	1.12	0.78		1.08	0.30
Hydrogen	1.32	0.12	1.87		0.13	0.03
Nitrogen	0.12	0.02	0.03			
Sulfur	0.07	0.05				
Oxygen (by diff)	12.40	1.99	14.26		0.08	0.56
Ash						
TOTAL	21.94	3.30	16.94	Negl.	1.29	0.89

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/17/78

Run No: M-36A
Date: 2/7-8/78
Time: 1700-0500

MATERIAL OUT

Temperature: 743°F
Pressure: 2500 psig
LHSV: 1.35
GHSV: 338

lbs/hr.

	STREAM DESCRIPTION	Cold Trap	V-8 Btms	F-1 Btms	V-9 Ovhd	Recycle S-7 Btms	Solvent-LEC Btms
		Sx-33	Sx-27	Sx-2	Sx-28	Sx-39	Sx-12
	Hydrogen - H ₂						
	Carbon Monoxide - CO						
	Carbon Dioxide - CO ₂						
	Hydrogen Sulfide - H ₂ S						
	Methane - CH ₄						
	Ethane - C ₂ H ₆						
	Propane - C ₃ H ₈						
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂						
	Ammonia - NH ₃						
Quantitative	Light Oil - (0-100°C @ 1.6 Torr)	0.05	4.02		79.02	8.48	3.41
	Solvent - (100-230°C @ 1.6 Torr)	0.11	0.19	9.28	1.53	61.65	2.83
	Heavy Oil - (230-255°C @ 1.6 Torr)					2.32	0.08
	SRL		0.26	10.57			
	Coal (MAF)		1.13	0.15			
	Ash		0.35	0.18			
	Water	0.27				0.12	
	Phenol						
	TOTAL	0.43	5.95	20.18	80.55	72.57	6.32
Elemental	Carbon	0.11	5.02	17.51	73.47	63.50	5.43
	Hydrogen	0.05	0.43	1.29	7.01	5.86	0.64
	Nitrogen		0.01	0.15		0.16	0.01
	Sulfur		0.02	0.20	0.02	0.92	0.04
	Oxygen (by diff)	0.27	0.12	0.85	0.05	2.13	0.20
	Ash		0.35	0.18			
	TOTAL	0.43	5.95	20.18	80.55	72.57	6.32

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/17/78

Run No: M-36A
Date: 2/7-8/78
Time: 1700-0500

MATERIAL OUT

Temperature: 743°F
Pressure: 2500 psig
LHSV: 1.35
GHSV: 338

lbs/hr.

Quantitative	STREAM DESCRIPTION					Total Out
	SAMPLE POINT					
	Hydrogen - H ₂					1.11
	Carbon Monoxide - CO					12.41
	Carbon Dioxide - CO ₂					10.80
	Hydrogen Sulfide - H ₂ S					0.13
	Methane - CH ₄					0.86
	Ethane - C ₂ H ₆					0.47
	Propane - C ₃ H ₈					0.22
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.13
	Ammonia - NH ₃					0.03
	Light Oil - (0-100°C @ 1.6 Torr)					96.85
	Solvent - (100-230°C @ 1.6 Torr)					75.89
	Heavy Oil - (230-255°C @1.6 Torr)					2.45
	SRL					10.83
	Cool (MAF)					1.28
	Ash					0.53
	Water					16.37
	Phenol					
	TOTAL					230.36

Elemental	Carbon					176.35
	Hydrogen					18.75
	Nitrogen					0.50
	Sulfur					1.32
	Oxygen (by diff)					32.91
	Ash					0.53
	TOTAL					230.36

RUN CONDITIONS

RUN NO. M-36C

DATE MADE 2/9/78

TEMPERATURE °F

PREHEATER E-I
SAND BATH 777
INLET 215
OUTLET 736

DISSOLVERS

R-1A (1) _____
(2) _____
(3) _____
(4) _____
R-1B (1) 719
(2) 792
(3) 768
(4) 820

REACTOR PRODUCT SEPARATORS

S-1 521
S-2 55
S-3 495
S-4A 71
S-4B 77

PREHEATER E-II

DOWTHERM 678
INLET 450
OUTLET 639

VACUUM FLASH F-1

OVERHEAD VAPOR 530
UPPER WALL 635
LOWER WALL 610
DOWNCOMER 433

CONDENSATE

ACCUMULATOR, S-7 85

PREHEATER E-13
DOWTHERM 425
INLET 76
OUTLET 189

LIGHT ENDS COLUMN, F-2

REBOILER 401
LOWER SECTION 296
FEED SECTION 246
OVERHEAD 192
S-8 75
S-9 _____

SOLVENT COLUMN, F-3

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

HEAVY ENDS COLUMN, F-4

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

VACUUM FLASH, F-0

OVERHEAD NIU
BOTTOMS _____

PRECIPITATION TOWER, V-8

ZONE (1) NIU
(2) _____
(3) _____

RECOVERY TOWER V-9

REBOILER NIU
OVERHEAD _____

PRESSURE, psig

SEPARATORS S-1 and S-2
2500

SEPARATOR S-3
227

SEPARATORS S-4A and S-4B
61

VACUUM FLASH, F-1
18 Torr

LIGHT ENDS COLUMN, F-2
Atm

SOLVENT COLUMN, F-3
NIU

HEAVY ENDS COLUMN, F-4
NIU

VACUUM FLASH, F-0
NIU

SETTLING TOWER, V-8
NIU

RECOVERY TOWER, V-7
NIU

	Start	End	Run No: M-36C
Date of Run	2/9/78	2/9/78	Units: lbs/hr, °F, Psig
Time of Run	0700	1900	Prep. By: BCL & GGB
Length of Run	12	hours	Updated: 5/8/78
Hours on Coal at End of Y.P.	76.0		

MASS RATES AND RUN CONDITIONS

152.29 TOTAL

OPEN

CLOSED

TOTAL 152.29

Numbers in parentheses are not included in the totals.

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
Normalized with forced carbon balance

Revised: 5/8/78

Run No: M-36C
Date: 2/9/78
Time: 0700-1900

SUMMARY

Temperature: 736°F
Pressure: 2500 Psig
LHSV: 1.36
GHSV: 328

	STREAM DESCRIPTION	Total In	Total Out	Net Yield		WT% MAF Coal	
	SAMPLE POINT						
Quantitative	Hydrogen - H ₂	1.24	1.08	-0.16			
	Carbon Monoxide - CO	17.07	11.93	-5.14			
	Carbon Dioxide - CO ₂		11.68	11.68			
	Hydrogen Sulfide - H ₂ S		0.15	0.15			
	Methane - CH ₄		0.92	0.92	8.25	28.51	
	Ethane - C ₂ H ₆		0.50	0.50			
	Propane - C ₃ H ₈		0.38	0.38			
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.21	0.12	-0.09			
	Ammonia - NH ₃		0.01	0.01			
	Light Oil - (0-100°C @ 1.6 Torr)	18.75	23.53	4.78			
	Solvent - (100-230°C @ 1.6 Torr)	63.11	66.23	3.12	18.18	62.82	
	Heavy Oil - (230-255°C @1.6 Torr)	1.76	1.72	-0.04			
	SRL		10.32	10.32			
	Cool (MAF)	28.94	3.78	-25.16		-86.94	
	Ash	2.99	2.30	-0.69		-2.39	
	Water	18.22	17.64	-0.58		-2.00	
	Phenol						
	TOTAL	152.29	152.29	-0-			
Elemental	Carbon	99.88	99.88	-0-			
	Hydrogen	11.54	11.67	0.13			
	Nitrogen	0.59	0.56	-0.03			
	Sulfur	1.03	1.15	0.12			
	Oxygen (by diff)	36.26	36.73	0.47			
	Ash	2.99	2.30	-0.69			
	TOTAL	152.29	152.29	-0-			

PROJECT LIGNITE
MEASURED & ADJUSTED MATERIAL BALANCE

Revised: 5/8/78

Temperature: 736°F
Pressure: 2500 Psig
LHSV 1.36
GHSV: 328

Run No: M-36C
Date: 2/9/78
Time: 0700-1900

MATERIAL IN

lbs/hr.

Quantitative	STREAM DESCRIPTION	Feed Gas	Lignite	Feed Solvent		Total In
	SAMPLE POINT	Sx-21	Sx-1	Sx-14		
	Hydrogen - H ₂	1.24				1.24
	Carbon Monoxide - CO	17.07				17.07
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂	0.21				0.21
	Ammonia - NH ₃					
	Light Oil - (0-100°C @ 1.6 Torr)			18.75		18.75
	Solvent - (100-230°C @ 1.6 Torr)			63.11		63.11
	Heavy Oil - (230-255°C @ 1.6 Torr)			1.76		1.76
	SRL					
	Coal (MAF)		28.94			28.94
	Ash		2.99			2.99
	Water		18.03	0.19		18.22
	Phenol					
	TOTAL	18.52	49.96	83.81		152.29

Elemental	Carbon	7.31	21.26	71.31		99.88
	Hydrogen	1.24	3.42	6.88		11.54
	Nitrogen	0.21	0.19	0.19		0.59
	Sulfur		0.13	0.90		1.03
	Oxygen (by diff)	9.76	21.97	4.53		36.26
	Ash		2.99			2.99
	TOTAL	18.52	49.96	83.81		152.29

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
Normalized with forced carbon balance

Revised 5/8/78

Run No: M-36C
Date: 2/9/78
Time: 0700-1900

MATERIAL OUT
lbs/hr.

Temperature: 736°F
Pressure: 2500 Psig
LHSV: 1.36
GHSV: 328

Quantitative	STREAM DESCRIPTION	Cold Trap	F-1 Btms	Recycle Solvent S-7 Btms	LEC Btms	Total Out
	SAMPLE POINT	Sx-33	Sx-2	Sx-39	Sx-12	
	Hydrogen - H ₂					1.08
	Carbon Monoxide - CO					11.93
	Carbon Dioxide - CO ₂					11.68
	Hydrogen Sulfide - H ₂ S					0.15
	Methane - CH ₄					0.92
	Ethane - C ₂ H ₆					0.50
	Propane - C ₃ H ₈					0.38
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.12
	Ammonia - NH ₃					0.01
	Light Oil - (0-100°C @ 1.6 Torr)	0.04		13.11	6.14	23.53
	Solvent - (100-230°C @ 1.6 Torr)		4.32	56.97	3.48	66.23
	Heavy Oil - (230-255°C @1.6 Torr)			1.59	0.13	1.72
	SRL		10.32			10.32
	Coal (MAF)		3.78			3.78
	Ash		2.30			2.30
	Water	0.27		0.53		17.64
	Phenol					
	TOTAL	0.31	20.72	72.20	9.75	152.29

Elemental	Carbon	0.04	15.65	61.50	8.23	99.88
	Hydrogen	0.03	1.03	5.78	0.94	11.67
	Nitrogen		0.21	0.17	0.02	0.56
	Sulfur		0.11	0.85	0.04	1.15
	Oxygen (by diff)	0.24	1.42	3.90	0.52	36.73
	Ash		2.30			2.30
	TOTAL	0.31	20.72	72.20	9.75	152.29

Updated: 5/8/78

Date: 2/9/78

Time: 0700-1900

WEIGHT PERCENT

Quantitative

Elemental

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/8/78

Run No: M-36C

Date: 2/9/78

Time: 0700-1900

WEIGHT PERCENT

SAMPLE DESCRIPTION	S-8 Total	Product Gas	Feed Gas	S-4B Oil	S-4B Water	S-4B Total
	Sx-16	Sx-19	Sx-21	*Sx-23	Sx-23	Sx-23
Hydrogen - H ₂		4.56	6.68			
Carbon Monoxide - CO		50.21	92.20			
Carbon Dioxide - CO ₂		38.05				
Hydrogen Sulfide - H ₂ S		0.45				
Methane - CH ₄		3.68				
Ethane - C ₂ H ₆		1.58				
Propane - C ₃ H ₈		0.93				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂		0.52	1.12			
Ammonia - NH ₃ (PPM)		0.02			0.08	0.07
Light Oils - (0-100°C @ 1.6 Torr)	73.95			70.20		10.50
Solvent - (100-230°C @ 1.6 Torr)	1.05			29.00		4.34
Heavy Oil - (230-255°C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water	25.00			0.80	99.92	85.09
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60				0.940		
Viscosity, cp						

ELEMENTAL	WEIGHT PERCENT					
	S-8 Total	Product Gas	Feed Gas	S-4B Oil	S-4B Water	S-4B Total
Carbon	62.81	36.68	39.51	82.90		12.40
Hydrogen	10.52	6.00	6.68	9.31	11.11	10.84
Nitrogen	0.05	0.54	1.12	0.19	0.07	0.09
Sulfur	0.03	0.42		0.49		0.07
Oxygen (by diff)	26.59	56.36	52.69	7.11	88.82	76.60
Ash						
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	--	2	2	2	2	--

*Insufficient oil sample. Assumed to be the same as the S-4A oil.

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/8/78

Run No: M-36C

Date: 2/9/78

Time: 0700-1900

WEIGHT PERCENT

SAMPLE DESCRIPTION	S-4A Ovhd	J-1 Disch	Cold Trap	S-7 Btms	S-4A Oil	S-4A Water
	Sx-30	Sx-32	Sx-33	Sx-34/39	Sx-37	Sx-37
Hydrogen - H ₂	1.61	0.90				
Carbon Monoxide - CO	20.18	10.79				
Carbon Dioxide - CO ₂	68.64	73.76				
Hydrogen Sulfide - H ₂ S	1.11					
Methane - CH ₄	2.20	3.09				
Ethane - C ₂ H ₆	2.78	4.59				
Propane - C ₃ H ₈	3.10	6.87				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.29					
Ammonia - NH ₃ (PPM)	0.09					0.09
Light Oils - (0-100° C @ 1.6 Torr)			13.43	18.16	70.20	
Solvent - (100-230° C @ 1.6 Torr)				78.90	29.00	
Heavy Oil - (230-255° C @ 1.6 Torr)				2.20		
SRL						
Coal (MAF)						
Ash						
Water			86.57	0.74	0.80	99.91
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60				1.022	0.930	
Viscosity, cp						

SAMPLE DESCRIPTION	S-4A Ovhd	J-1 Disch	Cold Trap	S-7 Btms	S-4A Oil	S-4A Water
	Sx-30	Sx-32	Sx-33	Sx-34/39	Sx-37	Sx-37
Carbon	33.78	36.35	11.25	85.18	82.90	
Hydrogen	3.37	3.84	11.01	8.01	9.31	11.12
Nitrogen	0.36		0.01	0.24	0.19	0.07
Sulfur	1.04			1.17	0.49	
Oxygen (by diff)	61.45	59.81	77.73	5.40	7.11	88.81
Ash						
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	2	2	1	4	2	2

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/8/78

Run No: M-36C

Date: 2/9/78

Time: 0700-1900

WEIGHT PERCENT

Quantitative	SAMPLE DESCRIPTION	S-4A Total	S-7 Btms		S-4B Btms		
	SAMPLE POINT	Sx-37	Sx-34/39		Calc		
	Hydrogen - H ₂						
	Carbon Monoxide - CO						
	Carbon Dioxide - CO ₂						
	Hydrogen Sulfide - H ₂ S						
	Methane - CH ₄						
	Ethane - C ₂ H ₆						
	Propane - C ₃ H ₈						
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂						
	Ammonia - NH ₃ (PPM)				0.06		
	Light Oils - (0-100°C @ 1.6 Torr)	69.09	18.16		16.26		
	Solvent - (100-230°C @ 1.6 Torr)	28.54	78.90		6.72		
	Heavy Oil - (230-255°C @ 1.6 Torr)		2.20				
	SRL						
	Coal (MAF)						
	Ash						
	Water	2.37	0.74		76.96		
	Phenol						
	TOTAL	100	100		100		
	Pyridine Solubles (Wt % Ash free)						
	Melting Point °F						
	Specific Gravity 60/60						
	Viscosity, cp						

Elemental	Carbon	81.59	85.18		19.20		
	Hydrogen	9.34	8.01		10.70		
	Nitrogen	0.19	0.24		0.09		
	Sulfur	0.48	1.17		0.11		
	Oxygen (by diff)	8.40	5.40		69.90		
	Ash						
	TOTAL	100	100		100		
	Number of Sample Avg.	-	4		-		

Updated: 5/8/78

Date: 2/9/78

Time: 0700-1900

MOLE PERCENT

Quantitative

Elemental

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-36C
DATE: 2/9/78
TIME: 0700-1900

Feed

SAMPLE DESCRIPTION	F-1 Btms	LEC Btms	Solvent	S-8 Oil	S-7 Btms
SAMPLE POINT	Sx-2	Sx-12	Sx-14	Sx-16	Sx-34/39
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5	5		5
IBP		37	28.2		31.8
5%		55.4	81.0		86.2
10%		63.2	93.2		99.0
20%		74	118.2		120.7
30%		82.8	140		151
40%		91.8	162.4		169.8
50%		103.0	175.2		178.6
60%		117.3	185.8		185.6
70%		136.5	192		192.4
80%		162.0	200.8		202.1
90%		183.0	218.8		224.7
95%		193.8	250.3		259.5
DRY PT.		199.6	250.3		
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR				743	
IBP				42	
5%				58	
10%				66	
20%				78	
30%				87	
40%				95	
50%				102	
60%				107	
70%				111	
80%				114	
90%				117	
95%				118	
DRY PT.				122	
ASPHALTENE TEST					
WT % ASH	11.10				
WT % UNCONVERTED COAL	18.24				
WT % PRE-ASPHALTENES	25.85				
WT % ASPHALTENES	17.39				
WT % MALTENES & DIST. OIL	27.42				
TOTAL	100.00				
SAYBOLT VISCOSITY, SSU					
100° F		9.5	11.2		12.0
210° F		7.7	8.4		8.4
MISCELLANEOUS					
KF H ₂ O WT%(Row Sx)		-	0.23		0.74
INFRARED RATIO		0.19	0.24		0.25
SP. GRAV. 60/60 F		0.952	1.007		1.022

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-36C
DATE: 2/9/78
TIME: 0700-1900

SAMPLE DESCRIPTION		S-4A Oil			
SAMPLE POINT		Sx-37			
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR					
VOLUME PERCENT → 5% 10% 20% 30% 40% 50% 60% 70% 80% 90% → 95%	IBP				
	TEMPERATURE, °C				
DRY PT.					
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR		740			
VOLUME PERCENT → 5% 10% 20% 30% 40% 50% 60% 70% 80% 90% → 95%	IBP	69			
	TEMPERATURE, °C	84			
		142			
		181			
		200			
		210			
		227			
		247			
		272			
		304			
	340				
DRY PT.		350			
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F					
210° F					
MISCELLANEOUS					
KF H ₂ O WT % (Raw Sx)		0.80			
INFRARED RATIO					
SP. GRAV. 60/60 F		0.930			

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL- RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE

Revised 5/8/78

Run No: M-36C
Date: 2/9/78
Time: 0700-1900

MATERIAL OUT

Temperature: 736°F
Pressure: 2500 psig
LHSV 1.36
GHSV: 328

LBS/HOUR

Quantitative	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
	Hydrogen - H ₂	1.00	0.06				0.01
	Carbon Monoxide - CO	11.01	0.80				0.08
	Carbon Dioxide - CO ₂	8.34	2.73				0.58
	Hydrogen Sulfide - H ₂ S	0.10	0.05				
	Methane - CH ₄	0.81	0.09				0.02
	Ethane - C ₂ H ₆	0.35	0.11				0.04
	Propane - C ₃ H ₈	0.20	0.12				0.05
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.11	0.01				
	Ammonia - NH ₃	0.01		0.02			
	Light Oil - (0-100°C @ 1.6 Torr)			2.25		0.74	
	Solvent - (100-230°C @ 1.6 Torr)			0.93		0.01	
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Cool (MAF)						
	Ash						
	Water			18.28		0.25	
	Phenol						
	TOTAL	21.93	3.97	21.48	Negl	1.00	0.78

Elemental	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)	Ash		
	8.04	1.32	0.12	0.09	12.36			
	1.34	0.13	0.02	0.04	2.44			
	2.66	2.33	0.02	0.02	16.45			
	0.63	0.10			0.27			
	0.28	0.03			0.47			
	TOTAL	21.93	3.97	21.48	Negl	1.00	0.78	

PROJECT LIGNITE
MEASURED MATERIAL BALANCE

Revised 5/8/78

MATERIAL OUT

Temperature: 736°F
Pressure: 2500 psig
LHSV: 1.36
GHSV: 328

Run No: M-36C
Date: 2/9/78
Time: 0700-1900

LBS/HOUR

Quantitative	STREAM DESCRIPTION	Cold Trap	F-1 Btms	Recycle S-7 Btms	Solvent LEC Btms	Total Out
	SAMPLE POINT					
	Hydrogen - H ₂					1.07
	Carbon Monoxide - CO					11.89
	Carbon Dioxide - CO ₂					11.65
	Hydrogen Sulfide - H ₂ S					0.15
	Methane - CH ₄					0.92
	Ethane - C ₂ H ₆					0.50
	Propane - C ₃ H ₈					0.37
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.12
	Ammonia - NH ₃					0.03
	Light Oil - (0-100°C @ 1.6 Torr)	0.04		13.11	6.14	22.28
	Solvent - (100-230°C @ 1.6 Torr)		4.31	56.97	3.48	65.70
	Heavy Oil - (230-255°C @ 1.6 Torr)			1.59	0.13	1.72
	SRL		10.28			10.28
	Cool (MAF)		3.77			3.77
	Ash		2.29			2.29
	Water	0.27		0.53		19.33
	Phenol					
	TOTAL	0.31	20.65	72.20	9.75	152.07

Elemental	Carbon	0.04	15.60	61.50	8.23	98.32
	Hydrogen	0.03	1.03	5.78	0.94	11.69
	Nitrogen		0.21	0.17	0.02	0.56
	Sulfur		0.11	0.85	0.04	1.15
	Oxygen (by diff)	0.24	1.41	3.90	0.52	38.06
	Ash		2.29			2.29
	TOTAL	0.31	20.65	72.20	9.75	152.07

RUN CONDITIONS

RUN NO. M-36D
DATE MADE 2/10/78

TEMPERATURE °F

PREHEATER E-I
SAND BATH 768
INLET 214
OUTLET 750

DISSOLVERS

R-1A (1) _____
(2) _____
(3) _____
(4) _____
R-1B (1) 753
(2) 799
(3) 809
(4) 829

REACTOR PRODUCT SEPARATORS

S-1 544
S-2 51
S-3 500
S-4A 71
S-4B 71

PREHEATER E-II

DOWTHERM 680
INLET 453
OUTLET 644

VACUUM FLASH F-1

OVERHEAD VAPOR 530
UPPER WALL 626
LOWER WALL 639
DOWNCOMER 447
CONDENSATE
ACCUMULATOR, S-7 81

PREHEATER E-I3

DOWTHERM 453
INLET 72
OUTLET 205

LIGHT ENDS COLUMN, F-2

REBOILER 412
LOWER SECTION 329
FEED SECTION 277
OVERHEAD 209
S-8 69
S-9 -

SOLVENT COLUMN, F-3

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

HEAVY ENDS COLUMN, F-4

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

VACUUM FLASH, F-0

OVERHEAD NIU
BOTTOMS _____

PRECIPITATION TOWER, V-8

ZONE (1) NIU
(2) _____
(3) _____

RECOVERY TOWER V-9

REBOILER NIU
OVERHEAD _____

PRESSURE, psig

SEPARATORS S-1 and S-2
2500

SEPARATOR S-3
237

SEPARATORS S-4A and S-4B
60

VACUUM FLASH, F-1
19 Torr

LIGHT ENDS COLUMN, F-2
Atm

SOLVENT COLUMN, F-3
NIU

HEAVY ENDS COLUMN, F-4
NIU

VACUUM FLASH, F-0
NIU

SETTLING TOWER, V-8
NIU

RECOVERY TOWER, V-7
NIU

	Start	End	Run No: M-36D
Date of Run	2/10/78	2/10/78	Units: lbs/hr, °F, psig
Time of Run	0600	1800	Prep. By: BCL & GGB
Length of Run	12	hrs.	Updated: 5/8/78
Hours on Coal at End of Y.P.	99.0		

MASS RATES AND RUN CONDITIONS

149.98 TOTAL

☒ OPEN

☒ CLOSED

TOTAL 149.98

Numbers in parentheses are not included in the totals.

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
Normalized

Revised: 5/8/78

Run No: M-36D
Date: 2/10/78
Time: 0600-1800.

SUMMARY

lbs/hr.

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.34
GHSV: 333

Quantitative	STREAM DESCRIPTION	Total In	Total Out	Net Yield		Wt % MAF Coal	
	SAMPLE POINT						
	Hydrogen - H ₂	1.27	1.16	-0.11	}		
	Carbon Monoxide - CO	17.12	12.20	-4.92			
	Carbon Dioxide - CO ₂		11.63	11.63			
	Hydrogen Sulfide - H ₂ S		0.16	0.16			
	Methane - CH ₄		0.92	0.92	}	8.50	32.21
	Ethane - C ₂ H ₆		0.47	0.47			
	Propane - C ₃ H ₈		0.34	0.34			
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.18	0.16	-0.02	}		
	Ammonia - NH ₃		0.03	0.03			
	Light Oil - (0-100°C @ 1.6 Torr)	20.07	26.19	6.12			
	Solvent - (100-230°C @ 1.6 Torr)	61.70	59.77	-1.93		15.10	57.22
	Heavy Oil - (230-255°C @ 1.6 Torr)	2.81	3.75	0.94	}		
	SRL		9.97	9.97			
	Cool (MAF)	26.39	3.08	-23.31		-88.33	
	Ash	2.80	2.26	-0.54		-2.05	
	Water	17.64	17.89	0.25		0.95	
	Phenol						
	TOTAL	149.98	149.98	-0-			

Elemental	Carbon						
	Hydrogen						
	Nitrogen						
	Sulfur						
	Oxygen (by diff)						
	Ash						
	TOTAL						

PROJECT LIGNITE
MEASURED & ADJUSTED MATERIAL BALANCE

Revised: 5/8/78

Run No: M-36D
Date: 2/10/78
Time: 0600-1800

MATERIAL IN

lbs/hr.

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.34
GHSV: 333

Quantitative	STREAM DESCRIPTION	Feed Gas	Lignite	Feed Solvent		Total In
	SAMPLE POINT	Sx-21	Sx-1	Sx-14		
	Hydrogen - H ₂	1.27				1.27
	Carbon Monoxide-CO	17.12				17.12
	Carbon Dioxide-CO ₂					
	Hydrogen Sulfide-H ₂ S					
	Methane-CH ₄					
	Ethane-C ₂ H ₆					
	Propane-C ₃ H ₈					
	Butane-C ₄ H ₁₀					
	Nitrogen-N ₂	0.18				0.18
	Ammonia-NH ₃					
	Light Oil-(0-100°C @ 1.6 Torr)			20.07		20.07
	Solvent-(100-230°C @ 1.6 Torr)			61.70		61.70
	Heavy Oil-(230-255°C @ 1.6 Torr)			2.81		2.81
	SRL					
	Cool (MAF)		26.39			26.39
	Ash		2.80			2.80
	Water		17.00	0.64		17.64
	Phenol					
	TOTAL	18.57	46.19	85.22		149.98

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

Normalized
Revised: 5/8/78

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.34
GHSV: 333

Run No: M-36D
Date: 2/10/78
Time: 0600-1800

MATERIAL OUT

lbs/hr.

	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
		Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
Quantitative	Hydrogen - H ₂	1.08	0.07				0.01
	Carbon Monoxide - CO	11.28	0.83				0.09
	Carbon Dioxide - CO ₂	8.75	2.30				0.58
	Hydrogen Sulfide - H ₂ S	0.12	0.04				
	Methane - CH ₄	0.81	0.09				0.02
	Ethane - C ₂ H ₆	0.35	0.09				0.03
	Propane - C ₃ H ₈	0.18	0.11				0.05
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.14	0.02				
	Ammonia - NH ₃			0.03			
	Light Oil - (0-100°C @ 1.6 Torr)			2.07		1.55	
	Solvent - (100-230°C @ 1.6 Torr)			0.70		0.03	
	Heavy Oil - (230-255°C @1.6 Torr)						
	SRL						
	Cool (MAF)						
	Ash						
	Water			16.92			
	Phenol						
	TOTAL	22.71	3.55	19.72	Negl.	1.58	0.78

Elemental	Carbon						
	Hydrogen						
	Nitrogen						
	Sulfur						
	Oxygen (by diff)						
	Ash						
	TOTAL						

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

Normalized
Revised: 5/8/78

Run No: M-36D
Date: 2/10/78
Time: 0600-1800

MATERIAL OUT

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.34
GHSV: 333

lbs/hr.

	STREAM DESCRIPTION	Cold Trap	F-1 Btms	Recycle Solvent		Total Out
				S-7 Btms	LEC Btms	
	SAMPLE POINT	Sx-33	Sx-2	Sx-39	Sx-12	
	Hydrogen - H ₂					1.16
	Carbon Monoxide - CO					12.20
	Carbon Dioxide - CO ₂					11.63
	Hydrogen Sulfide - H ₂ S					0.16
	Methane - CH ₄					0.92
	Ethane - C ₂ H ₆					0.47
	Propane - C ₃ H ₈					0.34
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.16
	Ammonia - NH ₃					0.03
Quantitative	Light Oil - (0-100°C @ 1.6 Torr)			15.47	7.10	26.19
	Solvent - (100-230°C @ 1.6 Torr)		2.99	52.38	3.67	59.77
	Heavy Oil - (230-255°C @ 1.6 Torr)			3.60	0.15	3.75
	SRL		9.97			9.97
	Coal (MAF)		3.08			3.08
	Ash		2.26			2.26
	Water	0.32		0.65		17.89
	Phenol					
	TOTAL	0.32	18.30	72.10	10.92	149.98

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/8/78

Run No: M-36D

Date: 2/10/78

Time: 0600-1800

WEIGHT PERCENT

	WEIGHT PERCENT					
	Lignite	F-1 Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
SAMPLE DESCRIPTION						
SAMPLE POINT	Sx-1	Sx-2	Sx-12	Sx-14	Sx-16	Sx-16
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)						
Light Oils - (0-100° C @ 1.6 Torr)			65.00	23.55	98.00	
Solvent - (100-230° C @ 1.6 Torr)		16.35	33.60	72.40	2.00	
Heavy Oil - (230-255° C @ 1.6 Torr)			1.40	3.30		
SRL		54.46				
Coal (MAF)	57.14	16.83				
Ash	6.06	12.36				
Water	36.80			0.75		100
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)		80.80				
Melting Point °F		336				
Specific Gravity 60/60			0.962	1.020		
Viscosity, cp						

	WEIGHT PERCENT					
	Lignite	F-1 Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	2	2	2	-

WEIGHT PERCENT

Quantitative

Elemental-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/8/78

Run No: M-36D

Date: 2/10/78

Time: 0600-1800

WEIGHT PERCENT

	WEIGHT PERCENT					
	S-4A Ovhd	J-1 Discharge	Cold Trap	S-7 Btms	S-4A Oil	S-4A Water
SAMPLE DESCRIPTION	Sx-30	Sx-32	Sx-33	Sx-34/39	Sx-37	Sx-37
SAMPLE POINT						
Hydrogen - H ₂	2.00	1.14				
Carbon Monoxide - CO	23.33	11.32				
Carbon Dioxide - CO ₂	64.88	73.88				
Hydrogen Sulfide - H ₂ S	1.19					
Methane - CH ₄	2.62	2.50				
Ethane - C ₂ H ₆	2.54	4.29				
Propane - C ₃ H ₈	2.99	6.87				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.41					
Ammonia - NH ₃ (PPM)	0.04					0.16
Light Oils - (0-100°C @ 1.6 Torr)			0.49	21.45	71.67	
Solvent - (100-230°C @ 1.6 Torr)				72.65	27.40	
Heavy Oil - (230-255°C @1.6 Torr)				5.00		
SRL						
Coal (MAF)						
Ash						
Water			99.51	0.90	0.93	99.84
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60				1.030	0.937	
Viscosity, cp						

	WEIGHT PERCENT					
	S-4A Ovhd	J-1 Discharge	Cold Trap	S-7 Btms	S-4A Oil	S-4A Water
Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	1	4	2	2

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/8/78

Run No: M-36D

Date: 2/10/78

Time: 0600-1800

WEIGHT PERCENT

Quantitative	SAMPLE DESCRIPTION	S-4A Total	S-7 Btms			
	SAMPLE POINT	Sx-37	Sx-34/39			
	Hydrogen - H ₂					
	Carbon Monoxide - CO					
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					
	Ammonia - NH ₃ (PPM)					
	Light Oils - (0-100° C @ 1.6 Torr)	69.62	21.45			
	Solvent - (100-230° C @ 1.6 Torr)	26.62	72.65			
	Heavy Oil - (230-255° C @ 1.6 Torr)		5.00			
	SRL					
	Coal (MAF)					
	Ash					
	Water	3.76	0.90			
	Phenol					
	TOTAL	100	100			
Elemental	Pyridine Solubles (Wt % Ash free)					
	Melting Point °F					
	Specific Gravity 60/60		1.030			
	Viscosity, cp					
	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					
	Number of Sample Avg.	2	4			

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/8/78

Run No: M-36D

Date: 2/10/78

Time: 0600-1800

MOLE PERCENT

SAMPLE DESCRIPTION	Product Gas	Feed Gas	S-4A Ovhd	J-1 Discharge		
	Sx-19	Sx-21	Sx-30	Sx-32		
Hydrogen - H ₂	44.34	50.75	27.24	18.34		
Carbon Monoxide - CO	33.15	48.75	22.65	13.01		
Carbon Dioxide - CO ₂	16.35		40.09	54.01		
Hydrogen Sulfide - H ₂ S	0.30		0.95			
Methane - CH ₄	4.15		4.45	5.02		
Ethane - C ₂ H ₆	0.95		2.30	4.60		
Propane - C ₃ H ₈	0.35		1.85	5.02		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.40	0.50	0.40			
Ammonia - NH ₃ (PPM)	0.01		0.07			
Light Oils - (0-100°C @ 1.6 Torr)						
Solvent - (100-230°C @ 1.6 Torr)						
Heavy Oil - (230-255°C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water						
Phenol						
TOTAL	100	100	100	100		
Calc Mole Wt.	18.68	14.80	27.19	32.17		
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

SAMPLE DESCRIPTION	Product Gas	Feed Gas	S-4A Ovhd	J-1 Discharge		
	Sx-19	Sx-21	Sx-30	Sx-32		
Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	2	2		

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-36D
DATE: 2/10/78
TIME: 0600-1800

SAMPLE DESCRIPTION	F-1 Btms	LEC Btms	Feed Solv.	S-8 Oil	S-4B Oil
SAMPLE POINT	Sx-2	Sx-12	Sx-14	Sx-16	Sx-23
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5	5		
IBP		44.2	21.8		
5%		61.2	81.8		
10%		66.8	90.2		
20%		75.4	111.0		
30%		84.2	130.4		
40%		91.4	156.6		
50%		100.8	171.4		
60%		113.5	180.6		
70%		132.6	189		
80%		155.3	199		
90%		180.2	221.2		
95%		191.3	260		
DRY PT.		199			
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR				746	743
IBP				41	66
5%				57	86
10%				68	150
20%				82	190
30%				94	203
40%				107	214
50%				117	225
60%				125	243
70%				132	265
80%				138	294
90%				143	340
95%				146	
DRY PT.				146	344
ASPHALTENE TEST					
WT % ASH	12.36				
WT % UNCONVERTED COAL	16.83				
WT % PRE-ASPHALTENES	27.54				
WT % ASPHALTENES	16.95				
WT % MALTENES & DIST. OIL	26.32				
TOTAL	100.00				
SAYBOLT VISCOSITY, SSU					
100° F		9.5	12.1		
210° F		7.6	8.6		
MISCELLANEOUS					
KF H ₂ O WT%(Rcw Sx)			0.75		0.90
INFRARED RATIO		0.20	0.27		
SP. GRAV. 60/60 F		0.962	1.020		

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-36D
DATE: 2/10/78
TIME: 0600-1800

SAMPLE DESCRIPTION	S-7 Btms	S-4A Oil			
SAMPLE POINT	Sx-34/39	Sx-37			
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR	5				
IBP	21.6				
5%	74.8				
10%	91.9				
20%	116.4				
30%	143.7				
40%	163.1				
50%	178.2				
60%	187.4				
70%	196.3				
80%	207.1				
90%	233.5				
95%	260				
DRY PT.					
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR		745			
IBP		77			
5%		89			
10%		149			
20%		189			
30%		202			
40%		211			
50%		226			
60%		241			
70%		266			
80%		295			
90%		334			
95%					
DRY PT.		342			
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F					
210° F					
MISCELLANEOUS					
KF H ₂ O WT% (Raw Sx)	0.90	0.93			
INFRARED RATIO	0.29				
SP. GRAV. 60/60 F	1.030				

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE

Revised: 5/8/78

MATERIAL OUT

Temperature: 750°F

Pressure: 2500 psig

LHSV : 1.34

GHSV: 333

Run No: M-36D

Date: 2/10/78

Time: 0600-1800

lbs/hr.

STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
Hydrogen - H ₂	1.06	0.07				0.01
Carbon Monoxide - CO	11.05	0.81				0.09
Carbon Dioxide - CO ₂	8.57	2.26				0.57
Hydrogen Sulfide - H ₂ S	0.12	0.04				
Methane - CH ₄	0.79	0.09				0.02
Ethane - C ₂ H ₆	0.34	0.09				0.03
Propane - C ₃ H ₈	0.18	0.10				0.05
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.13	0.02				
Ammonia - NH ₃			0.03			
Light Oil - (0-100°C @ 1.6 Torr)			2.03		1.52	
Solvent - (100-230°C @ 1.6 Torr)			0.68		0.03	
Heavy Oil - (230-255°C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water			16.57			
Phenol						
TOTAL	22.24	3.48	19.31	Negl	1.55	0.77

ELEMENTAL	Quantitative					
	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)	Ash
TOTAL						

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/8/78

Run No: M-36D
Date: 2/10/78
Time: 0600-1800

MATERIAL OUT

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.34
GHSV: 333

lbs/hr.

Quantitative	STREAM DESCRIPTION	Cold Trap	F-1 Btms	← Recycle S-7 Btms	Solvent → LEC Btms	Total Out
	SAMPLE POINT	Sx-33	Sx-2	Sx-39	Sx-12	
	Hydrogen - H ₂					1.14
	Carbon Monoxide - CO					11.95
	Carbon Dioxide - CO ₂					11.40
	Hydrogen Sulfide - H ₂ S					0.16
	Methane - CH ₄					0.90
	Ethane - C ₂ H ₆					0.46
	Propane - C ₃ H ₈					0.33
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.15
	Ammonia - NH ₃					0.03
	Light Oil - (0-100°C @ 1.6 Torr)			15.47	7.10	26.12
	Solvent - (100-230°C @ 1.6 Torr)		2.93	52.38	3.67	59.69
	Heavy Oil - (230-255°C @ 1.6 Torr)			3.60	0.15	3.75
	SRL		9.76			9.76
	Coal (MAF)		3.02			3.02
	Ash		2.21			2.21
	Water	0.31		0.65		17.53
	Phenol					
	TOTAL	0.31	17.92	72.10	10.92	148.60

Elemental	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					

APPENDIX D

Process Development Unit Run M-37 February 21-24, 1978

Run M-36 completed the study of lignites from various locations, and the next phase of operations involved processing subbituminous coal in Run M-37 at operating conditions similar to those considered optimum for processing Zap lignite with a hydrogen-carbon monoxide reducing gas atmosphere. A sample of Decker, Montana subbituminous coal obtained from the Grand Forks Energy Research Center was used. Run M-37 suffered from a number of operating problems, and no yield periods were completed.

Nominal run conditions were one reactor operating at 2500 psig and 820-830°F, a solvent to raw coal ratio of 1.8, and a total slurry feed rate of 126 pounds per hour. Syngas with near equal volume fractions of hydrogen and carbon monoxide was fed at the usual rate of about 8 SCFM. The lower slurry feed rate (126 lbs/hr vs 140 lbs/hr for lignite) was utilized to obtain the same MAF coal feed rate of about 32 pounds per hour that had been used in previous runs with lignite. The subbituminous coal is substantially lower in water content than lignites, about 22 weight percent as compared with 28-36 percent. The SRL extraction equipment was in use during the run with the solids settling tower operating at 450°F and 300 psig. The deashing solvent recovery tower was operated at 500°F reboiler temperature and 2-5 psig pressure to recover toluene, which was the diluent used to promote settling and extraction in the precipitation tower, V-8. Toluene flows were maintained at about 87 pounds per hour with half flowing directly to the precipitation tower as a wash stream and the other half to the mix loop for F-0 vacuum bottoms dilution.

Run M-37 suffered a number of minor mechanical problems during startup, largely with leaky tube fittings and partially plugged lines. When these problems were resolved, the unit responded well when coal was first introduced. However, because of a malfunctioning pressure control valve, the pressure in the liquefaction section built up to 2800-2900 psig and the rupture disc on the high pressure condensate accumulator burst. The rapid depressuring of the dissolver resulted in the flashing of most of the liquefaction solvent, leaving the reactor partially plugged. After replacing the rupture disc the plant was started up again, but a 200-300 psi pressure drop across the reactor was encountered. The dissolver was blown down in an attempt to clear all deposits from it, but this proved ineffective since the plugging material had coked on the dissolver walls. Since installation of the second reactor had not been completed and insufficient time remained to clean the plugged reactor and resume operations before the end of the week, the run was discontinued.

Although Run M-37 was not successful, some analytical data concerning the Decker subbituminous coal and the reactor contents are attached. Also, following is a summary of the actual operating conditions during the brief coal processing period:

Feed Rates

Coal	45.2 lbs/hr
Gas	440 SCFH
Solvent	84.1 lbs/hr

Pressures

Dissolver	2500 psig
Inter. Sep.	230 psig
Vacuum Flash	20 Torr

Temperatures, °F

Slurry Preheater Outlet	729
Reactor Inlet	695
Reactor 3 ft	783
Reactor 7 ft	844
Reactor 11 ft	851
Reactor 15 ft	865
Reactor Outlet	779
Vacuum Flash Preheater Outlet	583
Vacuum Flash Ovhd	492

During Run M-37, only 199 pounds of coal was charged. The temperature increase through the reactor was greater than that usually experienced with lignite.

Run M-37 Coal Analyses

Coal: Subbituminous Coal from Decker, Montana

Proximate Analysis, Wt %

Moisture	21.75
Ash	3.87
Volatile Matter	32.40
Fixed Carbon	41.98
Total	100.00

Ultimate Analysis, Wt % (Dry Basis)

Carbon	71.56
Hydrogen	4.87
Nitrogen	1.20
Oxygen (by difference)	17.02
Sulfur	0.40
Ash	4.95
Total	100.00

X-Ray Fluorescence Analysis of Ash, Wt %

Loss on Ignition @ 800°C	0.2
SiO ₂	37.9
Al ₂ O ₃	16.5
Fe ₂ O ₃	5.1
TiO ₂	0.6
P ₂ O ₅	0.2
CaO	16.7
MgO	3.4
Na ₂ O	7.2
K ₂ O	0.3
SO ₃	11.9
Total	100.0

Sieve Analysis

U.S. Screen Sieve Mesh	Total Percentages	
	On	Passing
65	0.3	99.7
100	1.2	98.8
140	4.8	95.2
170	8.7	91.3
230	21.0	79.0
325	45.3	54.7
Pan	100.0	0.0

*Run M-37 Reactor Solids Analysis

Ash and Pyridine Solubles Analysis, Wt %

Composite Sample

As Received

Pyridine Solubles	27.92
Pyridine Insolubles	63.45
Ash	8.63

Toluene-Washed

Pyridine Solubles	7.87
Pyridine Insolubles	82.92
Ash	9.21

Spot Samples

Distance from base of Reactor, ft	Pyr. Solubles	Wt % Pyr. Insolubles	Ash
Top	29.44	64.67	5.89
14	19.58	73.64	6.78
12			7.30
10	38.89	55.02	6.09
8	26.12	66.95	6.93
5	44.86	49.00	6.14
2	56.20	38.80	5.00
Btm	50.26	44.24	5.50

X-Ray Fluorescence Analysis of Ash

Sample	Toluene-Washed Reactor Solids
Loss on Ignition @ 800°C	-
SiO ₂	27.7
Al ₂ O ₃	19.6
Fe ₂ O ₃	6.4
TiO ₂	0.9
P ₂ O ₅	0.4
CaO	18.7
MgO	5.1
Na ₂ O	5.0
K ₂ O	0.5
SO ₃	15.7
Total	100.0

X-Ray Diffraction Pattern Analysis of Toluene Washed Reactor Solids

Quartz - most abundant
Calcite - 1/3 abundant as Quartz

*When the Reactor was cleared by drilling, 78 pounds of solids were removed.

APPENDIX E

Process Development Unit Run M-38 March 6-10, 1978

Run M-38 was the second PDU run made using subbituminous coal. It was made at conditions similar to those of Run M-37 in which no yield periods were completed because of a number of mechanical problems. Nominal operating conditions were 820-830°F maximum reaction temperature and 2500 psig pressure using one reactor and recycling all recoverable organic distillates boiling above about 300°F. As in Run M-37, the solvent to raw coal ratio was 1.8 to 1 and the slurry charge rate was a nominal 126 pounds per hour. The MAF coal feed rate was about 32 pounds per hour, roughly equivalent to the MAF coal feed rate used when processing North Dakota lignite. A 50-50 mixture of hydrogen and carbon monoxide gas was used for the reducing gas atmosphere. The product deashing unit was on stream during the first half of the run and was operated at 450°F and 300 psig using toluene as the SRC extractant.

Decker, Montana subbituminous coal was used initially, but when the supply ran short an additional quantity of subbituminous coal from the Rosebud mine in Montana was obtained from the Grand Forks Energy Research Center and inadvertently mixed with the Decker coal during part of the run.

Operating problems during Run M-38 were (1) air leaking into the vacuum flash solvent recovery tower resulting in lowest attainable vacuum being about 20 Torr, or about 5 Torr above normal operating pressure, (2) solids entrained in the product gas stream plugging the reactor back pressure control valve and causing, on one occasion, a complete process upset, and (3) plugging in the toluene slurry transfer loop which finally necessitated the early shutdown of the product deashing equipment.

Four yield periods were completed during Run M-38. The first data period was made with Decker subbituminous coal and the fourth with Rosebud coal, but the two in between were inadvertently, made with alternate charge drums of Decker and Rosebud coals. Thus, only yield periods M-38A and M-38D were worked up and for them both the measured and adjusted material balances along with the sample analyses, average run conditions and ASTM D86 and D1160 distillations are attached. The material balance closure was fair to good with M-38A having 99.8 percent recovery and M-38D having 101.4 percent.

Yield period M-38A was made with the product deashing unit on stream and was adjusted to 100 percent recovery by increasing the solids precipitation tower bottoms rate and then calculating the V-8 bottoms composition from the flow rates and compositions of the other streams entering and leaving the deashing section, assuming no loss or chemical change to take place and holdup in the settling tower to be insignificant. After these adjustments,

a forced carbon balance was made and the oil-water ratio of the waste water stream calculated.

Yield period M-38D was adjusted to 100 percent recovery by first normalizing the outgoing product streams, and then by making a forced carbon balance and calculating the oil content of the S-4B waste water stream.

A comparison of the two yield periods after adjustment is shown in Table 1 and a comparison of the two subbituminous coals is shown in Table 2. It appears that the higher ash content of the Rosebud coal catalyzed or otherwise affected the reaction resulting in lower net gas yields and higher net liquid yields with the overall SRC yield remaining about the same. Thus the Rosebud coal produced a higher yield of distillable oil (28% of the net liquids) than its lower ash counterpart, Decker coal (22% of the net liquids).

Overall conversion of MAF subbituminous coal to gas, oil and SRC was roughly 82 percent.

To demonstrate the differences in liquefaction yields when using subbituminous coal and lignite, a comparison of the results obtained with the two Montana subbituminous coals and a number of North Dakota lignites is presented in Table 3. Some variations in operating conditions exist for the various runs compared, but in general net gas yields when processing subbituminous coal are 1/3 lower per pound MAF coal than those obtained with lignite. Actual net gas yields for the subbituminous coal processed at 810°F and 2500 psig were roughly 18 weight percent of the MAF coal charged while lignite results in about a 30 weight percent yield.

The net liquid yield with subbituminous coal was comparable to its lignite counterpart and was in the range of 60-70 weight percent of the MAF coal fed. This was also true for the non-distillable, pyridine soluble material (SRC or SRL) which averaged about 50 weight percent of the MAF coal for both subbituminous and lignite coals tested.

Overall MAF coal conversion for the subbituminous coals was about 82 percent and for the North Dakota lignites 87 percent. The apparent lower conversion may in part be due to the slightly lower temperature used in processing the subbituminous coal (810°F vs 825°F for the lignites).

It is interesting to note that the coals with the higher ash contents, Rosebud subbituminous coal and Gascoyne lignite, in general yielded the higher overall conversions when compared with other coals of equal rank.

Reducing gas consumption on a hydrogen equivalent basis averaged about 2.1 weight percent of the MAF coal for North Dakota lignite

and about 1.5 weight percent for Montana subbituminous coal. The difference in reducing gas consumption is perhaps the result of the water gas shift reaction being more predominant with the higher moisture lignite. This is also indicated by the water consumptions when processing the two ranks of coal. For lignite, water consumption averaged 4.9 weight percent of the MAF coal fed and for subbituminous coal it averaged 2.1 weight percent.

Near the finish of Run M-38, the liquefaction reactor was cooled by flushing with solvent and then blown down. The recovered solids were analyzed both on a "as-received" and on a "toluene-washed" basis. The results of the various analyses are shown on the following page. The reactor solids from processing subbituminous coal contain less calcite than quartz, which is just the reverse of the results of processing lignite. It might be inferred that the higher oxygen content of lignite results in the formation of more calcium carbonate than is formed with subbituminous coal. The total amount of oily solids removed from the reactor at the end of Run M-38 was about 0.005 pounds per cubic foot of reactor per pound of coal fed as compared with 0.010 for Velva lignite and 0.018 for Gascoyne lignite. Here again the data indicate lignite to have a greater tendency to form dissolver deposits, either by particle agglomeration or by mineral growth, than the subbituminous coals.

Run M-38 Reactor Solids

Ash and Pyridine Extraction Test

	"As Received"	"Toluene-Washed"
Pyridine Solubles	82.09	2.23
Pyridine Insolubles	9.23	51.39
Ash	8.68	46.38
Total	100.00	100.00

X-Ray Fluorescence Analysis of Ash from Toluene Washed Solids, Wt %

Loss on Ignition @ 800°C	-
SiO ₂	28.7
Al ₂ O ₃	12.5
Fe ₂ O ₃	5.8
TiO ₂	0.4
P ₂ O ₅	0.5
CaO	36.5
MgO	5.7
Na ₂ O	2.4
K ₂ O	0.3
SO ₃	7.2
Total	100.0

X-Ray Diffraction Pattern Analysis of Toluene Washed Solids

Quartz - most abundant
 Calcite - 4/5 as abundant as quartz

Sieve Analysis of Toluene Washed Solids

U.S. Screen Sieve Mesh	Total Percentages	
	On	Passing
65	14.5	85.5
100	40.1	59.9
140	56.5	43.5
170	62.4	37.6
230	74.6	25.4
325	88.1	11.9
Pan	100.0	0.0

Table 1
Process Development Unit Run M-38

Yield Period Coal Mine	M-38A Decker	M-38D Rosebud
Reactor Used	R-1B	R-1B
LHSV, Hr ⁻¹	1.30	1.31
GHSV, Hr ⁻¹	302	306
Solvent/MAF Coal Ratio	2.66	2.97
H ₂ /CO Mole Ratio	1.09	1.09
Temperatures, °F		
Preheater Outlet E-1	750	742
Reactor 3 ft	738	737
Reactor 7 ft	776	777
Reactor 11 ft	791	786
Reactor 15 ft	813	806
Max Reaction	813	806
Vacuum Flash Ovhd	497	504
Dissolver Pressure, psig	2500	2500
Solvent Recycle, %		
Actual	97.7	97.8
Potential	105.1	106.7
Yields, Wt % MAF Coal		
Net Gas	20.9	16.0
Net Liquid	61.8	70.6
(Dist Oil)	(13.6)	(19.8)
(SRC)	(48.2)	(50.8)
Net H ₂ O and Ash	-2.8	-3.6
Unconverted Coal	20.1	17.0
Overall Material Balance, %	99.8	101.4
Percent Ash in Feed Coal	3.9	11.2

Table 2
Process Development Unit Run M-38

Analyses of Montana Subbituminous Coals

Mine Location	Decker	Rosebud
Proximate Analysis, Wt %		
Moisture	21.75	25.74
Ash	3.87	11.23
Volatile Matter	32.40	26.55
Fixed Carbon	41.98	36.48
Total	100.00	100.00
Ultimate Analyses, Wt % (Dry Basis)		
Carbon	71.56	64.61
Hydrogen	4.87	4.34
Nitrogen	1.20	0.80
Oxygen (by difference)	17.02	13.63
Sulfur	0.40	1.50
Ash	4.95	15.12
Total	100.00	100.00
X-Ray Fluorescence Analysis of Ash, Wt %		
Loss on Ignition @ 800°C.	0.2	
SiO ₂	37.9	40.4
Al ₂ O ₃	16.5	18.5
Fe ₂ O ₃	5.1	16.9
TiO ₂	0.6	0.8
P ₂ O ₅	0.2	0.3
CaO	16.7	9.1
MgO	3.4	3.0
Na ₂ O	7.2	0.4
K ₂ O	0.3	0.6
SO ₂	11.9	10.0
Total	100.0	100.0

Sieve Analyses

U.S. Screen Sieve	Total Percentages			
	On		Passing	
	Decker	Rosebud	Decker	Rosebud
65	0.3	0.4	99.7	99.6
100	1.2	1.0	98.8	99.0
140	4.8	3.9	95.2	96.1
170	8.7	17.0	91.3	83.0
230	21.0	45.4	79.0	54.6
325	45.3	80.6	54.7	19.4
Pan	100.0	100.0	0.0	0.0

Table 3

Process Development Unit Runs M-31, M-32, M-33, L-1, L-2, M-34, M-35, M-36 and M-38

Run	M-38A	M-38D	M-35 & 36	M-33 & 34	L-1 & 2	M-31 & 32	Average
Coal	Decker	Rosebud	Velva	Gascoyne	Zap	Larson	of N.D.
	← Subbituminous →		← Lignite →				Lignites
Reactor Used	R-1B	R-1B	R-1B	R-1B	-	R-1B	-
LHSV, Hr ⁻¹	1.30	1.31	1.38	1.42	1.41	1.42	1.41
GHSV, Hr ⁻¹	302	306	343	310	330	319	326
Solvent/MAF Coal Ratio	2.66	2.97	3.15	3.9	3.03	2.94	3.25
Mole Percent H ₂ in Feed Gas	51.9	52.0	51.4	51.5	49.1	52.2	51.0
Temperatures, °F							
Preheater Outlet, E-1	750	742	744	746	734	742	742
Reactor 3 ft	738	737	739	762	752	755	752
Reactor 7 ft	776	777	794	793	792	783	790
Reactor 11 ft	791	786	787	795	793	795	792
Reactor 15 ft	813	806	822	820	816	830	822
Max Reaction	813	806	822	820	816	830	822
Vacuum Flash Ovhd	497	504	522	520	508	513	516
Dissolver Pressure, psig	2500	2500	2500	2500	2500	2500	2500
Solvent Recycle, %							
Actual	97.7	97.8	95.8	93.7	92.6	85.7	92.0
Potential	105.1	106.7	105.7	103.2	104.0	101.4	103.6
Yields, Wt % MAF Coal							
Net Gas	20.9	16.0	29.9	35.6	31.0	33.1	32.4
Net Liquid	61.8	70.6	61.1	64.4	63.4	54.7	60.9
(Dist Oil)	(13.6)	(19.8)	(17.6)	(15.1)	(13.0)	(4.1)	(12.4)
(SRC)	(48.2)	(50.8)	(43.5)	(49.3)	(50.4)	(50.6)	(48.5)
Net H ₂ O and Ash	-2.8	-3.6	-2.7	-8.7	-4.5	-7.6	-5.9
Unconverted Coal	20.1	17.0	11.7	8.7	10.1	19.8	12.6
Overall Material Balance, %	99.8	101.4	99.4	99.0	98.9	99.1	99.1
Number of Y.P. Averaged	1	1	5	6	17	5	-

RUN CONDITIONS

RUN NO. M-38A

DATE MADE 3/7-8/78

TEMPERATURE °F

PREHEATER E-I
SAND BATH 766
INLET 268
OUTLET 750

DISSOLVERS
R-1A (1) NIU
(2) _____
(3) _____
(4) _____
R-1B (1) 738
(2) 776
(3) 791
(4) 813

REACTOR PRODUCT SEPARATORS
S-1 532
S-2 41
S-3 518
S-4A 72
S-4B 74

PREHEATER E-II
DOWTHERM 678
INLET 368
OUTLET 614

VACUUM FLASH F-I
OVERHEAD VAPOR 497
UPPER WALL 669
LOWER WALL 664
DOWNCOMER 388
CONDENSATE
ACCUMULATOR, S-7 77

PREHEATER E-13
DOWTHERM 427
INLET 72
OUTLET 217

LIGHT ENDS COLUMN, F-2
REBOILER 414
LOWER SECTION 326
FEED SECTION 270
OVERHEAD 207
S-8 69
S-9 -

SOLVENT COLUMN, F-3
REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

HEAVY ENDS COLUMN, F-4
REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

VACUUM FLASH, F-0
OVERHEAD 300
BOTTOMS -

PRECIPITATION TOWER, V-8
ZONE (1) 296
(2) 302
(3) 372

RECOVERY TOWER V-9
REBOILER 471
OVERHEAD 320

PRESSURE, psig

SEPARATORS S-1 and S-2
2500

SEPARATOR S-3
241

SEPARATORS S-4A and S-4B
61

VACUUM FLASH F-1
20 Torr

LIGHT ENDS COLUMN, F-2
Atm

SOLVENT COLUMN, F-3
NIU

HEAVY ENDS COLUMN, F-4
NIU

VACUUM FLASH, F-0
20 Torr

SETTLING TOWER, V-8
250

RECOVERY TOWER, V-7
3

	Start	End	Run No: M-38A
Date of Run	3/7/78	3/8/78	Units: lbs/hr. °F. psig
Time of Run	1300	0100	Prep. By: BCL & GGB
Length of Run	12	hrs.	Updated: 5/24/78
Hours on Coal at End of Y. P.	26.3		

MASS RATES AND RUN CONDITIONS

OPEN

CLOSED

Numbers in parentheses are not included in the totals.

TOTAL 235.18

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Adjusted with Forced Carbon Balance
Revised: 5/24/78

Run No: M-38A
Date: 3/7-8/78
Time: 1300-0100

SUMMARY

lbs/hr.

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.30
GHSV: 302

Quantitative	STREAM DESCRIPTION	Total In	Total Out	Net Yield		Wt % MAF Coal	
	SAMPLE POINT						
	Hydrogen - H ₂	1.18	1.15	-0.03			
	Carbon Monoxide - CO	15.16	10.16	-5.00			
	Carbon Dioxide - CO ₂		10.20	10.20			
	Hydrogen Sulfide - H ₂ S		0.11	0.11			
	Methane - CH ₄		0.79	0.79	6.73	20.94	
	Ethane - C ₂ H ₆		0.45	0.45			
	Propane - C ₃ H ₈		0.24	0.24			
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.18	0.14	-0.04			
	Ammonia - NH ₃		0.01	0.01			
	Light Oil - (0-100°C @ 1.6 Torr)	110.06	109.18	-0.88			
	Solvent - (100-230°C @ 1.6 Torr)	65.10	70.25	5.15	19.86	61.79	
	Heavy Oil - (230-255°C @ 1.6 Torr)	1.55	1.65	0.10			
	SRL		15.49	15.49			
	Coal (MAF)	32.14	6.45	-25.69		-79.93	
	Ash	1.71	1.70	-0.01		-0.03	
	Water	8.10	7.21	-0.89		-2.77	
	Phenol						
	TOTAL	235.18	235.18	-0-			

Elemental	Carbon	188.70	188.70	-0-			
	Hydrogen	19.13	19.49	0.36			
	Nitrogen	0.62	0.59	-0.03			
	Sulfur	1.22	1.17	-0.05			
	Oxygen (by diff)	23.80	23.53	-0.27			
	Ash	1.71	1.70	-0.01			
	TOTAL	235.18	235.18	-0-			

PROJECT LIGNITE
MEASURED & ADJUSTED MATERIAL BALANCE
Revised: 5/24/78

Run No: M-38A
Date: 3/7-8/78
Time: 1300-0100

MATERIAL IN

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.30
GHSV: 302

lbs/hr.

STREAM DESCRIPTION	Feed Gas	Coal	Feed Solvent	Deashing Solvent		Total In
	Sx-21	Sx-1	Sx-14	Sx-40		
Hydrogen - H ₂	1.18					1.18
Carbon Monoxide - CO	15.16					15.16
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.18					0.18
Ammonia - NH ₃						
Light Oil - (0-100°C @ 1.6 Torr)			19.91	90.15		110.06
Solvent - (100-230°C @ 1.6 Torr)			64.19	0.91		65.10
Heavy Oil - (230-255°C @ 1.6 Torr)			1.55			1.55
SRL						
Coal (MAF)		32.14				32.14
Ash		1.71				1.71
Water		7.93	0.17			8.10
Phenol						
TOTAL	16.52	41.78	85.82	91.06		235.18

Carbon	6.50	24.02	75.08	83.10		188.70
Hydrogen	1.18	2.62	7.41	7.92		19.13
Nitrogen	0.18	0.29	0.15			0.62
Sulfur		0.36	0.85	0.01		1.22
Oxygen (by diff)	8.66	12.78	2.33	0.03		23.80
Ash		1.71				1.71
TOTAL	16.52	41.78	85.82	91.06		235.18

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Adjusted with Forced Carbon Balance
Revised: 5/24/78

Run No: M-38A
Date: 3/7-8/78
Time: 1300-0100

MATERIAL OUT

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.30
GHSV: 302

lbs/hr.

Quantitative	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Calc	Sx-44	Sx-16	Sx-32
	Hydrogen - H ₂	1.07	0.08				
	Carbon Monoxide - CO	9.31	0.80				0.05
	Carbon Dioxide - CO ₂	7.98	2.03				0.19
	Hydrogen Sulfide - H ₂ S	0.06	0.05				
	Methane - CH ₄	0.68	0.10				0.01
	Ethane - C ₂ H ₆	0.35	0.09				0.01
	Propane - C ₃ H ₈	0.15	0.08				0.01
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.12	0.02				
	Ammonia - NH ₃			0.01			
	Light Oil - (0-100°C @ 1.6 Torr)			0.45		1.98	
	Solvent - (100-230°C @ 1.6 Torr)			0.12		0.02	
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Cool (MAF)						
	Ash						
	Water			6.65		0.18	
	Phenol						
	TOTAL	19.72	3.25	7.23	Negl.	2.18	0.27

Elemental	Carbon	7.08	1.11	0.48		1.73	0.09
	Hydrogen	1.34	0.14	0.80		0.26	0.01
	Nitrogen	0.13	0.02	0.01			
	Sulfur	0.05	0.04				
	Oxygen (by diff)	11.12	1.94	5.94		0.19	0.17
	Ash						
	TOTAL	19.72	3.25	7.23	Negl.	2.18	0.27

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Adjusted with Forced Carbon Balance
Revised: 5/24/78

Run No: M-38A
Date: 3/7-8/78
Time: 1300-0100

MATERIAL OUT

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.30
GHSV: 302

	lbs/hr.					
	Cold Trap	V-8 Btms	F-1 Btms	V-9 Ovhd	← Recycle S-7 Btms	Solvent → LEC Btms
STREAM DESCRIPTION	Sx-33	Calc	Sx-1	Sx-28	Sx-39	Sx-12
SAMPLE POINT						
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃						
Light Oil - (0-100°C @ 1.6 Torr)	0.16	1.11	-	85.68	15.53	4.27
Solvent - (100-230°C @ 1.6 Torr)	0.08	0.38	6.49	1.04	60.14	1.98
Heavy Oil - (230-255°C @ 1.6 Torr)					1.55	0.10
SRL		0.34	15.15			
Cool (MAF)		3.10	3.35			
Ash		0.88	0.82			
Water	0.10				0.28	
Phenol						
TOTAL	0.34	5.81	25.81	86.72	77.50	6.35

Carbon	0.17	3.97	21.86	79.13	67.78	5.30
Hydrogen	0.04	0.32	1.54	7.55	6.84	0.65
Nitrogen		0.03	0.25		0.14	0.01
Sulfur		0.04	0.12	0.01	0.89	0.02
Oxygen (by diff)	0.13	0.57	1.22	0.03	1.85	0.37
Ash		0.88	0.82			
TOTAL	0.34	5.81	25.81	86.72	77.50	6.35

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
V-8 Btms Adjusted with Forced Carbon Balance
Revised: 5/24/78

Run No: M-38A
Date: 3/7-8/78
Time: 1300-0100

MATERIAL OUT

lbs/hr.

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.30
GHSV: 302

Quantitative	STREAM DESCRIPTION					Total Out
	SAMPLE POINT					
	Hydrogen - H ₂					1.15
	Carbon Monoxide - CO					10.16
	Carbon Dioxide - CO ₂					10.20
	Hydrogen Sulfide - H ₂ S					0.11
	Methane - CH ₄					0.79
	Ethane - C ₂ H ₆					0.45
	Propane - C ₃ H ₈					0.24
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.14
	Ammonia - NH ₃					0.01
	Light Oil - (0-100°C @ 1.6 Torr)					109.18
	Solvent - (100-230°C @ 1.6 Torr)					70.25
	Heavy Oil - (230-255°C @ 1.6 Torr)					1.65
	SRL					15.49
	Cool (MAF)					6.45
	Ash					1.70
	Water					7.21
	Phenol					
	TOTAL					235.18

Elemental	Carbon					188.70
	Hydrogen					19.49
	Nitrogen					0.59
	Sulfur					1.17
	Oxygen (by diff)					23.53
	Ash					1.70
	TOTAL					235.18

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/24/78

Run No: M-38A

Date: 3/7-8/78

Time: 1300-0100

WEIGHT PERCENT

	SAMPLE DESCRIPTION	WEIGHT PERCENT				
		Coal	P-1 Btms	LEC Btms	Feed Solvent	S-8 Oil
	SAMPLE POINT	Sx-1	Sx-2	Sx-12	Sx-14	Sx-16
	Hydrogen - H ₂					
	Carbon Monoxide - CO					
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					
	Ammonia - NH ₃ (PPM)					
	Light Oils - (0-100°C @ 1.6 Torr)			67.30	23.20	99.00
	Solvent - (100-230°C @ 1.6 Torr)		25.15	31.20	74.80	1.00
	Heavy Oil - (230-255°C @1.6 Torr)			1.50	1.80	
	SRL		58.71			
	Coal (MAF)	76.92	12.97			
	Ash	4.10	3.17			
	Water	18.98			0.20	100
	Phenol					
	TOTAL	100	100	100	100	100
	Pyridine Solubles (Wt % Ash free)		86.61			
	Melting Point °F		167			
	Specific Gravity 60/60				1.003	
	Viscosity, cp					

	SAMPLE DESCRIPTION	WEIGHT PERCENT				
		Coal	P-1 Btms	LEC Btms	Feed Solvent	S-8 Oil
	SAMPLE POINT	Sx-1	Sx-2	Sx-12	Sx-14	Sx-16
	Hydrogen - H ₂					
	Carbon Monoxide - CO					
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					
	Ammonia - NH ₃ (PPM)					
	Light Oils - (0-100°C @ 1.6 Torr)			67.30	23.20	99.00
	Solvent - (100-230°C @ 1.6 Torr)		25.15	31.20	74.80	1.00
	Heavy Oil - (230-255°C @1.6 Torr)			1.50	1.80	
	SRL		58.71			
	Coal (MAF)	76.92	12.97			
	Ash	4.10	3.17			
	Water	18.98			0.20	100
	Phenol					
	TOTAL	100	100	100	100	100
	Pyridine Solubles (Wt % Ash free)		86.61			
	Melting Point °F		167			
	Specific Gravity 60/60				1.003	
	Viscosity, cp					

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/24/78

Run No: M-38A

Date: 3/7-8/78

Time: 1300-0100

WEIGHT PERCENT

SAMPLE DESCRIPTION	S-8	Product	Feed	S-4B	S-4B	S-4B
	Total	Gas	Gas	Oil	Water	Total
SAMPLE POINT	Sx-16	Sx-19	Sx-21	*Sx-23	Sx-23	Sx-23
Hydrogen - H ₂		5.42	7.16			
Carbon Monoxide - CO		47.21	91.78			
Carbon Dioxide - CO ₂		40.45				
Hydrogen Sulfide - H ₂ S		0.29				
Methane - CH ₄		3.47				
Ethane - C ₂ H ₆		1.78				
Propane - C ₃ H ₈		0.74				
Butane - C ₄ H ₁₀						
Nitrogen - N ₂		0.63	1.06			
Ammonia - NH ₃ (PPM)		0.01			0.10	0.10
Light Oils - (0-100° C @ 1.6 Torr)	90.75			78.50		0.94
Solvent - (100-230° C @ 1.6 Torr)	0.92			21.20		0.25
Heavy Oil - (230-255° C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water	8.33			0.30	99.90	98.71
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60				0.901		
Viscosity, cp						

Carbon	79.20	35.89	39.33	84.82		1.01
Hydrogen	11.86	6.80	7.16	10.03	11.12	11.11
Nitrogen	0.07	0.64	1.06	0.14	0.08	0.08
Sulfur	0.04	0.27		0.41		
Oxygen (by diff)	8.83	56.40	52.45	4.60	88.80	87.80
Ash						
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	-	2	2	2	2	-

*Insufficient oil sample. Assumed to be same as S-4A oil.

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/24/78

Run No: M-38A

Date: 3/7-8/78

Time: 1300-0100

WEIGHT PERCENT

SAMPLE DESCRIPTION	V-8 Btms	V-9 Ovhd	S-4A Ovhd	J-1 Discharge	Cold Trap	S-7 Btms
	Sx-27	Sx-28	Sx-30	Sx-32	Sx-33	Sx-34/39
Hydrogen - H ₂			2.51	1.67		
Carbon Monoxide - CO			24.67	18.54		
Carbon Dioxide - CO ₂			62.52	71.23		
Hydrogen Sulfide - H ₂ S			1.41			
Methane - CH ₄			2.91	2.33		
Ethane - C ₂ H ₆			2.91	2.69		
Propane - C ₃ H ₈			2.52	3.54		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂			0.50			
Ammonia - NH ₃ (PPM)			0.05			
Light Oils - (0-100°C @ 1.6 Torr)	27.25	98.80			48.06	20.04
Solvent - (100-230°C @ 1.6 Torr)	22.08	1.20			23.40	77.60
Heavy Oil - (230-255°C @ 1.6 Torr)						2.00
SRL	4.09					
Coal (MAF)	33.29					
Ash	13.29					
Water					28.54	0.36
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60						1.001
Viscosity, cp						

Carbon	74.21	91.25	34.19	34.18	48.92	87.46
Hydrogen	5.76	8.71	4.37	3.43	10.68	8.83
Nitrogen	0.50		0.54		0.11	0.18
Sulfur	0.79	0.01	1.33		0.10	1.14
Oxygen (by diff)	5.45	0.03	59.57	62.39	40.19	2.39
Ash	13.29					
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	3	2	2	3	1	4

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/24/78

Run No: M-38A

Date: 3/7-8/78

Time: 1300-0100

WEIGHT PERCENT

	Quantitative					
	S-4A Oil	S-4A Water	S-4A Total	S-7 Btms	Deashing Solvent	V-8 Btms
SAMPLE DESCRIPTION	Sx-37	Sx-37	Sx-37	Sx-34/39	Sx-40	Calc.
SAMPLE POINT						
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)		0.08				
Light Oils - (0-100° C @ 1.6 Torr)	78.50		77.05	20.04	99.00	19.08
Solvent - (100-230° C @ 1.6 Torr)	21.20		20.81	77.60	1.00	6.57
Heavy Oil - (230-255° C @ 1.6 Torr)				2.00		
SRL						5.85
Coal (MAF)						53.35
Ash						15.15
Water	0.30	99.92	2.14	0.36		
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60	0.901			1.001		
Viscosity, cp						

	Elemental					
Carbon	84.82		83.25	87.46	91.26	68.33
Hydrogen	10.03	11.11	10.05	8.83	8.70	5.43
Nitrogen	0.14	0.07	0.14	0.18	-	0.56
Sulfur	0.41		0.40	1.14	0.01	0.67
Oxygen (by diff)	4.60	88.82	6.16	2.39	0.03	9.86
Ash						15.15
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	2	2	-	4	2	-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/24/78

Run No: M-38A

Date: 3/7-8/78

Time: 1300-0100

WEIGHT PERCENT

Quantitative	SAMPLE DESCRIPTION	S-4B Btms				
	SAMPLE POINT	Calc				
	Hydrogen - H ₂					
	Carbon Monoxide - CO					
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					
	Ammonia - NH ₃ (PPM)	0.09				
	Light Oils - (0-100°C @ 1.6 Torr)	6.19				
	Solvent - (100-230°C @ 1.6 Torr)	1.67				
	Heavy Oil - (230-255°C @ 1.6 Torr)					
	SRL					
	Coal (MAF)					
	Ash					
	Water	92.05				
	Phenol					
	TOTAL	100				
	Pyridine Solubles (Wt % Ash free)					
	Melting Point °F					
	Specific Gravity 60/60					
	Viscosity, cp					

Elemental	Carbon	6.69				
	Hydrogen	11.04				
	Nitrogen	0.08				
	Sulfur	0.03				
	Oxygen (by diff)	82.16				
	Ash					
	TOTAL	100				
	Number of Sample Avg.	-				

PROJECT LIGNITE

SAMPLE ANALYSIS

Updated: 5/24/78

Run No: M-38A

Date: 3/7-8/78

Time: 1300-0100

MOLE PERCENT

Quantitative	SAMPLE DESCRIPTION	Product Gas	Feed Gas	S-4A Ovhd	J-1 Discharge		
	SAMPLE POINT	Sx-19	Sx-21	Sx-30	*Sx-32		
	Hydrogen - H_2	48.04	51.90	31.69	24.37		
	Carbon Monoxide - CO	29.90	47.55	22.29	19.28		
	Carbon Dioxide - CO_2	16.30		35.94	47.15		
	Hydrogen Sulfide - H_2S	0.15		1.05			
	Methane - CH_4	3.85		4.60	4.25		
	Ethane - C_2H_6	1.05		2.45	2.61		
	Propane - C_3H_8	0.30		1.45	2.34		
	Butane - C_4H_{10}						
	Nitrogen - N_2	0.40	0.55	0.45			
	Ammonia - NH_3 (PPM)	0.01		0.08			
	Light Oils - (0-100°C @ 1.6 Torr)						
	Solvent - (100-230°C @ 1.6 Torr)						
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Coal (MAF)						
	Ash						
	Water						
	Phenol						
	TOTAL	100	100	100	100		
	Calc Mole Wt	17.73	14.51	25.29	29.12		
	Melting Point °F						
	Specific Gravity 60/60						
	Viscosity, cp						

Elemental						
	Carbon					
	Hydrogen					
	Nitrogen					
	Sulfur					
	Oxygen (by diff)					
	Ash					
	TOTAL					
	Number of Sample Avg.	2	2	2	3	

*Insufficient sample. Analysis assumed to be average of M-38C and D J-1 discharge sample analyses. -189-

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-38A
DATE: 3/7-8/78
TIME: 1300-0100

SAMPLE DESCRIPTION	F-1 Btms	LEC Btms	Feed Solv.	S-8 Btms	V-9 Ovhd
SAMPLE POINT	Sx-2	Sx-12	Sx-14	Sx-16	Sx-28
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5	5		
IBP		45	24.6		
5%		58.4	77.8		
10%		62.8	91.4		
20%		71	117.3		
30%		78.2	140.8		
40%		88	162.6		
50%		97.6	176.5		
60%		111.3	183.8		
70%		129.4	191.2		
80%		157.2	196.4		
90%		164.3	208.6		
95%		195.2	232		
DRY PT.		199.3	232		
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR				746	744
IBP				40	110
5%				69	112
10%				80	113
20%				91	113
30%				100	114
40%				106	114
50%				111	114
60%				112	114
70%				114	114
80%				116	114
90%				118	114
95%				122	114
DRY PT.				122	117
ASPHALTENE TEST					
WT % ASH	3.17				
WT % UNCONVERTED COAL	12.97				
WT % PRE-ASPHALTENES	21.06				
WT % ASPHALTENES	22.11				
WT % MALTENES & DIST. OIL	40.69				
TOTAL	100.00				
SAYBOLT VISCOSITY, SSU					
100° F		8.8	10.3		
210° F		7.6	8.3		
MISCELLANEOUS					
KF H ₂ O WT% (Row Sx)			0.20		
INFRARED RATIO		0.17	0.22		
SP. GRAV. 60/60 F			1.003		

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-38A
DATE: 3/7-8/78
TIME: 1300-0100

SAMPLE DESCRIPTION	V-9 Btms	Cold Trab Oil	S-7 Btms	S-4A Oil	Leaching Solv
SAMPLE POINT	Sx-29	Sx-33	Sx-34/39	Sx-37	Sx-40
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR	5		5		
IBP	112		20		
5%	176		81.2		
10%	184		95		
20%	195		121		
30%	199		144.7		
40%	208		163		
50%	226		176.4		
60%	251		182.8		
70%	260		189.1		
80%			196.4		
90%			209.8		
95%			231.4		
DRY PT.			231.4		
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR		740		748	744
IBP		98		58	112
5%		117		95	112
10%		121		120	113
20%		130		145	113
30%		138		164	114
40%		151		193	114
50%		197		210	114
60%		211		226	114
70%		228		251	114
80%		304		277	114
90%				323	114
95%				346	114
DRY PT.		322		353	122
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F			10.4		
210° F			8.5		
MISCELLANEOUS					
KF H ₂ O WT % (Raw Sx)			0.36	0.30	
INFRARED RATIO			0.23		
SP. GRAV. 60/60 F			1.001	0.901	

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/24/78

Run No: M-38A
Date: 3/7-8/78
Time: 1300-0100

MATERIAL OUT

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.30
GHSV: 302

lbs/hr.

	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
Quantitative	Hydrogen - H ₂	1.07	0.08				
	Carbon Monoxide - CO	9.31	0.80				0.05
	Carbon Dioxide - CO ₂	7.98	2.03				0.19
	Hydrogen Sulfide - H ₂ S	0.06	0.05				
	Methane - CH ₄	0.68	0.10				0.01
	Ethane - C ₂ H ₆	0.35	0.09				0.01
	Propane - C ₃ H ₈	0.15	0.08				0.01
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.12	0.02				
	Ammonia - NH ₃			0.01			
	Light Oil - (0-100°C @ 1.6 Torr)			0.07		1.98	
	Solvent - (100-230°C @ 1.6 Torr)			0.02		0.02	
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Coal (MAF)						
	Ash						
	Water			7.13		0.18	
	Phenol						
	TOTAL	19.72	3.25	7.23	Negl.	2.18	0.27

Elemental	Carbon	7.08	1.11	0.07		1.73	0.09
	Hydrogen	1.34	0.14	0.80		0.26	0.01
	Nitrogen	0.13	0.02	0.01			
	Sulfur	0.05	0.04				
	Oxygen (by diff)	11.12	1.94	6.35		0.19	0.17
	Ash						
	TOTAL	19.72	3.25	7.23	Negl.	2.18	0.27

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/24/78

MATERIAL OUT

Run No: M-38A
Date: 3/7-8/78
Time: 1300-0100

lbs/hr.

Temperature: 750°F
Pressure: 2500 psig
LHSV : 1.30
GHSV: 302

	STREAM DESCRIPTION	Cold Trap	V-8 Btms	F-1 Btms	V-9 Ovhd	←Recycle	Solvent→
						S-7 Btms	LEC Btms
	SAMPLE POINT	Sx-33	Sx-27	Sx-1	Sx-28	Sx-39	Sx-12
	Hydrogen - H ₂						
	Carbon Monoxide - CO						
	Carbon Dioxide - CO ₂						
	Hydrogen Sulfide - H ₂ S						
	Methane - CH ₄						
	Ethane - C ₂ H ₆						
	Propane - C ₃ H ₈						
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂						
	Ammonia - NH ₃						
Quantitative	Light Oil - (0-100°C @ 1.6 Torr)	0.16	1.48		85.68	15.53	4.27
	Solvent - (100-230°C @ 1.6 Torr)	0.08	1.20	6.49	1.04	60.14	1.98
	Heavy Oil - (230-255°C @1.6 Torr)					1.55	0.10
	SRL		0.22	15.15			
	Coal (MAF)		1.81	3.35			
	Ash		0.72	0.82			
	Water	0.10				0.28	
	Phenol						
	TOTAL	0.34	5.43	25.81	86.72	77.50	6.35

Elemental	Carbon	0.17	4.03	21.86	79.13	67.78	5.30
	Hydrogen	0.04	0.31	1.54	7.55	6.84	0.65
	Nitrogen		0.03	0.25		0.14	0.01
	Sulfur		0.04	0.12	0.01	0.89	0.02
	Oxygen (by diff)	0.13	0.30	1.22	0.03	1.85	0.37
	Ash		0.72	0.82			
	TOTAL	0.34	5.43	25.81	86.72	77.50	6.35

PROJECT LIGNITE
MEASURED MATERIAL BALANCE

Revised: 5/24/78

MATERIAL OUT

Temperature: 750°F
Pressure: 2500 psig
LHSV: 1.30
GHSV: 302

Run No: M-38A
Date: 3/7-8/78
Time: 1300-0100

lbs/hr.

Quantitative	STREAM DESCRIPTION					Total Out
	SAMPLE POINT					
	Hydrogen - H ₂					1.15
	Carbon Monoxide - CO					10.16
	Carbon Dioxide - CO ₂					10.20
	Hydrogen Sulfide - H ₂ S					0.11
	Methane - CH ₄					0.79
	Ethane - C ₂ H ₆					0.45
	Propane - C ₃ H ₈					0.24
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.14
	Ammonia - NH ₃					0.01
	Light Oil - (0-100°C @ 1.6 Torr)					109.17
	Solvent - (100-230°C @ 1.6 Torr)					70.97
	Heavy Oil - (230-255°C @ 1.6 Torr)					1.65
	SRL					15.37
	Cool (MAF)					5.16
	Ash					1.54
	Water					7.69
	Phenol					
	TOTAL					234.80

Elemental	Carbon					188.35
	Hydrogen					19.48
	Nitrogen					0.59
	Sulfur					1.17
	Oxygen (by diff)					23.67
	Ash					1.54
	TOTAL					234.80

RUN CONDITIONS

RUN NO. M-38D
DATE MADE 3/10/78

TEMPERATURE °F

PREHEATER E-I
SAND BATH 762
INLET 265
OUTLET 742

DISSOLVERS

R-1A (1) NIU
(2) _____
(3) _____
(4) _____
R-1B (1) 737
(2) 777
(3) 786
(4) 806

REACTOR PRODUCT SEPARATORS

S-1 538
S-2 43
S-3 520
S-4A 75
S-4B 75

PREHEATER E-II

DOWTHERM 660
INLET 466
OUTLET 624

VACUUM FLASH F-I

OVERHEAD VAPOR 504
UPPER WALL 653
LOWER WALL 574
DOWNCOMER 392

CONDENSATE

ACCUMULATOR, S-7 80

PREHEATER E-13
DOWTHERM 426
INLET 76
OUTLET 203

LIGHT ENDS COLUMN, F-2

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____
S-8 NIU
S-9 73

SOLVENT COLUMN, F-3

REBOILER 320
LOWER SECTION 331
FEED SECTION 300
OVERHEAD 241

HEAVY ENDS COLUMN, F-4

REBOILER NIU
LOWER SECTION _____
FEED SECTION _____
OVERHEAD _____

VACUUM FLASH, F-O

OVERHEAD NIU
BOTTOMS _____

PRECIPITATION TOWER, V-8

ZONE (1) NIU
(2) _____
(3) _____

RECOVERY TOWER V-9

REBOILER NIU
OVERHEAD _____

PRESSURE, psig

SEPARATORS S-1 and S-2
2500

SEPARATOR S-3
200

SEPARATORS S-4A and S-4B
60

VACUUM FLASH, F-I
20 Torr

LIGHT ENDS COLUMN, F-2
NIU

SOLVENT COLUMN, F-3
1

HEAVY ENDS COLUMN, F-4
NIU

VACUUM FLASH, F-O
NIU

SETTLING TOWER, V-8
NIU

RECOVERY TOWER, V-7
NIU

	Start	End	Run No: M-38D
Date of Run	3/10/78	3/10/78	Units: lbs/hr, °F, psig
Time of Run	0430	1430	Prep. By: BCL&GGB
Length of Run	10	hours	Updated: 5/23/78
Hours on Coal at End of Y.P.	79.3		

MASS RATES AND RUN CONDITIONS

142.31 TOTAL

✕ OPEN

✕ CLOSED

TOTAL 142.31

Numbers in parentheses are not included in the totals.

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
Normalized with forced carbon balance

Run No: M-38D
Date: 3/10/78
Time: 0430-1430

Revised: 5/23/78

SUMMARY
lbs/hr.

Temperature: 742°F
Pressure: 2500 psig
LHSV 1.31
GHSV: 306

	STREAM DESCRIPTION	Total In	Total Out	Net Yield	Wt. %	
					MAF Coal	
	SAMPLE POINT					
	Hydrogen - H ₂	1.20	1.05	-0.15		
	Carbon Monoxide - CO	15.36	10.68	-4.68		
	Carbon Dioxide - CO ₂		8.06	8.06		
	Hydrogen Sulfide - H ₂ S		0.30	0.30		
	Methane - CH ₄		0.49	0.49	4.49	16.03
	Ethane - C ₂ H ₆		0.24	0.24		
	Propane - C ₃ H ₈		0.21	0.21		
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂	0.15	0.15	-0-		
	Ammonia - NH ₃		0.02	0.02		
	Light Oil - (0-100°C @ 1.6 Torr)	21.99	25.69	3.70		
	Solvent - (100-230°C @ 1.6 Torr)	59.60	60.80	1.20	19.78	70.59
	Heavy Oil - (230-255°C @ 1.6 Torr)	1.67	2.32	0.65		
	SRL		14.23	14.23		
	Coal (MAF)	28.02	4.75	-23.27	-83.05	
	Ash	5.00	4.40	-0.60	-2.14	
	Water	9.32	8.92	-0.40	-1.43	
	Phenol					
	TOTAL	142.31	142.31	-0-		

	Carbon	100.79	100.79	-0-		
	Hydrogen	10.79	10.89	0.10		
	Nitrogen	0.65	0.72	0.07		
	Sulfur	1.37	1.40	0.03		
	Oxygen (by diff)	23.71	24.11	0.40		
	Ash	5.00	4.40	-0.60		
	TOTAL	142.31	142.31	-0-		

PROJECT LIGNITE
MEASURED & ADJUSTED MATERIAL BALANCE

Revised: 5/23/78

Run No: M-38D
Date: 3/10/78
Time: 0430-1430

MATERIAL IN
lbs/hr.

Temperature: 742°F
Pressure: 2500 psig
LHSV 1.31
GHSV: 306

Quantitative	STREAM DESCRIPTION	Feed Gas	Coal	Feed Solvent			Total In
	SAMPLE POINT	Sx-21	Sx-1	Sx-14			
	Hydrogen - H ₂	1.20					1.20
	Carbon Monoxide - CO	15.36					15.36
	Carbon Dioxide - CO ₂						
	Hydrogen Sulfide - H ₂ S						
	Methane - CH ₄						
	Ethane - C ₂ H ₆						
	Propane - C ₃ H ₈						
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.15					0.15
	Ammonia - NH ₃						
	Light Oil - (0-100°C @ 1.6 Torr)			21.99			21.99
	Solvent - (100-230°C @ 1.6 Torr)			59.60			59.60
	Heavy Oil - (230-255°C @ 1.6 Torr)			1.67			1.67
	SRL						
	Coal (MAF)		28.02				28.02
	Ash		5.00				5.00
	Water		8.99	0.33			9.32
	Phenol						
	TOTAL	16.71	42.01	83.59			142.31

Elemental	Carbon	6.58	21.67	72.54			100.79
	Hydrogen	1.20	2.34	7.25			10.79
	Nitrogen	0.15	0.23	0.27			0.65
	Sulfur		0.55	0.82			1.37
	Oxygen (by diff)	8.78	12.22	2.71			23.71
	Ash		5.00				5.00
	TOTAL	16.71	42.01	83.59			142.31

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
Normalized with forced carbon balance

Revised: 5/23/78

Temperature: 742°F

Run No: M-38D

MATERIAL OUT

Pressure: 2500 psig

Date: 3/10/78

LHSV 1.31

Time: 0430-1430

lbs/hr.

GHSV: 306

	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Calc.	Sx-44	Sx-16	Sx-32
Quantitative	Hydrogen - H ₂	0.97	0.08				
	Carbon Monoxide - CO	9.79	0.89				
	Carbon Dioxide - CO ₂	6.23	1.83				
	Hydrogen Sulfide - H ₂ S	0.17	0.13				
	Methane - CH ₄	0.42	0.07				
	Ethane - C ₂ H ₆	0.18	0.06				
	Propane - C ₃ H ₈	0.14	0.07				
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.14	0.01				
	Ammonia - NH ₃	0.01		0.01			
	Light Oil - (0-100°C @ 1.6 Torr)			3.23		0.69	
	Solvent - (100-230°C @ 1.6 Torr)			0.78		0.01	
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Cool (MAF)						
	Ash						
	Water			8.43			
	Phenol						
	TOTAL	18.05	3.14	12.45	Negl	0.70	Negl

Elemental	Carbon	6.47	1.04	3.42		0.57	
	Hydrogen	1.15	0.13	1.34		0.10	
	Nitrogen	0.15	0.01	0.02			
	Sulfur	0.16	0.12	0.01			
	Oxygen (by diff)	10.12	1.84	7.66		0.03	
	Ash						
	TOTAL	18.05	3.14	12.45	Negl	0.70	Negl

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

Normalized with forced carbon balance

Run No: M-38D
Date: 3/10/78
Time: 0430-1430

Revised: 5/23/78

MATERIAL OUT

lbs/hr.

Temperature: 742°F
Pressure: 2500 psig
LHSV 1.31
GHSV: 306

STREAM DESCRIPTION	Cold Trap	F-1 Btms	Recycle		Solvent LEC Btms	Total Out
			S-7 Btms	Sx-34		
SAMPLE POINT	Sx-33	Sx-2	Sx-34	Sx-12		
Hydrogen - H ₂						1.05
Carbon Monoxide - CO						10.68
Carbon Dioxide - CO ₂						8.06
Hydrogen Sulfide - H ₂ S						0.30
Methane - CH ₄						0.49
Ethane - C ₂ H ₆						0.24
Propane - C ₃ H ₈						0.21
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						0.15
Ammonia - NH ₃						0.02
Light Oil - (0-100°C @ 1.6 Torr)			16.84	4.93		25.69
Solvent - (100-230°C @ 1.6 Torr)		2.83	55.28	1.90		60.80
Heavy Oil - (230-255°C @1.6 Torr)			2.20	0.12		2.32
SRL		14.23				14.23
Coal (MAF)		4.75				4.75
Ash		4.40				4.40
Water	0.01		0.48			8.92
Phenol						
TOTAL	0.01	26.21	74.80	6.95		142.31

Carbon		18.44	64.96	5.89		100.79
Hydrogen		1.29	6.16	0.72		10.89
Nitrogen		0.27	0.25	0.02		0.72
Sulfur		0.35	0.73	0.03		1.40
Oxygen (by diff)	0.01	1.46	2.70	0.29		24.11
Ash		4.40				4.40
TOTAL	0.01	26.21	74.80	6.95		142.31

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/23/78

Run No: M-38D

Date: 3/10/78

Time: 0430-1430

WEIGHT PERCENT

SAMPLE DESCRIPTION	Coal	F-1 Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
	Sx-1	Sx-2	Sx-12	Sx-14	Sx-16	Sx-16
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)						
Light Oils - (0-100° C @ 1.6 Torr)			71.00	26.30	98.00	
Solvent - (100-230° C @ 1.6 Torr)		10.80	27.30	71.30	2.00	
Heavy Oil - (230-255° C @ 1.6 Torr)			1.70	2.00		
SRL		54.28				
Coal (MAF)	66.71	18.14				
Ash	11.89	16.78				
Water	21.40			0.40		100
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)		78.20				
Melting Point °F		272				
Specific Gravity 60/60				1.006		
Viscosity, cp						

ELEMENTAL	WEIGHT PERCENT					
	Coal	F-1 Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
Carbon	51.58	70.37	84.80	86.78	81.41	
Hydrogen	5.57	4.93	10.34	8.68	13.70	11.11
Nitrogen	0.55	1.01	0.28	0.32	0.16	
Sulfur	1.31	1.35	0.44	0.98	0.21	
Oxygen (by diff)	29.10	5.56	4.14	3.24	4.52	88.89
Ash	11.89	16.78				
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	2	2	2	2	2	-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/23/78

Run No: M-38D

Date: 3/10/78

Time: 0430-1430

WEIGHT PERCENT

	SAMPLE DESCRIPTION	S-8 Total	Product Gas	Feed Gas	S-4B Oil	S-4B Water	S-4B Total
	SAMPLE POINT	Sx-16	Sx-19	Sx-21	*Sx-23	Sx-23	Sx-23
	Hydrogen - H ₂		5.38	7.18			
	Carbon Monoxide - CO		54.23	91.95			
	Carbon Dioxide - CO ₂		34.50				
	Hydrogen Sulfide - H ₂ S		0.96				
	Methane - CH ₄		2.31				
	Ethane - C ₂ H ₆		1.02				
	Propane - C ₃ H ₈		0.75				
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂		0.79	0.87			
	Ammonia - NH ₃ (PPM)		0.06			0.07	0.05
Quantitative	Light Oils - (0-100°C @ 1.6 Torr)	98.00			79.90		17.24
	Solvent - (100-230°C @ 1.6 Torr)	2.00			19.30		4.16
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Coal (MAF)						
	Ash						
	Water				0.80	99.93	78.55
	Phenol						
	TOTAL	100	100	100	100	100	100
	Pyridine Solubles (Wt % Ash free)						
	Melting Point °F						
	Specific Gravity 60/60				0.923		
	Viscosity, cp						

Elemental	Carbon	81.41	35.81	39.41	84.64		18.26
	Hydrogen	13.70	6.37	7.18	10.00	11.11	10.87
	Nitrogen	0.16	0.84	0.87	0.28	0.06	0.11
	Sulfur	0.21	0.90		0.30		0.06
	Oxygen (by diff)	4.52	56.08	52.54	4.78	88.83	70.70
	Ash						
	TOTAL	100	100	100	100	100	100
	Number of Sample Avg.	-	2	2	2	2	-

*Insufficient sample for analysis. Assumed to be the same as the S-4A oil.
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PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/23/78

Run No: M-38D

Date: 3/10/78

Time: 0430-1430

WEIGHT PERCENT

Quantitative	SAMPLE DESCRIPTION	S-4A Ovhd	J-1 Disch	Cold Trap	S-7 Btms	S-4A Oil	S-4A Water
	SAMPLE POINT	Sx-30	Sx-32	Sx-33	Sx-34/39	Sx-37	Sx-37
	Hydrogen -H ₂	2.58	1.89				
	Carbon Monoxide-CO	28.31	19.58				
	Carbon Dioxide-CO ₂	58.20	69.28				
	Hydrogen Sulfide-H ₂ S	4.04					
	Methane-CH ₄	2.13	2.75				
	Ethane-C ₂ H ₆	1.99	2.69				
	Propane-C ₃ H ₈	2.21	3.81				
	Butane-C ₄ H ₁₀						
	Nitrogen-N ₂	0.40					
	Ammonia-NH ₃ (PPM)	0.14					0.08
	Light Oils-(0-100°C @ 1.6 Torr)			22.10	22.51	79.90	
	Solvent-(100-230°C @ 1.6 Torr)			18.00	73.90	19.30	
	Heavy Oil-(230-255°C @1.6 Torr)				2.95		
	SRL						
	Coal (MAF)						
	Ash						
	Water			59.90	0.64	0.80	99.92
	Phenol						
	TOTAL	100	100	100	100	100	100
Elemental	Pyridine Solubles (Wt % Ash free)						
	Melting Point °F						
	Specific Gravity 60/60				1.012	0.927	
	Viscosity, cp						

Elemental	Carbon	33.00	34.61	35.94	86.84	84.64	
	Hydrogen	4.17	3.81	10.61	8.24	10.00	11.11
	Nitrogen	0.52		0.12	0.33	0.28	0.07
	Sulfur	3.80		0.08	0.98	0.30	
	Oxygen (by diff)	58.51	61.58	53.25	3.61	4.78	88.82
	Ash						
	TOTAL	100	100	100	100	100	100
Elemental	Number of Sample Avg.	2	2	1	4	2	-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/23/78

Run No: M-38D

Date: 3/10/78

Time: 0430-1430

WEIGHT PERCENT

Quantitative	SAMPLE DESCRIPTION	S-4A Total	S-7 Btms		S-4 B Btms		
	SAMPLE POINT	Sx-37	Sx-34/39		Calc		
	Hydrogen - H ₂						
	Carbon Monoxide - CO						
	Carbon Dioxide - CO ₂						
	Hydrogen Sulfide - H ₂ S						
	Methane - CH ₄						
	Ethane - C ₂ H ₆						
	Propane - C ₃ H ₈						
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂						
	Ammonia - NH ₃ (PPM)				0.05		
	Light Oils - (0-100°C @ 1.6 Torr)	79.90	22.51		25.93		
	Solvent - (100-230°C @ 1.6 Torr)	19.30	73.90		6.26		
	Heavy Oil - (230-255°C @1.6 Torr)		2.95				
	SRL						
	Coal (MAF)						
	Ash						
	Water	0.80	0.64		67.76		
	Phenol						
	TOTAL	100	100		100		
	Pyridine Solubles (Wt % Ash free)						
	Melting Point °F						
	Specific Gravity 60/60	0.927	1.012				
	Viscosity, cp						

Elemental	Carbon	84.64	86.84		27.47		
	Hydrogen	10.00	8.24		10.75		
	Nitrogen	0.28	0.33		0.13		
	Sulfur	0.30	0.98		0.10		
	Oxygen (by diff)	4.78	3.61		61.55		
	Ash						
	TOTAL	100	100		100		
	Number of Sample Avg.	-	4		-		

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/23/78

Run No: M-38D

Date: 3/10/78

Time: 0430-1430

MOLE PERCENT

Quantitative	SAMPLE DESCRIPTION	Product Gas	Feed Gas	S-4A Ovhd	J-1 Disch		
	SAMPLE POINT	Sx-19	Sx-21	Sx-30	Sx-32		
	Hydrogen - H ₂	47.47	52.00	32.15	26.46		
	Carbon Monoxide - CO	34.18	47.55	25.18	19.61		
	Carbon Dioxide - CO ₂	13.84		32.94	44.16		
	Hydrogen Sulfide - H ₂ S	0.50		2.96			
	Methane - CH ₄	2.55		3.31	4.82		
	Ethane - C ₂ H ₆	0.60		1.65	2.52		
	Propane - C ₃ H ₈	0.30		1.25	2.43		
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.50	0.45	0.35			
	Ammonia - NH ₃ (PPM)	0.06		0.21			
	Light Oils - (0-100°C @ 1.6 Torr)						
	Solvent - (100-230°C @ 1.6 Torr)						
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Coal (MAF)						
	Ash						
	Water						
	Phenol						
	TOTAL	100	100	100	100		
	Calc Mole Wt.	17.65	14.48	24.90	28.05		
	Melting Point °F						
	Specific Gravity 60/60						
	Viscosity, cp						

Elemental	Carbon						
	Hydrogen						
	Nitrogen						
	Sulfur						
	Oxygen (by diff)						
	Ash						
	TOTAL						
	Number of Sample Avg.	2	2	2	2		

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-38D
DATE: 3/10/78
TIME: 0430-1430

SAMPLE DESCRIPTION	LEC Btms	Feed Solv	S-8 Oil	Cold Trap	S-7 Btms
SAMPLE POINT	Sx-12	Sx-14	Sx-16	Sx-33	Sx-34/39
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR	5	5			5
IBP	49.4	30.6			40.5
5%	61.4	78.2			82.8
10%	65.8	89.0			93.7
20%	73.2	109.2			115.4
30%	79.8	132.3			137
40%	87.8	155.3			157
50%	96.6	172.2			171
60%	107.2	182.0			179.4
70%	123.2	191.3			187.3
80%	144.8	199.0			196.5
90%	175.6	223.0			219.3
95%	199.6	258.6			260
DRY PT.	199.6	258.6			
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR			739	732	
IBP			40	84	
5%			64	96	
10%			73	97	
20%			84	100	
30%			93	104	
40%			101	108	
50%			109	194	
60%			116	206	
70%			123	218	
80%			131	243	
90%			142		
95%			148		
DRY PT.			149	302	
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F	9.1	11.2			11.6
210° F	7.9	8.6			8.4
MISCELLANEOUS					
KF H ₂ O WT% (Raw Sx)		0.40		59.90	0.64
INFRARED RATIO	0.18	0.24			0.24
SP. GRAV. 60/60 F		1.006			1.012

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE

SAMPLE ANALYSES

RUN NO: M-38D

DATE: 3/10/78

TIME: 0430-1430

SAMPLE DESCRIPTION		S-4A Oil			
SAMPLE POINT		Sx-37			
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR					
VOLUME PERCENT IBP 5% 10% 20% 30% 40% 50% 60% 70% 80% 90% 95% DRY PT.	TEMPERATURE, °C				
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR		736			
VOLUME PERCENT IBP 5% 10% 20% 30% 40% 50% 60% 70% 80% 90% 95% DRY PT.	TEMPERATURE, °C	71			
		82			
		150			
		184			
		196			
		204			
		216			
		229			
		249			
		274			
320					
336					
340					
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F					
210° F					
MISCELLANEOUS					
KF H ₂ O WT% (Raw Sx)		0.80			
INFRARED RATIO					
SP. GRAV. 60/60 F		0.927			

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL

ASPHALTENES - BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL

MALTENES - N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES

DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE

Revised: 5/23/78

Run No: M-38 D
Date: 3/10/78
Time: 0430-1430

MATERIAL OUT

lbs/hr.

Temperature: 742°F
Pressure: 2500 psig
LHSV: 1.31
GHSV: 306

Quantitative	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
	Hydrogen - H ₂	1.00	0.08				
	Carbon Monoxide - CO	10.13	0.92				
	Carbon Dioxide - CO ₂	6.44	1.89				
	Hydrogen Sulfide - H ₂ S	0.18	0.13				
	Methane - CH ₄	0.43	0.07				
	Ethane - C ₂ H ₆	0.19	0.07				
	Propane - C ₃ H ₈	0.14	0.07				
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.15	0.01				
	Ammonia - NH ₃	0.01	0.01	0.01			
	Light Oil - (0-100°C @ 1.6 Torr)			2.22		0.71	
	Solvent - (100-230°C @ 1.6 Torr)			0.53		0.01	
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Cool (MAF)						
	Ash						
	Water			10.11			
	Phenol						
	TOTAL	18.67	3.25	12.87	Negl	0.72	Negl
Elemental	Carbon	6.69	1.07	2.35		0.59	
	Hydrogen	1.19	0.14	1.40		0.10	
	Nitrogen	0.15	0.02	0.01			
	Sulfur	0.17	0.12	0.01			
	Oxygen (by diff)	10.47	1.90	9.10		0.03	
	Ash						
	TOTAL	18.67	3.25	12.87	Negl	0.72	Negl

PROJECT LIGNITE
MEASURED MATERIAL BALANCE

Revised: 5/23/78

Run No: M-38D
Date: 3/10/78
Time: 0430-1430

MATERIAL OUT

lbs/hr.

Temperature: 742°F
Pressure: 2500 psig
LHSV 1.31
GHSV: 306

STREAM DESCRIPTION	Cold Trap	F-1 Btms	Recycle		Solvent	Total Out
			S-7 Btms	LEC Btms		
SAMPLE POINT	Sx-33	Sx-2	Sx-39	Sx-12		
Hydrogen - H ₂						1.08
Carbon Monoxide - CO						11.05
Carbon Dioxide - CO ₂						8.33
Hydrogen Sulfide - H ₂ S						0.31
Methane - CH ₄						0.50
Ethane - C ₂ H ₆						0.26
Propane - C ₃ H ₈						0.21
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						0.16
Ammonia - NH ₃						0.03
Light Oil - (0-100°C @ 1.6 Torr)			16.84	4.93		24.70
Solvent - (100-230°C @ 1.6 Torr)		2.93	55.28	1.90		60.65
Heavy Oil - (230-255°C @ 1.6 Torr)			2.20	0.12		2.32
SRL		14.71				14.71
Coal (MAF)		4.91				4.91
Ash		4.55				4.55
Water	0.01		0.48			10.60
Phenol						
TOTAL	0.01	27.10	74.80	6.95		144.37

Carbon		19.07	64.96	5.89		100.62
Hydrogen		1.34	6.16	0.72		11.05
Nitrogen		0.27	0.25	0.02		0.72
Sulfur		0.36	0.73	0.03		1.42
Oxygen (by diff)	0.01	1.51	2.70	0.29		26.01
Ash		4.55				4.55
TOTAL	0.01	27.10	74.80	6.95		144.37

APPENDIX F

Process Development Unit Run M-39 March 20-23, 1978

Run M-39 was the last run in the 1977-78 PDU test program. It was scheduled for 96 hours of operation rather than the usual 120 hours because of the Good Friday holiday.

Run M-39 was made using Zap lignite at operating conditions considered optimum with this type of lignite but using pure hydrogen instead of a 50-50 hydrogen-carbon monoxide syngas. The run was made using one reactor with specified nominal conditions of 2500 psig pressure, 820-830°F maximum dissolver temperature, 1.8 solvent to raw coal ratio and 140 pounds per hour total slurry feed rate. Hydrogen was fed at the rate of about 8 SCF per minute. The operating conditions for the deashing section were the same as in the previous runs. The solids settling tower, V-8, was operated at 450°F and 300 psig pressure. The solvent (toluene) recovery tower, V-9, was operated at 500°F reboiler temperature and 2-5 psig pressure. Toluene flows were roughly 0.2 gpm, with half flowing directly to the settling tower as a wash stream while the remainder went to the mix loop to dilute the partial vacuum flash drum, F-0, bottoms.

During the first two days of operation, the run was plagued with various leaks and mechanical problems. The worst leak occurred at the bottom closure of reactor, R-1B, forcing shutdown of that dissolver. Switching to the standby reactor R-1A, caused a delay which resulted in the completion of only one yield period during the run.

The completed yield period, M-39A, had a measured material balance of 98.5 percent and was adjusted to 100 percent recovery by normalizing the outgoing product streams and by making a forced carbon balance. As an intermediate step in adjustment, the composition of the V-8 bottoms was calculated based on the flow rates and compositions of the streams flowing in and out of the deashing section. The completed material balance workup including both quantitative and elemental breakdowns along with sample analyses and the results of ASTM D86 and D1160 distillations is attached. A summary of the M-39A operating conditions and run results is shown in Table 1 along with similar summaries for yield periods L-1K and L-2D which were also made using Zap lignite but with varying amounts of hydrogen in the feed gas.

It appears that as the reducing gas atmosphere becomes enriched in hydrogen, the overall conversion of MAF lignite to gas, oil and SRL decreases. Net gas and organic liquid yields also decrease as more of the carbon monoxide in the feed gas is replaced by hydrogen. Over the range of feed gas compositions studied, the various liquefaction yields seems to decrease almost linearly with increasing concentration of feed gas hydrogen, as illustrated in Figure 1.

In Run M-39, the overall MAF lignite conversion to Gas, oil and SRL was 82.1 percent with net gas and organic liquid yields being 16.8 and 54.2 weight percent of the MAF coal charged, respectively.

Hydrogen consumption was about 2.3 SCF per pound raw coal or 1.91 weight percent of the MAF coal fed. During the run, water was made with a net yield of about 11.8 weight percent of the MAF coal.

Results of the M-39A deashing operations were fair with the total for ash and pyridine insolubles reduced to 1.3 weight percent of the final product.

About midway through the run, when reactor R-1B was shutdown because of a leaky bottom closure, the accumulated solids were removed by reactor blowdown. Approximately 75 pounds of material were recovered following about 32 hours of coal processing. At the end of the run, reactor R-1A was cooled to about 400°F and then blowdown; 81 pounds of material was recovered of which 19 pounds were oily solids. This followed 19 hours of coal processing in R-1A.

The R-1A blowdown material was analyzed for ash and pyridine soluble material; then a representative sample was washed with hot toluene, dried and the tests repeated. In addition, X-Ray diffraction and sieve analyses were made on the toluene washed and dried solids along with X-Ray fluorescence analysis of the ash. The results of these test are shown in Table 2.

In addition to concluding the PDU test program, Run M-39 also ended the study of dissolver solids buildup. A summary of the findings for the six runs starting with L-3 is presented in Table 3. Results of the study are inconclusive; however, three general observations can be made: (1) particle agglomeration or crystal growth seems to be a random function as illustrated by the particle size variation in the sieve analyses for the reactor solids of Runs M-35 and M-36, which were made at similar operating conditions for approximately equal periods of coal processing time using Velve lignite. (2) Reactor deposits apparently result via two mechanisms: settling of clay minerals such as feldspar and quartz along with large particles of pyridine insoluble organic materials, and/or formation and settling of calcium carbonate crystals, such as calcite and dolomite. It appears that when processing Montana subbituminous coal that little calcium carbonate is formed and that most reactor solids result from the deposition of clay minerals and unconverted coal. In processing North Dakota lignite, calcium carbonate in addition to the clay minerals and unconverted coal deposits are present in the reactor solids. (3) For Zap lignite it appears that the level of total solids remaining in the reactor after solvent flushing following shutdown increases rapidly at first and

then levels off and remains relatively constant at about 29 or 30 pounds per cubic foot of reactor volume (See Figure 2). Similar curves are presented for the accumulated ash and pyridine indoluble material although the curve for the ash does not level off but rather shows a slight increasing slope over the time span studied.

Table 1
Process Development Unit Run M-39

Yield Period	M-39A	L-1K	L-2D
Reactor Used	R-1A	R-1A	R-1A
LHSV	1.39	1.40	1.38
GHSV	399	341	320
Solvent/MAF Coal Ratio	2.94	2.91	3.14
Mol Percent H ₂ in Feed Gas	99.6	52.5	62.9
Temperatures, °F			
Preheater Outlet, E-1	766	757	747
Reactor 3 ft	774	758	752
Reactor 7 ft	794	774	809
Reactor 11 ft	804	773	793
Reactor 15 ft	771	804	808
Max Reaction	804	804	809
Vacuum Flash Ovhd	507	504	503
Dissolver Pressure, psig	2500	2500	2450
Solvent Recycle, %			
Actual	95.6	86.4	93.1
Potential	102.8	107.6	100.0
Yields, Wt % MAF Lignite			
Net Gas	16.8	26.9	23.6
Net Liquid	54.2	65.8	58.6
(Dist Oil)	(8.4)	(22.2)	(-0.1)
(SRL)	(45.8)	(43.6)	(58.7)
Net Water & Ash	11.1	-3.5	0.7
Unconverted Coal	17.9	10.8	17.1
Overall Material Balance, %	98.5	99.3	99.9

Table 2
Process Development Unit Run M-39

Reactor R-1A Blowdown Material Analyses

Ash and Pyridine Extraction Analysis

	Raw Sample	Toluene-Washed Sample
Pyridine Solubles	88.68	2.29
Pyridine Insolubles	7.07	67.23
Ash	4.25	30.48
Total	100.00	100.00

X-Ray Diffraction Pattern Analysis

Sample: Toluene Washed R-1A Solids

Calcite - most abundant
Quartz - 1/5 as abundant as calcite
Dolomite - about 1/10 as abundant as calcite

X-Ray Fluorescence Analysis of Ash, Wt %

Sample:	Reactor Solids	*Feed Coal
Loss on Ignition @ 800°C	-	0.4
SiO ₂	16.9	11.2
Al ₂ O ₃	7.6	12.6
Fe ₂ O ₃	3.1	16.1
TiO ₂	0.3	0.5
P ₂ O ₅	1.2	0.0
CaO	53.2	17.8
MgO	9.9	10.0
Na ₂ O	1.7	9.5
K ₂ O	0.3	0.5
SO ₃	5.8	21.4
Total	100.0	100.0

Sieve Analysis

U.S. Screen Sieve Mesh	Total Percentages			
	On		Passing	
	Feed Coal	Reactor Solids	Feed Coal	Reactor Solids
65	0.7	6.9	99.3	93.1
100	2.8	38.0	97.2	62.0
140	9.8	67.0	90.2	33.0
170	26.9	80.7	73.1	19.3
230	51.3	91.8	48.7	8.2
325	78.6	96.0	21.4	4.0
Pan	100.0	100.0	0.0	0.0

*Determined by Atomic Absorption and colorametric methods.

Table 3
Process Development Unit Runs L-3, M-34, M-35, M-36, M-38 and M-39
Reactor Solids

Run	L-3	M-34	M-35	M-36	M-38	M-39
Hours of Coal processing before reactor blowdown	61.4	72.9	96.7	99.0	79.3	19
Pounds of MF Coal processed	2015	2034	2925	2992	2752	621
Source of Feed Coal	Zap	Gascoyne	Velva	Velva	Decker & Rosebud	Zap
Pounds of reactor solids recovered	41	85	66	76	24.6	19.5
Pounds of Toluene-washed solids (calculated)	39.6	66.5	34.7	57.7	4.6	11.3
Analysis of Toluene-washed solids, Wt %						
Pyridine Solubles	-	3.3	1.9	2.8	2.2	2.3
Pyridine Insolubles	-	45.8	40.6	41.4	51.4	67.2
Ash	12.35	50.9	57.5	55.8	46.4	30.5
Sieve Analysis - Total percentage on screen						
U.S. Screen Sieve						
Mesh						
65	82.6	29.3	1.1	39.2	14.5	6.9
100	95.4	71.5	17.9	76.4	40.1	38.0
140	98.3	89.3	61.7	90.4	56.5	67.0
170	98.8	95.2	81.5	95.5	62.4	80.7
230	99.3	99.3	97.3	99.1	74.6	91.8
325	99.5	99.8	99.4	99.6	88.1	96.0
Pan	100.0	100.0	100.0	100.0	100.0	100.0
Particle density of washed solids, lbs/cu. ft.	101	138	160	140	-	-
Bulk density of washed solids, lbs/cu. ft.	50.3	60.7	63.5	65.2	51.7	42.3
X-Ray Diffraction Pattern Analysis of toluene washed reactor solids						
Most abundant mineral	Calcite	Calcite	Calcite	Calcite	Quartz	Calcite
Second most abundant mineral	Dolomite	Quartz	Dolomite	Feldspar	Calcite	Quartz

FIGURE I
Process Development Unit Run M-39
Liquefaction Yields
Versus
Mol Percent Hydrogen in Charge Gas

FIGURE 2
Reactor Solids Accumulations
Runs L-2, L-3 and M-39
Zap Lignite

RUN CONDITIONS

RUN NO. M-39A

DATE MADE 3/23/78

TEMPERATURE °F

PREHEATER E-I
SAND BATH 789
INLET 262
OUTLET 766

DISSOLVERS
R-1A (1) 774
(2) 794
(3) 804
(4) 771
R-1B (1) NIU
(2)
(3)
(4)

REACTOR PRODUCT SEPARATORS
S-1 564
S-2 52
S-3 467
S-4A 72
S-4B 70

PREHEATER E-II
DOWTHERM 668
INLET 509
OUTLET 637

VACUUM FLASH F-I
OVERHEAD VAPOR 507
UPPER WALL 674
LOWER WALL 678
DOWNCOMER 411
CONDENSATE
ACCUMULATOR, S-7 76

PREHEATER E-13
DOWTHERM 425
INLET 70
OUTLET 185

LIGHT ENDS COLUMN, F-2
REBOILER NIU
LOWER SECTION
FEED SECTION
OVERHEAD
S-8 NIU
S-9 62

SOLVENT COLUMN, F-3
REBOILER 218
LOWER SECTION 232
FEED SECTION 204
OVERHEAD 181

HEAVY ENDS COLUMN, F-4
REBOILER NIU
LOWER SECTION
FEED SECTION
OVERHEAD

VACUUM FLASH, F-O
OVERHEAD 272
BOTTOMS

PRECIPITATION TOWER, V-8
ZONE (1) 342
(2) 388
(3) 431

RECOVERY TOWER V-9
REBOILER 503
OVERHEAD 231

PRESSURE, psig

SEPARATORS S-1 and S-2
2500

SEPARATOR S-3
200

SEPARATORS S-4A and S-4B
60

VACUUM FLASH, F-1
17 Torr

LIGHT ENDS COLUMN, F-2
NIU

SOLVENT COLUMN, F-3
1

HEAVY ENDS COLUMN, F-4
NIU

VACUUM FLASH, F-O
17 Torr

SETTLING TOWER, V-8
240

RECOVERY TOWER, V-7
1

	Start	End	Run No: M-39A
Date of Run	3/23/78	3/23/78	Units: lbs/hr., °F, psig
Time of Run	0430	1630	Prep. By: BCL & GGB
Length of Run	12	hours	Updated: 5/25/78
Hours on Coal at End of Y.P.	51.1		

MASS RATES AND RUN CONDITIONS

224.45 TOTAL

OPEN

CLOSED

TOTAL 224.45

Numbers in parentheses are not included in the totals.

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

Normalized with V-8 Btms Composition Calculated followed by forced Carbon Balance
Revised: 5/25/78

Run No: M-39A
Date: 3/23/78
Time: 0430-1630

SUMMARY

Temperature: 766°F
Pressure: 2500 psig
LHSV: 1.39
GHSV: 399

lbs/hr.

Quantitative	STREAM DESCRIPTION	Total In	Total Out	Net Yield		Wt % MAF Coal	
	SAMPLE POINT						
	Hydrogen - H ₂	2.99	2.42	-0.57			
	Carbon Monoxide - CO		0.21	0.21			
	Carbon Dioxide - CO ₂		3.14	3.14			
	Hydrogen Sulfide - H ₂ S		0.19	0.19			
	Methane - CH ₄		1.19	1.19	5.01	16.74	
	Ethane - C ₂ H ₆		0.56	0.56			
	Propane - C ₃ H ₈		0.41	0.41			
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.17	0.05	-0.12			
	Ammonia - NH ₃						
	Light Oil - (0-100°C @ 1.6 Torr)	113.23	116.42	3.19			
	Solvent - (100-230°C @ 1.6 Torr)	60.29	59.15	-1.14	16.22	54.21	
	Heavy Oil - (230-255°C @ 1.6 Torr)	1.15	1.61	0.46			
	SRL		13.71	13.71			
	Coal (MAF)	29.92	5.36	-24.56		-82.08	
	Ash	3.03	2.82	-0.21		-0.70	
	Water	13.67	17.21	3.54		11.83	
	Phenol						
	TOTAL	224.45	224.45	-0-			

Elemental	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)	Ash	
	177.69	21.34	0.57	1.15	20.67	3.03	
	177.69	21.88	0.44	1.26	20.36	2.82	
	-0-	0.54	-0.13	0.11	-0.31	-0.21	
	TOTAL	224.45	224.45	-0-			

PROJECT LIGNITE
MEASURED & ADJUSTED MATERIAL BALANCE
Revised: 5/25/78

Run No: M-39A
Date: 3/23/78
Time: 0430-1630

MATERIAL IN

Temperature: 766°F
Pressure: 2500 psig
LHSV: 1.39
GHSV: 399

lbs/hr.

Quantitative	STREAM DESCRIPTION	Feed Gas	Lignite	Feed Solvent	Deashing Solvent	Total In
	SAMPLE POINT	Sx-21	Sx-1	Sx-14	Sx-40	
	Hydrogen - H ₂	2.99				2.99
	Carbon Monoxide - CO					
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂	0.17				0.17
	Ammonia - NH ₃					
	Light Oil - (0-100°C @ 1.6 Torr)			28.32	84.91	113.23
	Solvent - (100-230°C @ 1.6 Torr)			58.47	1.82	60.29
	Heavy Oil - (230-255°C @ 1.6 Torr)			1.15		1.15
	SRL					
	Coal (MAF)		29.92			29.92
	Ash		3.03			3.03
	Water		13.29	0.38		13.67
	Phenol					
	TOTAL	3.16	46.24	88.32	86.73	224.45

Elemental	Carbon		21.41	77.16	79.12	177.69
	Hydrogen	2.99	2.70	8.10	7.55	21.34
	Nitrogen	0.17	0.22	0.18		0.57
	Sulfur		0.13	1.00	0.02	1.15
	Oxygen (by diff)		18.75	1.88	0.04	20.67
	Ash		3.03			3.03
	TOTAL	3.16	46.24	88.32	86.73	224.45

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

Normalized with V-8 Btms Composition Calculated followed by Forced Carbon Balance
Revised: 5/25/78

Run No: M-39A
Date: 3/23/78
Time: 0430-1630

MATERIAL OUT

Temperature: 766°F
Pressure: 2500 psig
LHSV: 1.39
GHSV: 399

Time: 0430-1630		lbs/hr.			GHSV: 399		
Quantitative	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
	Hydrogen - H ₂	2.27	0.14				0.01
	Carbon Monoxide - CO	0.20	0.01				
	Carbon Dioxide - CO ₂	2.37	0.66				0.11
	Hydrogen Sulfide - H ₂ S	0.11	0.08				
	Methane - CH ₄	1.04	0.14				0.01
	Ethane - C ₂ H ₆	0.39	0.15				0.02
	Propane - C ₃ H ₈	0.23	0.15				0.03
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.05					
	Ammonia - NH ₃						
	Light Oil - (0-100°C @ 1.6 Torr)			2.72		1.70	
	Solvent - (100-230°C @ 1.6 Torr)			1.07		0.06	
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Coal (MAF)						
	Ash						
	Water			16.68			
	Phenol						
	TOTAL	6.66	1.33	20.47	Negl	1.76	0.18

Elemental	Carbon	2.00	0.52	3.28		1.48	0.08
	Hydrogen	2.66	0.24	2.25		0.24	0.02
	Nitrogen	0.06	0.01	0.01			
	Sulfur	0.10	0.08	0.02		0.01	
	Oxygen (by diff)	1.84	0.48	14.91		0.03	0.08
	Ash						
		TOTAL	6.66	1.33	20.47	Negl	1.76

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

Normalized with V-8 Btms Composition Calculated followed by Forced Carbon Balance

Revised: 5/25/78

Run No: M-39A
Date: 3/23/78
Time: 0430-1630

MATERIAL OUT

Temperature: 766°F
Pressure: 2500 psig
LHSV: 1.39
GHSV: 399

lbs/hr.

STREAM DESCRIPTION	Cold Trap	V-8 Btms	F-1 Btms	V-9 Ovhd	←Recycle S-7 Btms	Solvent → IEC Btms
	Sx-33	Sx-27	Sx-2	Sx-28	Sx-39	Sx-12
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃						
Light Oil - (0-100°C @ 1.6 Torr)	0.12	4.32		80.59	16.99	9.98
Solvent - (100-230°C @ 1.6 Torr)	0.01	0.32	0.95	1.39	50.91	4.44
Heavy Oil - (230-255°C @ 1.6 Torr)					1.36	0.25
SRL		0.09	13.62			
Cool (MAF)		5.32	0.04			
Ash		2.67	0.15			
Water					0.53	
Phenol						
TOTAL	0.13	12.72	14.76	81.98	69.79	14.67

Carbon	0.11	8.11	12.95	74.80	61.76	12.60
Hydrogen	0.01	0.63	0.91	7.14	6.32	1.46
Nitrogen		0.04	0.13		0.16	0.03
Sulfur		0.03	0.10	0.02	0.82	0.08
Oxygen (by diff)	0.01	1.24	0.52	0.02	0.73	0.50
Ash		2.67	0.15			
TOTAL	0.13	12.72	14.76	81.98	69.79	14.67

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE

Normalized with V-8 Btms Composition. Calculated followed by a Forced Carbon Balance
Revised: 5/25/78

Run No: M-39A
Date: 3/23/78
Time: 0430-1630

MATERIAL OUT

lbs/hr.

Temperature: 766°F
Pressure: 2500 psig
LHSV: 1.39
GHSV: 399

Quantitative	STREAM DESCRIPTION					Total Out
	SAMPLE POINT					
	Hydrogen - H ₂					2.42
	Carbon Monoxide - CO					0.21
	Carbon Dioxide - CO ₂					3.14
	Hydrogen Sulfide - H ₂ S					0.19
	Methane - CH ₄					1.19
	Ethane - C ₂ H ₆					0.56
	Propane - C ₃ H ₈					0.41
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.05
	Ammonia - NH ₃					
	Light Oil - (0-100°C @ 1.6 Torr)					116.42
	Solvent - (100-230°C @ 1.6 Torr)					59.15
	Heavy Oil - (230-255°C @1.6 Torr)					1.61
	SRL					13.71
	Cool (MAF)					5.36
	Ash					2.82
	Water					17.21
	Phenol					
	TOTAL					224.45

Elemental	Carbon					177.69
	Hydrogen					21.88
	Nitrogen					0.44
	Sulfur					1.26
	Oxygen (by diff)					20.36
	Ash					2.82
	TOTAL					224.45

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/25/78

Run No: M-39A

Date: 3/23/78

Time: 0430-1630

WEIGHT PERCENT

	WEIGHT PERCENT					
	Lignite	F-1 Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
SAMPLE DESCRIPTION	Sx-1	Sx-2	Sx-12	Sx-14	Sx-16	Sx-16
SAMPLE POINT						
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)						
Light Oils - (0-100° C @ 1.6 Torr)			68.00	32.07	96.80	
Solvent - (100-230° C @ 1.6 Torr)		6.40	30.30	66.20	3.20	
Heavy Oil - (230-255° C @ 1.6 Torr)			1.70	1.30		
SRL		92.30				
Coal (MAF)	64.70	0.28				
Ash	6.56	1.02				
Water	28.74			0.43		100
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)		99.72				
Melting Point °F						
Specific Gravity 60/60				1.003		
Viscosity, cp						

	WEIGHT PERCENT					
	Lignite	F-1 Btms	LEC Btms	Feed Solvent	S-8 Oil	S-8 Water
Carbon	46.29	87.74	85.88	87.37	83.92	
Hydrogen	5.83	6.15	9.98	9.17	13.98	11.11
Nitrogen	0.48	0.87	0.22	0.20	0.09	
Sulfur	0.29	0.67	0.52	1.13	0.25	
Oxygen (by diff)	40.55	3.55	3.40	2.13	1.76	88.89
Ash	6.56	1.02				
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	2	2	2	2	2	2

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/25/78

Run No: M-39A

Date: 3/23/78

Time: 0430-1630

WEIGHT PERCENT

Quantitative	SAMPLE DESCRIPTION	S-8 Total	Product Gas	Feed Gas	S-4B Oil	S-4B Water	S-4B Total
	SAMPLE POINT	Sx-16	Sx-19	Sx-21	*Sx-23	Sx-23	Sx-23
	Hydrogen - H ₂		34.10	94.68			
	Carbon Monoxide - CO		2.97				
	Carbon Dioxide - CO ₂		35.65				
	Hydrogen Sulfide - H ₂ S		1.64				
	Methane - CH ₄		15.59				
	Ethane - C ₂ H ₆		5.79				
	Propane - C ₃ H ₈		3.39				
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂		0.81	5.32			
	Ammonia - NH ₃ (PPM)		0.06			0.01	0.01
	Light Oils - (0-100°C @ 1.6 Torr)	96.80			71.22		9.69
	Solvent - (100-230°C @ 1.6 Torr)	3.20			28.10		3.82
	Heavy Oil - (230-255°C @1.6 Torr)						
	SRL						
	Coal (MAF)						
	Ash						
	Water				0.68	99.99	86.48
	Phenol						
	TOTAL	100	100	100	100	100	100
	Pyridine Solubles (Wt % Ash free)						
	Melting Point °F						
	Specific Gravity 60/60				0.923		
	Viscosity, cp						

Elemental	Carbon	83.92	30.08		85.96		11.69
	Hydrogen	13.98	39.89	94.68	10.38	11.11	11.01
	Nitrogen	0.09	0.86	5.32	0.17	0.01	0.03
	Sulfur	0.25	1.54		0.56		0.08
	Oxygen (by diff)	1.76	27.63		2.93	88.88	77.19
	Ash						
	TOTAL	100	100	100	100	100	100
	Number of Sample Avg.	-	2	2	2	2	-

*Insufficient oil sample. Assumed to be the same as the S-4A oil.

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/25/78

Run No: M-39A

Date: 3/23/78

Time: 0430-1630

WEIGHT PERCENT

	WEIGHT PERCENT					
	V-8 Btms	V-9 Ovhd	S-4A Ovhd	J-1 Discharge	Cold Trap	S-7 Btms
SAMPLE DESCRIPTION						
SAMPLE POINT	Sx-27	Sx-28	Sx-30	Sx-32	Sx-33	Sx-34/39
Hydrogen - H ₂			10.58	5.31		
Carbon Monoxide - CO			0.78			
Carbon Dioxide - CO ₂			49.62	60.66		
Hydrogen Sulfide - H ₂ S			6.06			
Methane - CH ₄			10.32	6.93		
Ethane - C ₂ H ₆			10.92	12.39		
Propane - C ₃ H ₈			10.97	14.66		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂			0.55			
Ammonia - NH ₃ (PPM)			0.20			
Light Oils - (0-100°C @ 1.6 Torr)	59.83	98.30			90.68	24.34
Solvent - (100-230°C @ 1.6 Torr)	30.64	1.70			4.70	72.95
Heavy Oil - (230-255°C @ 1.6 Torr)						1.95
SRL	9.24					
Coal (MAF)	0.00					
Ash	0.29					
Water					4.62	0.76
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60						1.008
Viscosity, cp						

Elemental	Carbon	90.23	91.24	39.32	43.68	82.86	88.49
	Hydrogen	8.29	8.71	17.73	12.20	12.19	9.05
	Nitrogen	0.11		0.71		0.08	0.23
	Sulfur	0.71	0.02	5.70		0.15	1.18
	Oxygen (by diff)	0.37	0.03	36.54	44.12	4.72	1.05
	Ash	0.29					
	TOTAL	100	100	100	100	100	100
	Number of Sample Avg.	2	2	2	2	1	4

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/25/78

Run No: M-39A

Date: 3/23/78

Time: 0430-1630

WEIGHT PERCENT

	WEIGHT PERCENT					
	S-4A Oil	S-4A Water	S-4A Total	S-7 Btms	Deashing Solvent	V-8 Btms
SAMPLE DESCRIPTION						
SAMPLE POINT	Sx-37	Sx-37	Sx-37	Sx-34/39	Sx-40	Calc
Hydrogen - H ₂						
Carbon Monoxide - CO						
Carbon Dioxide - CO ₂						
Hydrogen Sulfide - H ₂ S						
Methane - CH ₄						
Ethane - C ₂ H ₆						
Propane - C ₃ H ₈						
Butane - C ₄ H ₁₀						
Nitrogen - N ₂						
Ammonia - NH ₃ (PPM)		0.06				
Light Oils - (0-100°C @ 1.6 Torr)	71.22		69.07	24.34	97.90	33.96
Solvent - (100-230°C @ 1.6 Torr)	28.10		27.25	72.95	2.10	2.52
Heavy Oil - (230-255°C @ 1.6 Torr)				1.95		
SRL						0.71
Coal (MAF)						41.82
Ash						20.99
Water	0.68	99.94	3.68	0.76		
Phenol						
TOTAL	100	100	100	100	100	100
Pyridine Solubles (Wt % Ash free)						
Melting Point °F						
Specific Gravity 60/60	0.923					
Viscosity, cp						

	WEIGHT PERCENT					
	S-4A Oil	S-4A Water	S-4A Total	S-7 Btms	Deashing Solvent	V-8 Btms
Carbon	85.96		83.36	88.49	91.22	63.75
Hydrogen	10.38	11.11	10.41	9.05	8.71	4.93
Nitrogen	0.17	0.05	0.17	0.23		0.33
Sulfur	0.56		0.54	1.18	0.02	0.23
Oxygen (by diff)	2.93	88.84	5.52	1.05	0.05	9.77
Ash						20.99
TOTAL	100	100	100	100	100	100
Number of Sample Avg.	2	2	2	4	2	-

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/25/78

Run No: M-39A

Date: 3/23/78

Time: 0430-1630

WEIGHT PERCENT

Quantitative	SAMPLE DESCRIPTION	S-4B Btms				
	SAMPLE POINT	Calc				
	Hydrogen - H ₂					
	Carbon Monoxide - CO					
	Carbon Dioxide - CO ₂					
	Hydrogen Sulfide - H ₂ S					
	Methane - CH ₄					
	Ethane - C ₂ H ₆					
	Propane - C ₃ H ₈					
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					
	Ammonia - NH ₃ (PPM)	0.01				
	Light Oils - (0-100° C @ 1.6 Torr)	13.29				
	Solvent - (100-230° C @ 1.6 Torr)	5.24				
	Heavy Oil - (230-255° C @ 1.6 Torr)					
	SRL					
	Coal (MAF)					
	Ash					
	Water	81.46				
	Phenol					
	TOTAL	100				
Elemental	Pyridine Solubles (Wt % Ash free)					
	Melting Point °F					
	Specific Gravity 60/60					
	Viscosity, cp					

Elemental	Carbon	16.04				
	Hydrogen	10.98				
	Nitrogen	0.04				
	Sulfur	0.10				
	Oxygen (by diff)	72.84				
	Ash					
	TOTAL	100				
Elemental	Number of Sample Avg.	-				

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 5/25/78

Run No: M-39A

Date: 3/23/78

Time: 0430-1630

MOLE PERCENT

SAMPLE DESCRIPTION	Product Gas	Feed Gas	S-4A Ovhd	J-1 Discharge		
	Sx-19	Sx-21	Sx-30	Sx-32		
Hydrogen - H ₂	88.38	99.60	66.85	50.88		
Carbon Monoxide - CO	0.55		0.35			
Carbon Dioxide - CO ₂	4.20		14.25	26.44		
Hydrogen Sulfide - H ₂ S	0.25		2.25			
Methane - CH ₄	5.05		8.15	8.37		
Ethane - C ₂ H ₆	1.00		4.60	7.92		
Propane - C ₃ H ₈	0.40		3.15	6.39		
Butane - C ₄ H ₁₀						
Nitrogen - N ₂	0.15	0.40	0.25			
Ammonia - NH ₃ (PPM)	0.02		0.15			
Light Oils - (0-100°C @ 1.6 Torr)						
Solvent - (100-230°C @ 1.6 Torr)						
Heavy Oil - (230-255°C @ 1.6 Torr)						
SRL						
Coal (MAF)						
Ash						
Water						
Phenol						
TOTAL	100	100	100	100		
Calc Mole Wt.	5.18	2.10	12.64	19.18		
Melting Point °F						
Specific Gravity 60/60						
Viscosity, cp						

Carbon						
Hydrogen						
Nitrogen						
Sulfur						
Oxygen (by diff)						
Ash						
TOTAL						
Number of Sample Avg.	2	2	2	2		

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-39A
DATE: 3/23/78
TIME: 0430-1630

SAMPLE DESCRIPTION	F-1 Btms	LEC Btms	Feed Solv.	S-8 Oil	V-9 Ovhd
SAMPLE POINT	Sx-2	Sx-12	Sx-14	Sx-16	Sx-28
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5	5		
IBP		20	21.2		
5%		38.2	66		
10%		51	77		
20%		68.2	96.2		
30%		77.3	115		
40%		86.2	138.2		
50%		95	161.8		
60%		108.6	175.6		
70%		129.2	184.6		
80%		159.3	193.2		
90%		184.5	207.3		
95%		198.6	230		
DRY PT.		198.6	239.3		
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR				736	732
IBP				34	112
5%				50	112
10%				59	112
20%				68	113
30%				78	113
40%				87	113
50%				97	114
60%				105	114
70%				112	114
80%				118	114
90%				130	114
95%					116
DRY PT.				152	126
ASPHALTENE TEST					
WT % ASH	1.02				
WT % UNCONVERTED COAL	0.28				
WT % PRE-ASPHALTENES	27.88				
WT % ASPHALTENES	38.34				
WT % MALTENES & DIST. OIL	32.48				
TOTAL	100.00				
SAYBOLT VISCOSITY, SSU					
100° F		8.9	10.2		
210° F		7.6	8.3		
MISCELLANEOUS					
KF H ₂ O WT% (Raw Sx)			0.43		
INFRARED RATIO		0.20	0.24		
SP. GRAV. 60/60 F			1.003		

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-39A
DATE: 3/23/78
TIME: 0430-1630

SAMPLE DESCRIPTION	V-9 Btms	Cold Trap	S-7 Btms	S-4A Oil	Deash.Solv.
SAMPLE POINT	Sx-29	Sx-33	Sx-34/39	Sx-37	Sx-40
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR	5		5		
IBP	95		18.3		
5%	166		79.1		
10%	175		89.5		
20%	184		111.5		
30%	190		133.7		
40%	198		155.9		
50%	210		170.1		
60%	220		178.4		
70%	240		185.2		
80%	276		193.3		
90%			202.8		
95%			224.4		
DRY PT.			241.1		
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR		735		729	748
IBP		83		74	112
5%		87		89	113
10%		87		143	114
20%		104		182	114
30%		120		200	114
40%		128		212	114
50%		134		230	114
60%		142		244	114
70%		155		271	114
80%		186		305	114
90%		200		342	114
95%		221		350	118
DRY PT.		222		352	126
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F			10.7		
210° F			8.4		
MISCELLANEOUS					
KF H ₂ O WT% (Row Sx)		4.62	0.76	0.68	
INFRARED RATIO			0.25		
SP. GRAV. 60/60 F			1.008		

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
SAMPLE ANALYSES
RUN NO: M-39A
DATE: 3/23/78
TIME: 0430-1630

SAMPLE DESCRIPTION		F-0 Btms			
SAMPLE POINT		Sx-41			
ASTM D-1160 DISTILLATION DATA					
PRESSURE, TORR		5			
VOLUME PERCENT → 5% 10% 20% 30% 40% 50% 60% 70% 80% 90% → 95%	IBP	112			
		134			
		179			
		189			
		197			
		206			
		217			
		255			
TEMPERATURE, °C					
DRY PT.					
ASTM D-86 DISTILLATION DATA					
BAROMETRIC PRESSURE, TORR					
VOLUME PERCENT → 5% 10% 20% 30% 40% 50% 60% 70% 80% 90% → 95%	IBP				
TEMPERATURE, °C					
DRY PT.					
ASPHALTENE TEST					
WT % ASH					
WT % UNCONVERTED COAL					
WT % PRE-ASPHALTENES					
WT % ASPHALTENES					
WT % MALTENES & DIST. OIL					
TOTAL					
SAYBOLT VISCOSITY, SSU					
100° F					
210° F					
MISCELLANEOUS					
KF H ₂ O WT% (Raw Sx)					
INFRARED RATIO					
SP. GRAV. 60/60 F					

PREASPHALTENES - PYRIDINE SOLUBLE, BENZENE INSOLUBLE MATERIAL
ASPHALTENES- BENZENE SOLUBLE, N-PENTANE INSOLUBLE MATERIAL
MALTENES- N-PENTANE SOLUBLE MATERIAL NOT RECOVERABLE BY STANDARD DISTILLATION PROCEDURES
DISTILLABLE OIL - RECOVERABLE BY DISTILLATION.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/25/78

Run No: M-39A
Date: 3/23/78
Time: 0430-1630

MATERIAL OUT

Temperature: 766°F
Pressure: 2500 psig
LHSV : 1.39
GHSV: 399

lbs/hr.

Quantitative	STREAM DESCRIPTION	Product Gas	S-4A Ovhd	S-4B Btms	S-8 Ovhd	S-8 Btms	J-1 Exhaust
	SAMPLE POINT	Sx-19	Sx-30	Sx-23	Sx-44	Sx-16	Sx-32
	Hydrogen - H ₂	2.14	0.13				0.01
	Carbon Monoxide - CO	0.19	0.01				
	Carbon Dioxide - CO ₂	2.24	0.62				0.11
	Hydrogen Sulfide - H ₂ S	0.10	0.07				
	Methane - CH ₄	0.98	0.13				0.01
	Ethane - C ₂ H ₆	0.36	0.14				0.02
	Propane - C ₃ H ₈	0.21	0.14				0.03
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂	0.05	0.01				
	Ammonia - NH ₃						
	Light Oil - (0-100°C @ 1.6 Torr)			1.87		1.61	
	Solvent - (100-230°C @ 1.6 Torr)			0.74		0.05	
	Heavy Oil - (230-255°C @ 1.6 Torr)						
	SRL						
	Cool (MAF)						
	Ash						
	Water			16.67			
	Phenol						
		TOTAL	6.27	1.25	19.28	Negl.	1.66

Elemental	Carbon	1.89	0.49	2.25		1.39	0.08
	Hydrogen	2.50	0.22	2.12		0.23	0.02
	Nitrogen	0.05	0.01	0.01			
	Sulfur	0.10	0.07	0.02		0.01	
	Oxygen (by diff)	1.73	0.46	14.88		0.03	0.08
	Ash						
		TOTAL	6.27	1.25	19.28	Negl	1.66

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/25/78

Run No: M-39A
Date: 3/23/78
Time: 0430-1630

MATERIAL OUT

Temperature: 766°F
Pressure: 2500 psig
LHSV: 1.39
GHSV: 399

lbs/hr.

Quantitative	STREAM DESCRIPTION	Cold Trap	V-8 Btms	F-1 Btms	V-9 Ovhd	←Recycle S-7 Btms	Solvent → LEC Btms
	SAMPLE POINT	Sx-33	Sx-27	Sx-2	Sx-28	Sx-39	Sx-12
	Hydrogen - H ₂						
	Carbon Monoxide - CO						
	Carbon Dioxide - CO ₂						
	Hydrogen Sulfide - H ₂ S						
	Methane - CH ₄						
	Ethane - C ₂ H ₆						
	Propane - C ₃ H ₈						
	Butane - C ₄ H ₁₀						
	Nitrogen - N ₂						
	Ammonia - NH ₃						
	Light Oil - (0-100°C @ 1.6 Torr)	0.11	7.17		80.59	16.99	9.98
	Solvent - (100-230°C @ 1.6 Torr)	0.01	3.67	0.89	1.39	50.91	4.44
	Heavy Oil - (230-255°C @ 1.6 Torr)					1.36	0.25
	SRL		1.11	12.83			
	Cool (MAF)			0.04			
	Ash		0.03	0.14			
	Water					0.53	
	Phenol						
	TOTAL	0.12	11.98	13.90	81.98	69.79	14.67

Elemental							
	Carbon	0.10	10.81	12.20	74.80	61.76	12.60
	Hydrogen	0.01	0.99	0.85	7.14	6.32	1.46
	Nitrogen		0.01	0.12		0.16	0.03
	Sulfur		0.09	0.09	0.02	0.82	0.08
	Oxygen (by diff)	0.01	0.05	0.64	0.02	0.73	0.50
	Ash		0.03				
	TOTAL	0.12	11.98	13.90	81.98	69.79	14.67

PROJECT LIGNITE
MEASURED MATERIAL BALANCE
Revised: 5/25/78

Run No: M-39A
Date: 3/23/78
Time: 0430-1630

MATERIAL OUT

Temperature: 766°F
Pressure: 2500 psig
LHSV : 1.39
GHSV: 399

lbs/hr.

Quantitative	STREAM DESCRIPTION					Total Out
	SAMPLE POINT					
	Hydrogen - H ₂					2.28
	Carbon Monoxide - CO					0.20
	Carbon Dioxide - CO ₂					2.97
	Hydrogen Sulfide - H ₂ S					0.17
	Methane - CH ₄					1.12
	Ethane - C ₂ H ₆					0.52
	Propane - C ₃ H ₈					0.38
	Butane - C ₄ H ₁₀					
	Nitrogen - N ₂					0.06
	Ammonia - NH ₃					
	Light Oil - (0-100°C @ 1.6 Torr)					118.32
	Solvent - (100-230°C @ 1.6 Torr)					62.10
	Heavy Oil - (230-255°C @1.6 Torr)					1.61
	SRL					13.94
	Coal (MAF)					0.04
	Ash					0.17
	Water					17.20
	Phenol					
	TOTAL					221.08

Elemental	Carbon					178.37
	Hydrogen					21.86
	Nitrogen					0.39
	Sulfur					1.30
	Oxygen (by diff)					19.13
	Ash					0.03
	TOTAL					221.08