FE-1224-58
Distribution Category UC-90d

PROJECT LIGNITE

QUARTERLY TECHNICAL PROGRESS REPORT NO. 8

JANUARY, FEBRUARY AND MARCH 1976

Donald E. Severson, Principal Investigator
Engineering Experiment Station
University of North Dakota
Grand Forks, North Dakota 58202

July 14, 1976

PREPARED FOR THE UNITED STATES
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

Under Contract No. E(49-18)-1224
UND Account No. 4567

CONTENTS

I. OBJECTIVE 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 is to make recommendations for a refinery based on lignite as feedstock, and the experimental portion of the program is 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 bench-scale work have been carried out and a continuous process development unit is being operated to demonstrate the feasibility of liquefaction of lignite. Economic studies have been carried out by an economist attached to the program, and under a project subcontract the Department of Chemistry of UND has developed information on catalytic hydrogenation of solvent refined lignite.

The continuous process currently under development is the two stage conversion of lignite to fuel liquids with solvent refined lignite as an intermediate 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 will be the catalytic hydrogenation to premium liquid fuels of the solvent refined lignite produced in the first stage.

II. SUMMARY

A. PDU OPERATIONS

Following the replacement of type 316 stainless steel with Incoloy 800 in the high-pressure, high-temperature area of the Process Development Unit liquefaction operations were again initiated, and five runs were started during February and March. Initially, solvent recovery was low, and the major effort was devoted to a search for conditions that would improve recycle solvent recovery.

Changes in operating conditions and more attention to control of operations resulted in reductions of solvent losses from about 22 percent of the solvent charged to about 6 percent. Specifically, the pressure was increased from 1500 to 2500 psig, the reactor volume was halved by by-passing one of the two dissolvers, the preheater outlet temperature was kept up to the desired point of 750°F, the reactor temperatures were carefully monitored and the adiabatic heaters adjusted to minimize the effect of heat losses, the syngas feed rate was maintained at the maximum output rate of one compressor, the vacuum flash column was maintained at 600°F. or more, all vacuum condensate was recycled as solvent, and the bottoms from the distillation of light oils was recycled as solvent. Preliminary indications are that there are still some recoverable solvent fractions in the PDU products, and more efforts will be made to recover them for recycle.

Two adjustments proved impractical because they presented operability problems. Lowering the solvent to coal ratio resulted in difficulties both in charging the slurry and in pumping out the vacuum bottoms. Increasing the vacuum flash temperature caused coking in the vacuum flash column.

One run in the Solid-Liquid Separation section was terminated because of difficulties in feeding molten vacuum bottoms from the heated drum into the mixing circuit and because of diaphram failure in the pump for slurry circulation in the mixing circuit.

B. PDU MAINTENANCE AND CONSTRUCTION

After completion of installation of the Incoloy 800 preheater coil dissolvers, and associated piping, activities were concentrated on modifications, repairs and maintenance necessary to ensure operation of the liquefaction section of the PDU. Overhaul of compressors, replacement of valves, repair of leaks and removal of plugs in equipment were accomplished.

C. EXPERIMENTATION - PROJECT LIGNITE LABORATORY

Efforts in laboratory work were directed primarily at analytical support for the PDU operations. However, several liquefaction and deashing experiments were conducted.

Four batch autoclave runs made to check previous tests made on lignite after 70 weeks storage resulted in about the same conversions and liquid yields as the original tests, though gas production and water consumption were both slightly higher. During the course of the storage tests, there appeared to be no significant change in the liquefaction properties of lignite stored under water or in nitrogen, whereas the reactivity decreased somewhat for lignite stored in air.

One autoclave run was made using as liquefaction solvent a sample of the PDU vacuum condensate taken during Run M-5. This sample was selected because of its unusually high concentration of heavy oil which must have been coal derived. However, the ash content was later found to be almost two percent, so it must have been contaminated with feed slurry to the vacuum flash drum spilled over or entrained in the flash vapor. The reactor product proved difficult to filter, and a product work-up based on pyridine extraction indicated conversion and liquid yield to be low and gas yield to be high compared with liquefaction with the usual solvent.

In solvent deashing experiments, ash-containing SRL from a laboratory autoclave liquefaction test and undeashed SRC from Wilsonville, Alabama, were extracted with toluene at elevated temperature and pressure. Ash rejection was about 98 percent in both cases, and product recovery was 55 percent for the SRL and 71 percent for SRC. In this and previous experiments higher product recoveries have been realized from undeashed SRC than from undeashed SRL.

III. TECHNICAL PROGRESS

A. PDU OPERATIONS

1. General

Five runs were made with the liquefaction section of the PDU, M-1 through M-5. Each run included a series of yield or balance periods. Additionally, a shake-down run (S-1) using the solid-liquids separation system was attempted. Material balances and test conditions for yield periods M-1C, M-2A and M-2C are given in Parts 1, 2, and 3 of Appendix A. A sketch of the Solid-Liquid Separation section is shown in Figure 1. A summary table showing the operating conditions is on the next page.

The vacuum flash system was operated for several days prior to Run M-1 to prepare start-up solvent of the appropriate boiling range from the FS 120 carbon black feedstock. Approximately 180 gallons of process solvent and heavy oil was produced.

Operating conditions during the liquefaction experiments were modified in attempts to improve solvent recovery. Improvements were made, but the best recovery still indicated about a 6 percent loss of solvent which would be unsatisfactory for commercial operations. Operating problems encountered were usually related to major changes in operating variables.

The various runs are discussed in more detail in the following sections:

2. Area 03: Solid-Liquid Separation (Run S-1)

Run S-1 was a shakedown test using the solids-liquid separation unit (area 03) on March 1 - 3.

Three major modifications had been made to the unit prior to this attempt. The toluene-slurry mix tank had been replaced by a pressurized circulating and mixing loop; a small piston-type, positive displacement pump had been installed to charge F-1 vacuum bottoms to the system; and the ash removal system beneath the precipitation tower V-8 had been modified by altering the piping to provide direct downfall of solids through two pneumatically operated ball valves in a captive-volume, pressure-reducing system into a heated accumulator.

The planned operating conditions for the S-1 separations run were as follows: Liquefied F-1 vacuum bottoms from PDU Run M-1 was to be charged at 450^{o}F and 0.2 lbs/min to the pressurized mixing loop along with 0.1 gal/min of diluent (toluene). The mixing was to be conductedat 250^{o}F and 350 psig. Excess slurry in the mixing loop would automatically feed through a check valve into the precipitation tower, operating at 350^{o}F and 350 psig.

SUMMARY OF OPERATING CONDITIONS

Run No.	Yield Period	Preheater Outlet Temperature, °F.	Dissolver Pressure, psig	Vacuum Flash Temperature, °F.	Feed Rates LHSV	Feed Rates GHSV	Solvent/Raw Coal Ratio
M-1	A	693	1500	607	0.87	150	2.70/1
	B	716	"	602	0.99	152	1.98/1
	C	702	"	591	0.90	164	2.30/1
M-2	A	733	1500	547	0.87	159	1.80/1
	B	692	"	606	0.97	128	2.30/1
	C	698	"	610	0.66	161	1.70/1
M-3	A	729	2000	593	1.81	301*	2.30/1
	B	745	"	572	1.71	284	1.88/1
M-4	-	726	2500	603	1.43*	301*	2.23/1
M-5	A	754	2500	615	1.32	288	1.92/1
	B	744	"	575	1.43	293	1.78/1
	C	750	"	601	1.38	334	1.92/1
	D	742	"	598	1.39	277	1.65/1
	E	750*	"	590	1.43*	301*	-
	F	750*	"	586	1.63	387	1.90/1

* Nominal Values

Two major problems were experienced during the run. First, little or no vacuum bottoms feed was charged to the mixing loop because of solidification in an inadequately heated section of the "Verquad" check valve assembly on the charge pump. Second, the Viton diaphrams in the toluene-slurry circulating pump failed after only 34 hours of continuous operation.

Repairs and modifications were made to the equipment. The problem of melting and feeding the F-1 vacuum bottoms in the system should be reduced when the separation system is operated in conjunction with the liquefaction unit and the bottoms charged directly as produced. A flow diagram of the unit is shown in Figure 1 of Appendix A.

3. Run M-1

In liquefaction test M-1 the complete solubilization system up to but not including the solid-liquid separations system was operated continuously for five days (February 2-6). The nominal operating conditions were 750°F. preheater outlet temperature, 1500 psig process pressure, 50 lb/hr. of raw lignite charged, 3:1 solvent to MAF lignite ratio, liquid hourly space velocity 2.8, gas hourly space velocity 620, and gas inlet composition 50% CO - 50% H_2. The vacuum flash was operated at 600°F. The two dissolvers were used in series.

Three yield periods were obtained during the week's operation, two of which were of 12 hours duration. Average input rates were 54 lb/hr lignite, 16.7 lb/hr gas and 135 lb/hr solvent.

Solvent recovery was low, at about 82 to 85 percent of solvent charged. Some solvent appeared in the F-1 bottoms product and some in the water-oil mixture in the low pressure product separator. Some of the solvent may have been decomposed or converted to light oils during processing. Conversion of MAF lignite to gas, light oils and SRL was from 72 to 80 percent. Product gas composition and quantity corresponded closely to those assumed in the PDU design.

One yield period, M-1C, of this run was worked up completely. The results are given in Part 1 of Appendix A.

4. Run M-2

Run M-2 was commenced at the same set of operating conditions as M-1, but with improved startup procedures to assure a smooth approach to steady state. After one yield period, because of continued low solvent recovery, the preheater outlet temperature was lowered from 750°F. to 700°F. without changes in other variables for the balance of the run.

Three yield periods were obtained during the run. The first was 12 hours long, the others were shortened because of operational problems.

The first yield period, at the conditions of M-1, gave similar results. Lowering the temperature for the balance of the run caused lowering of conversion of MAF lignite to below 60 percent. After 8 hours of operation in Yield Period M-2B, the F-1 bottoms viscosity became too high for pumping and the test was terminated. Yield Period M-2C was terminated after 9 hours because of a shortage of makeup solvent. The run was terminated because of F-1 bottoms plugging.

Two yield periods of this run, M-2A and M-2C, were worked up completely. The results are given in Parts 2 and 3 of Appendix A.

5. Run M-3

In this run an attempt was made to increase lignite conversion by raising the operating pressure to 2000 psig with the preheater outlet at the nominal 750°F. temperature. An attempt was made to reduce solvent breakdown by halving the residence time and therefore increasing the space velocity two-fold. This was done by using only one dissolver and by-passing the other. Because of continued solvent loss, the solvent-coal ratio was lowered to 2.3 for the balance of the run, and the vacuum flash temperature increased to 650°. All of the vacuum condensate from F-1 was returned to solvent recycle as was the bottoms from the light ends column F-2.

One uninterrupted 12 hour yield period was achieved (M-3B) at the 2.3 solvent-coal ratio. Conversion of lignite to gas, light oils and SRL was over 80 percent but solvent recovery remained low. The PDU was shut down after 3 days of operation because of difficulty with intermittent overflows of the vacuum flash drum F-1 into the vacuum condensate receiver S-7.

One yield period, M-3B, of this run is being worked up and the results will be presented in the next Quarterly Technical Progress Report.

6. Run M-4

Continuing the effort to find conditions under which solvent balance could be maintained, this run was made at an increased pressure of 2500 psig and preheater outlet of 750°F. A single dissolver was used as in M-3, and the solvent-coal ratio of 2.2 and vacuum flash temperature of 650°F during the latter part of M-3 was maintained.

Plugging of the vacuum flash drum F-1 terminated this run after about a day's operation. No yield periods were completed during this operation.

7. Run M-5

Nominal run conditions for Run M-5 were 2500 psig pressure,

750° preheater outlet temperature, and one dissolver used, resulting in a liquid hourly space velocity of 1.4, and a gas hourly space velocity of 301. In this run the solvent to MAF coal was increased to 3 and the vacuum flash temperature lowered to 600°F to reduce coking.

This run was easily the most successful to date. Six 12-hour yield periods were obtained. The system operated smoothly, although solvent recovery was still not adequate. In one series, data was gathered for 36 hours in three back-to-back yield periods. Detailed data for this run will be presented next quarter.

8. Problem Areas:

The major problem was in poor recovery of recycle solvent. Loss of about 20 percent was experienced initially, but in Run M-5, this was reduced to about 6 percent, a major improvement.

The operational problem causing the greatest concern is a gray, gritty residue, high in mineral matter which is left in the dissolvers at completion of a run. A significant quantity of this residue remains after only four to five days of operation which may occupy 15 to 25 percent of the reactor volume. Formation of this residue is unexplained and no satisfactory method of control or handling has been developed. Presence of this material in the effluent from the reactor may be part of trouble experienced with plugging and wear of control valves.

Better control of feed solvent to the slurry mix tank was achieved by replacement of the control valve with a variable speed metering pump. Other control valves also presented problems with wearing of valve stems giving inadequate seating. The F-1 bottoms tend to become more difficult to remove with increased time of run. The heavier material also seems to affect function of the level controller. Corrosion problems with light oils and water in the vacuum pumps are still troublesome in spite of installation of cold traps. Failure of high pressure diaphrams in the compressors occurred after 1000 to 1500 hours of operation.

B. PDU MAINTNEANCE AND CONSTRUCTION

1. General

Activities were concentrated on maintenance items related to operation of the PDU. Other work was accomplished when schedules permitted.

2. Area 00: Buildings and Utilities

Major maintenance work was done in the two control-air compressors in which new rings and valves were installed. Additionally, the crankshaft and main bearing were replaced on the south compressor.

Final inspection of the electrical installation was made and contract completion was accepted. The carbon monoxide alarms were recalibrated and one was found to be defective in that calibration could not be maintained. The excessive pressure drop in the control-air supply was corrected by replacement of the sintered metal filter element in the Norgren prefilter. The second level catwalk in the east bay was extended to permit easy access to the cold trap ahead of J-1. An alarm system was installed to warn of shutdown of instrument air compressors, circulating pump and air cooler in the west bay.

3. Area 01A: Coal Crushing and Grinding

Replacement of two cloth chutes for use in control of dust emissions from the pulverizer was the only work done. Otherwise, no problems were encountered and this area is fully operational.

4. Area 01B: Slurry Preparation and Pumping

The 1/2 inch tubing in the circulation line from the slurry mix tank (V-6 to P-6 around P-3A and P-3B and back to V-6) was replaced by 3/4 inch tubing. Needle valves were replaced by ball valves. Circulation rate was increased from 1 gpm to 3.5 gpm. A 480V motor circuit was extended to service this area for use with the new mixer to be installed later in the slurrying system. A smaller brass impeller was installed on the slurry circulating pump in an effort to reduce the overload experienced with the larger stainless-steel impeller at lower solvent-coal ratios. A Zenith metering pump was assembled on a U.S. Varidrive and installed as solvent charge pump to the mix tank.

5. Area 02: Slurry Preheating, Dissolving and Gas Separation

Installation of the two new Incoloy 800 dissolvers was completed. All piping from the outlet of the sand bath preheater (E-1) to the first separator (S-1) following the dissolvers were replaced with Incoloy 800 tubing. New piping was traced with heat tapes and insulated. The system was pressure tested hydrostatically and with nitrogen. Additional wall heaters installed on the dissolvers will significantly reduce start-up time.

General maintenance included repair of leaks and valves. Additional cooling fins were added to the air cooler (E-2) Separator S-4A was modified to give increased volume for longer separation time of water and oil. A 100 psig air line was piped to the top of the fluidized sand-bath preheater (E-1) to allow back flushing to clean the exhaust air filter.

6. Area 03: Solid-Liquid Separation

Installation of the drum heater for use in remelting vacuum bottoms was finished. A Hills-McCanna piston pump and necessary piping were installed to remove the vacuum bottoms. The pump can also take fluid from the bottoms pump (P-11) below the vacuum flash tower (F-1). Heating tapes and insulation were applied to the piping.

A Wanner Engineering Hydra-cell diaphram pump was evaluated for use in recirculation of the toluene-vacuum bottoms slurry. In one hour, the Viton diaphrams in the pump failed. The circulation temperature of 340^{o}F was decreased to 215^{o}F, and operation was satisfactory for 12 hours. However, 215^{o}F is considered to be too low to maintain the slurry in suspension. A M-D Pneumatic Inc. lobe pump was purchased for use in this service and will be installed on receipt of supporting equipment. Replacement diaphrams of KALREZ were ordered from DuPont for the Hydra-cell pump for use until the lobe pump's performance can be evaluated. Pneumatic operated ball valves installed in the bottoms discharge line from V-8 proved to perform well. Cleaning of lines was performed routinely.

7. Area 04: Liquid Separation and Recycle

All vessels and piping were hydrostatically tested at 2700 psig. Replacement was made of the level transmitter on light ends column reboiler (F-2). Ball bearings on the vacuum pumps were replaced with cast iron sleeve bearings to reduce corrosion. Heating tapes were checked, some piping relocated to improve flow characteristics, minor leaks repaired and lines cleaned.

8. Area 05: Gas Purification and Recycle

The diaphrams on the high pressure heads of both compressors developed leaks on the oil side and were replaced. The manufacture of the displacement meter (DM-6) for excess recycle gas had supplies an incorrect pressure rated meter (100 psi rather than 250 psi). The meter failed, but will be replaced by the manufacturer. The feed gas analyzer was checked and appears satisfactory.

9. Area 06: SRL-Hydrogenation

The reactor was placed in position. Completion of the system will be postponed until operating data from Areas 1 through 5 have been obtained. All materials and items of

equipment have been received with the exception of two high-pressure separators for which purchase authorization has not yet been given.

C. EXPERIMENTAL

1. Project Lignite Laboratory

a. General

Major emphasis in the laboratory was given to support analytical capability for the PDU operations. Autoclave experiments are now conducted only during periods of reduced analytical load or when required to furnish data for the PDU. Five batch autoclave liquefaction and two ash-SRL separation experiments were carried out. Four autoclave tests continued the investigation of the effects of storage conditions on liquefaction characteristics. The other autoclave test was conducted to determine the effect of using recycle solvent from PDU Run M-3A (S-7 bottoms) on liquefaction. The two solvent deashing experiments used toluene as extraction solvent and laboratory prepared SRL (with ash) and a Wilsonville undeashed SRC as feedstock.

Two visiting chemical engineers from Santiago, Chile participated in some of the experiments as part of their orientation program and training in processing of lignite.

Test conditions and analytical data from the autoclave experiments are given in Tables 1 and 2 of Appendix B. Data from the deashing experiments are presented in Table 3 of Appendix B. Experimental conditions and results are discussed in the following.

b. Liquefaction Activity Following Storage

Following termination of the test series concerned with liquefaction of samples stored under controlled conditions, the lignite samples were kept for several weeks in sealed plastic bags. Tests 613 through 616 with these samples were repeat experiments of 592 through 595 in which unusually high gas yields and total conversions were obtained.

Standard liquefaction procedures were used of a half hour at 752^{o}F with a solvent to MAF lignite ratio of 2/1 and a CO/H_2 ratio of 1/1 at 1000 psig initial pressure. Following the reaction period at 752^{o}F, the autoclave was cooled to 400^{o}F and the products removed. The liquid-solid product was suction filtered at 400^{o}F to remove ash and unreacted lignite. Detailed equipment discriptions and analytical procedures for the batch autoclave work were given in Quarterly Technical Progress Report No. 1, FE-1224-36.

Yield data for Runs 613 through 616 (using Autoclave "B") are compared with the baseline test data of Runs 504 and 505 (using Autoclave "A") in the following:

Run No.	504,505	613	614	615	616
Storage	None	N_2Strg	Air Strg	N_2 Strg	H_2O Strg
Yields, Wt% MAF Coal					
Gas	35.4	28.8	35.1	33.7	33.8
Liquid	66.0	67.7	57.6	61.4	66.7
(Light Oil)	(20.7)	(16.6)	(20.5)	(19.7)	(22.3)
(Net SRL)	(45.3)	(51.1)	(37.1)	(41.7)	(44.4)
Unconverted	8.9	11.8	12.4	6.7	7.4
Water and Ash	-10.3	-8.3	-5.1	-1.8	-7.9
Solvent Recovery, Wt%					
100% Recovery Basis	97.8	91.5	88.7	83.1	87.7

The following observations can be noted:

(1) Air storage appears to have a significant adverse effect on lignite liquefaction in that yields of net liquid and net SRL are reduced as is total.

(2) Lignite stored in an air-free environment for 70 weeks has about the same reactivity as fresh lignite.

Results from Runs 613 through 616 compare favorably with the yield test data observed for other Reactivity Series tests except for Runs 592, 593, 594 and 595 in Autoclave "A". Because of a problem with the ring seal in Autoclave "A" it was sent to the factory for repairs, and until its return Autoclave "B" was used for the tests. Of the 26 tests conducted in the Reactivity Series, 10 tests utilized Autoclave "A" and 16 tests were made with Autoclave "B". In Autoclave "B" lower reactor pressures at reaction temperatures were experienced as compared with Reactor "A". However, except for the tests after 70 weeks storage, there appeared to be no significant differences between the results in the two autoclaves. In the 70 week tests, Autoclave "B" gave lower gas yields and lower water consumption than Autoclave "A" though conversions and liquid yields were comparable.

c. Liquefaction with PDU Recycle Solvent

Standard liquefaction test procedures were employed in Run 617 (see b.). However, the solvent contained ash and some heavy oil with a boiling point in excess of 230°C at 1.6 Torr. Suction filtration was not possible because of plugging of the autoclave reactor transfer tube. After cooling to room temperature, the solid-liquid mixture was removed by hand, and extracted with pyridine. The extract and residue were analyzed to determine extent of conversion.

Part of the plugging problem was caused by the nature of the solvent, which contained some ash and heavy oil boiling above 230°C at 1.6 Torr. When the autoclave was disassembled for product recovery, large globules of the initial reaction

mixture were found distributed throughout the interior of the reactor chamber. The heavy oil concentration in the solvent could have partly been responsible for the plugging problem. The solvent had been chosen because of the heavy oil content, since a large portion of this fraction was thought to be coal-derived, and an evaluation of lignite derived solvent for solution-hydrogenation of lignite was desired. However, it was later found that the solvent contained about two percent ash, apparently because of slurry carryover from the vacuum flash tower to the vacuum condensate accumulator.

Yield data from Run 617 are compared with the average yields of 19 runs with fresh UNDFS120-74 containing essentially no heavy oil boiling above 230°C at 1.6 Torr:

Yields, wt% MAF Coal	Baseline Test (Avg. of 19 Runs with UNDFS120-74)	Run 617
Gas	34.0	40.0
Liquid	64.6	54.5
(Lt. Oil)	(6.6)	(1.5)
(Net SRL)	(58.0)	(53.0)
Unconverted	11.4	15.2
Water/Ash	-10.0	-9.7
Total	100.0	100.0

The conversion to net liquids in Run 617 is significantly less than in the baseline tests, though conversion may have been hampered by poor mixing of the raw lignite and the heavy PDU recycle solvent.

The high boiling ash-containing fraction of the recycle solvent does not improve dissolution of the raw lignite.

d. Deashing of PDU Vacuum Bottoms

The deashing experiments used extraction temperature and pressure of 500°F and 550 psig respectively. A description of both the test apparatus and the procedures used in these tests can be found in Quarterly Technical Progress Report No. 6 for July, August, and September 1975. The recovered SRL-solvent fractions from the deashing experiments were distilled at 1.6 Torr to remove the extraction solvent; the resulting vacuum bottoms solid was analyzed for ash content and its weight yield determined. The solid bottom layer (pot residue) recovered from each deashing test was stripped of residual deashing solvent by distillation and analyzed for ash content and pyridine solubility

A summary of results from the two deashing experiments follows:

Run No.	BEX-19	BEX-20
Solvent	Toluene	Toluene
Undeashed Feed	UND Lab SRL	Wilsonville SRC
Temperature, ^{O}F	500	500
Pressure, psig	550	550
% SRL or SRC in Feed		
Input Basis	79	76
Output Basis	80	63
% of SRL or SRC Extracted		
Input Basis	55	71
Output Basis	54	86
% of Original Ash in Extract	2.13	1.71
Ash Content of Extract, Wt%	0.50	0.38

Removal of mineral matter originally in the laboratory-prepared SRL or the Wilsonville SRC was high, 98 percent.

SRC (from Wilsonville) gave a higher yield of extract than laboratory produced undeashed SRL with toluene under the same test procedures and conditions of 500^{O}F and 550 psig.

A larger percentage of the pyridine soluble material (SRL or SRC) originally in the undeashed products of liquefaction was recovered from Tacoma or Wilsonville products from bituminous coal than has been recovered from laboratory or PDU products from lignite.

IV. CONCLUSIONS

A. PDU OPERATIONS

1. Five runs were made in the liquefaction section with multiple yield periods established under various operating conditions.

2. Recovery of recycle solvent was low, about 80 percent in initial runs with 94 percent being achieved in the last run.

3. Conversion of MAF lignite to SRL, light oils, and gases was as high as 90 percent.

4. Operation at a low solvent to lignite ratio presented operational problems and did not improve solvent recovery.

5. Operation of the vacuum flash drum at 650°F. to increase solvent recovery resulted in coking and subsequent plugging of the vessel. No coking was observed at 600°F.

6. Best operating conditions employed seemed to be 750°F at preheater outlet and 2500 psig operating pressure. Solvent to lignite ratio was maintained at 1.8 and hourly space velocity of liquid was 1.4 and of gases, 301.

7. The modified liquid-solids separation step did not operate satisfactory during a shakedown test because of difficulty in feeding of SRL from a heated drum.

B. PDU MAINTENANCE AND CONSTRUCTION

1. Installation of the Incoloy 800 preheater and dissolvers, and replacement of associated piping with Incoloy enabled operations to be resumed without the problems of stress corrosion cracking.

C. EXPERIMENTATION - PROJECT LIGNITE LABORATORY

1. Liquefaction in a batch autoclave under standard conditions indicated liquefaction characteristics of lignite stored for 70 weeks in an air free environment are not significantly different than freshly mined lignite.

2. Storage in air adversely effects liquefaction to a small extent by reducing yields of net liquid and SRL.

3. Use of recycle solvent containing high boiling fractions and ash resulted in poor liquefaction.

V. APPENDICES

A. PDU OPERATIONS

1. Yield Period M-1C

2. Yield Period M-2A

3. Yield Period M-2C

4. Solid-Liquid Separation

Figure 1. Flow Diagram of Solid-Liquid Separation System

B. LABORATORY EXPERIMENTATION

1. Batch Autoclave Operations

Table 1. Material Balance and Yield Data

Table 2. Analytical Data

2. Deashing Experiments

Table 3. Deashing by Solvent Extraction and Settling

APPENDIX A: PART 1

Process Development Unit Yield Period M-1C

February 5, 1976

M-1 was the first run made after the reconstruction of the liquefaction section of the PDU. It was to be run under similar conditions to those used in the design of the PDU, 750°F slurry preheater outlet temperature, a liquid hourly space velocity of 0.78, a gas hourly space velocity of 173, and with a solvent/raw coal ratio of about 1.86/1. However, the operating pressure was to be 1500 psig rather than the design of 3000 psig. In practice, the coal and solvent rates were somewhat higher and the gas rate somewhat lower than planned. There was a considerable increase in temperature across the dissolvers, so that the preheater outlet temperature was kept lower than planned in order to hold the maximum dissolver temperature to about 750°F.

Three twelve hour yield periods were made during the run. Of these, M-1C provided the most accurate and representative results, and was worked up completely. The operating conditions for yield period M-1C are summarized below:

	Nominal	Actual
LHSV	0.75	0.90
GHSV	155	164
Solvent/Coal Ratio	1.86	2.30
Dissolver Pressure, psig	1500	1500
Temperatures		
Preheater Outlet	750	702
R-1A Lower	-	708
R-1A Upper	-	748
R-1B Lower	-	716
R-1B Upper	-	735

In the following pages, the average run conditions, sample analyses, and overall component, and elemental material balances are presented.

In this case, product recovery was essentially 100 percent of the material charged, so that no adjustments to the overall material balance were necessary. However, the overall carbon recovery was slightly high and the overall oxygen recovery slightly low. This may be a result of the reproducibility of some of the elemental analytical procedures, particularly since oxygen is determined by difference in the solid and liquid samples, or it may indicate that compensating adjustments to flow rates should be made. The rates are corrected for differences in hold-up where the hold-up can be measured, but there may be accumulations of solid materials in the dissolvers and the vacuum flash drum for which corrections cannot be made. There may be errors in the weight of lignite charged, since feed drums of lignite must be changed during the run and this can affect the readings of the weigh cells used in the lignite charge system. There is also the possibility of undetected leaks in the gas system, particularly through leaking safety valves. Another problem is the accurate determination of the oil/water ratio in streams where emulsions can occur when the oil/water separators are not performing properly. A number of criteria have been developed for use in adjusting various streams when low material balances are encountered. Of course, the adjustments should result in 100 percent overall balance, with as close to 100 percent balance in carbon, hydrogen, and oxygen as is practical, and should not result in yields that are questionable, i.e., there should be water consumption because of the water gas shift reaction with a concomitant production of carbon dioxide, so that there should be a substantial gas make. A rough order of preference for adjusting streams if a low material balance has been obtained is as follows:

(1) Adjust the coal feed rate

(2) Increase the product gas rate

(3) Increase the waste gas rate

(4) Increase or decrease the product water rate, sometimes with an inverse change in the light oil rate

Utilizing the above guides can result in simple adjustments of certain flow rates that result in a reasonable overall yield structure.

A summary of the yields obtained in Run M-1C is given in the following table, as compared with the yield structure assumed for the PDU design:

	←——— M-1C ———→			←——— Design ———→		
Wt.%	Charge	Product	Net Yields(1)	Charge	Product	Net Yields(1)
Gas	25.3	30.3	10.4	20.3	39.8	39.5
MAF Coal	48.6	13.0	-73.3	49.4	4.8	-90.3
Water	21.4	19.5	- 4.0	24.7	19.2	-11.1
Ash	4.7	3.5	- 2.5	5.6	5.6	0
Net Light Oil		0(2)	0		4.6	9.3
Net SRL		33.7(2)	69.4		26.0	52.6
Total	100.0	100.0	0	100.0	100.0	0

(1) As weight percent of MAF coal charged.

(2) Not all solvent was recovered for recycle; some was present in the light oil and SRL streams.

The net gas production was about 10 weight percent of the MAF lignite charged and hydrocarbon liquid yields amounted to approximately 69 weight percent as determined largely by the pyridine extraction of the vacuum bottoms. The amount of reducing gases consumed, on a hydrogen equivalent basis, was 2.3 weight percent of the MAF lignite fed. Water consumption was nine percent of the initial water charged and conversion of lignite to gas and liquids was 73 percent.

Solvent recovery for recycle was far from the optimum at 86 percent. The unrecovered solvent was found by analyses to be in side streams (i.e., F-2 ovhd, F-3 ovhd) and in the F-1 vacuum bottoms.

Run Conditions

Run No: M-1C

2/5/76

TEMPERATURE

PREHEATER E-1

SAND BATH 764°F

INLET 307°F

OUTLET 702°F

DISSOLVERS

R-1A (1) 708°F

(2) 725°F

(3) 746°F

(4) 748°F

R-1B (1) 716°F

(2) 724°F

(3) 728°F

(4) 735°F

REACTOR PRODUCT SEPARATORS

S-1 548°F

S-2 58°F

S-3 497°F

S-4A 68°F

S-4B 75°F

PREHEATER E-11

DOWTHERM 619°F

INLET 456°F

OUTLET 594°F

VACUUM FLASH F-1

OVERHEAD VAPOR 572°F

UPPER WALL 591°F

LOWER WALL 542°F

DOWNCOMER 436°F

CONDENSATE ACCUMULATOR S-7 135°F

PREHEATER E-13

DOWTHERM 514°F

INLET 155°F

OUTLET 496°F

LIGHT ENDS COLUMN F-2

REBOILER 635°F

LOWER SECTION 426°F

FEED SECTION 388°F

OVERHEAD 287°F

SEPARATOR S-8 68°F

SEPARATOR S-9 72°F

SOLVENT COLUMN F-3

REBOILER 276°F

LOWER SECTION 230°F

FEED SECTION 221°F

OVERHEAD 209°F

HEAVY ENDS COLUMN F-4

REBOILER

LOWER SECTION

FEED SECTION

OVERHEAD

PRESSURE

SEPARATORS S-1&2 1500 psig

SEPARATORS S-3 350 psig

SEPARATORS 4A&B 46 psig

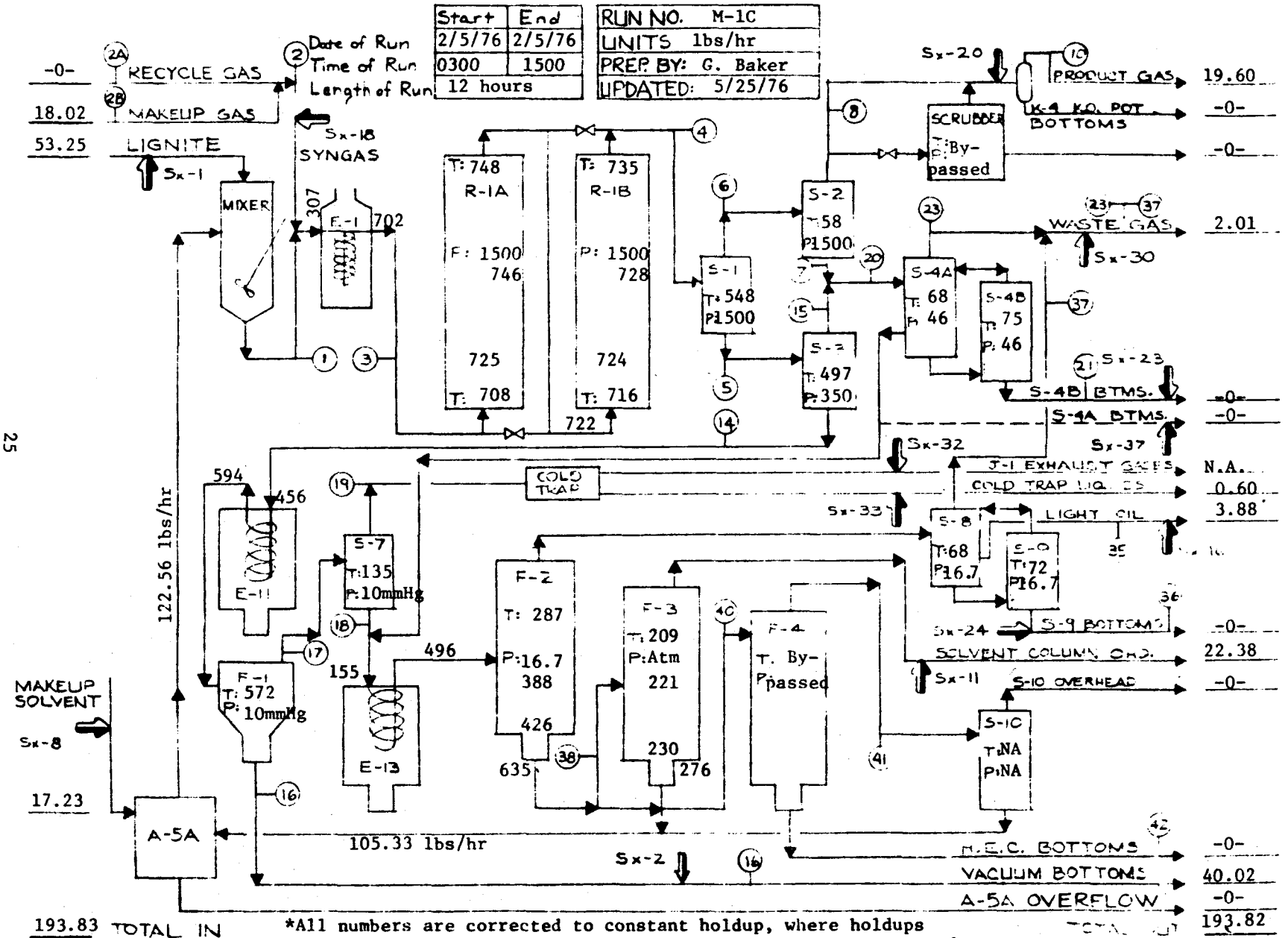
VACUUM FLASH F-1 10 mm Hg

L.E.C. F-2 16.7 psig

COLUMN F-3 0 psig

H.E.C. F-4 Not in use

*MASS RATES AND RUN CONDITIONS



*All numbers are corrected to constant holdup, where holdups are known. Sample weights and compositions have been assigned to appropriate streams.

PROJECT LIGNITE

Measured Material Balance

Updated: 6/30/76

Run No: M-1C
Date: 2/5/76
Time: 0300-1500

Summary

Temperature: 700-750°F
Pressure: 1500 psig
LHSV: 0.90
GHSV: 164

LBS/HOUR

	Stream Description	Total Charge	Total Product	Net Yield		Wt.% of MAF Coal	
Quantitative	Sample Point						
	Pressure (psig)						
	Temperature (°F)						
	Hydrogen - H_2	1.29	0.96	-0.33			
	Carbon Monoxide - CO	16.56	9.81	-6.75			
	Carbon Dioxide - CO_2		9.48	9.48			
	Hydrogen Sulfide - H_2S		0.11	0.11			
	Methane - CH_4		0.68	0.68	3.59	10.37	
	Ethane - C_2H_6		0.31	0.31			
	Propane - C_3H_8		0.02	0.02			
	Butane - C_4H_{10}						
	Nitrogen - N_2	0.17	0.24	0.07			
	Ammonia - NH_3						
	Light Oil (0-100°C @ 1.6 Torr)	4.87	10.02	5.15			
	Solvent (100-230°C @ 1.6 Torr)	100.25	95.97	-4.28	24.04	69.44	
	Heavy Oil (230-255°C @ 1.6 Torr)	17.44	13.32	-4.12			
	SRL		27.29	27.29			
	Coal (MAF)	34.62	9.23	-25.39		-73.33	
	Ash	3.35	2.50	-0.85		- 2.45	
	Water	15.28	13.88	-1.40		-4.04	
	Total	193.83	193.82	-0.01		-0.01	

Elemental	Carbon	140.91	143.68	2.77			
	Hydrogen	14.83	15.05	0.22			
	Nitrogen	0.79	0.75	-0.04			
	Sulfur	2.91	3.02	0.11			
	Oxygen (by diff)	30.99	28.82	-2.17			
	Ash	3.40	2.50	-0.90			
	Total	193.83	193.82	-0.01			

Prepared by:

PROJECT LIGNITE
MEASURED MATERIAL BALANCE

Updated: 6/30/76

Run No: M-1C
Date: 2/5/76
Time: 0300-1500

Material In
LBS/HOUR

Temperature: 700-750°F
Pressure: 1500 psig
LHSV: 0.90
GHSV: 164

Quantitative

Stream Description	Feed Syngas	Feed Lignite	Makeup Solvent	+ Feed Solvent		Total In
Sample Point	Sx-18	Sx-1	Sx-8	Sx-14		
Pressure (psig)	66	-	-	-		
Temperature (°F)	65	RT	RT	100		
Hydrogen - H_2	1.29					1.29
Carbon Monoxide - CO	16.56					16.56
Carbon Dioxide - CO_2						
Hydrogen Sulfide - H_2S						
Methane - CH_4						
Ethane - C_2H_6						
Propane - C_3H_8						
Butane - C_4H_{10}						
Nitrogen - N_2	0.17					0.17
Ammonia - NH_3						
Light Oil (0-100°C @ 1.6 Torr)			0.29	4.87		4.87
Solvent (100-230°C @ 1.6 Torr)			12.00	100.25		100.25
Heavy Oil (250-255°C @ 1.6 Torr)			4.94	17.44		17.44
SRL						
Coal (MAF)		34.62				34.62
Ash		3.35				3.35
Water		15.28				15.28
Total	18.02	53.25	17.23	122.56		193.83

Elemental

Carbon	7.10	24.80	15.39	109.01		140.91
Hydrogen	1.29	3.40	1.42	10.14		14.83
Nitrogen	0.17	0.35	0.02	0.27		0.79
Sulfur	-	0.32	0.38	2.59		2.91
Oxygen (By Diff.)	9.46	21.03	0.02	0.50		30.99
Ash		3.35		0.05		3.40
Total	18.02	53.25	17.23	122.56		193.83

Prepared by:

Note: Total In does not include makeup solvent as it is part of the feed solvent

+Feed solvent was determined by using the recorded flow and the planimeter.

PROJECT LIGNITE
MEASURED MATERIAL BALANCE

Updated: 6/30/76

Run No: M-1C
Date: 2/5/76
Time: 0300-1500

Material Out
LBS/HOUR

Temperature: 700-750°F
Pressure: 1500 psig
LHSV: 0.90
GHSV: 164

	Stream Description	Product Gas	*S-4A Ovhd	Cold Traps	K-1 Btms	J-1 Exhaust	S-8 Btms
	Sample Point	Sx-20	Sx-30	Sx-33	Sx-31	Sx-32	Sx-16
Quantitative	Pressure (psig)	100		-14.2	-14.2	0	15
	Temperature (°F)	72			RT	70	
	Hydrogen - H_2	0.93	0.03				
	Carbon Monoxide - CO	9.44	0.37				
	Carbon Dioxide - CO_2	8.02	1.46				
	Hydrogen Sulfide - H_2S	0.09	0.02				
	Methane - CH_4	0.64	0.04			NOT AVAILABLE	
	Ethane - C_2H_6	0.26	0.05				
	Propane - C_3H_8		0.02				
	Butane - C_4H_{10}						
	Nitrogen - N_2	0.22	0.02				
	Ammonia - NH_3						
	Light Oil (0-100°C @ 1.6 Torr)			0.19	-		0.59
	Solvent (100-230°C @ 1.6 Torr)			0.29	0.03		2.21
	Heavy Oil (230-255°C @ 1.6 Torr)			0.04	0.01		0.28
	SRL						
	Coal (MAF)						
	Ash						
	Water			0.04			0.80
	Total	19.60	2.01	0.56	0.04		3.88
Elemental	Carbon	6.92	0.64	0.45	0.035		2.71
	Hydrogen	1.15	0.06	0.05	0.003		0.35
	Nitrogen	0.22	0.02	-			0.01
	Sulfur	0.08	0.02	0.01	0.001		0.06
	Oxygen (by diff.)	11.23	1.27	0.05	0.001		0.75
	Ash						
	Total	19.60	2.01	0.56	0.04		3.88

* Composition estimated from M-2A data.

Prepared by:

PROTEK - LIGNITE
MEASURED MATERIAL BALANCE

Updated: 6/30/76

Run No: M-1C
Date: 2/5/76
Time: 0300-1500

Material Out
LBS/HOUR

Temperature: 700-750°F
Pressure: 1500 psig
LHSV: 0.90
GHSV: 164

	Stream Description	S-9 Btms	F-3 Ovhd	***F-1 Btms	Recycle Solvent		Total Out
	Sample Point	Sx-24	Sx-11	Sx-2	Sx-13		
	Pressure (psig)		-0-				
	Temperature (°F)		209				
Quantitative	Hydrogen - H_2						0.96
	Carbon Monoxide - CO						9.81
	Carbon Dioxide - CO_2						9.48
	Hydrogen Sulfide - H_2S						0.11
	Methane - CH_4						0.68
	Ethane - C_2H_6						0.31
	Propane - C_3H_8						0.02
	Butane - C_4H_{10}						
	Nitrogen - N_2						0.24
	Ammonia - NH_3						
	Light Oil (0-100°C @ 1.6 Torr)		4.61		4.63		10.02
	Solvent (100-230°C @ 1.6 Torr)		4.57	0.76	88.11		95.97
	Heavy Oil (230-255°C @ 1.6 Torr)		0.16	0.24	12.59		13.32
	SRL			27.29			27.29
	Coal (MAF)			9.23			9.23
	Ash			2.50			2.50
	Water		13.04				13.88
	Total	-0-	22.38	40.02	105.33		193.82
Elemental	Carbon		7.42	32.78	92.72		143.68
	Hydrogen		2.35	2.23	8.86		15.05
	Nitrogen		0.02	0.28	0.20		0.75
	Sulfur		0.09	0.55	2.21		3.02
	Oxygen (by Diff)		12.50	1.68	1.34		28.82
	Ash			2.50			2.50
	Total		22.38	40.02	105.33		193.82

*** Ash & pyridine soluables are based on lab samples 84 & 114, 2/5/76. Solvent composition based on sample 84. Conversion = 73%

Prepared by:

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 6/30/76

Run No: M-1C
Date: 2/5/76
Time: 0300-1500

WEIGHT PERCENT

Quantitative

Sample Description	Feed Lignite	F-1 Btms	Makeup Solvent	F-3 Ovhd (Oil)	F-3 Ovhd (Water)	F-3 Ovhd (Total)
Sample Point	Sx-1	Sx-2	Sx-8	Sx-11	Sx-11	Sx-11
Hydrogen - H_2						
Carbon Monoxide - CO						
Carbon Dioxide - CO_2						
Hydrogen Sulfide - H_2S						
Methane - CH_4						
Ethane - C_2H_6						
Propane - C_3H_8						
Butane - C_4H_{10}						
Nitrogen - N_2						
Ammonia - NH_3 (PPM)					(97)	
Light Oils (0-100°C @ 1.6 Torr)			1.70	49.35		20.61
Solvent (100-230°C @ 1.6 Torr)		1.90	69.65	48.90		20.43
Heavy Oil (230-255°C @ 1.6 Torr)		0.60	28.65	1.75		0.73
SRL		68.20				
Coal (MAF)	65.01	23.05				
Ash	6.30	6.25				
Water	28.69				100	58.23
Total	100	100	100	100	100	100
Specific Gravity 60/60			1.062			
Viscosity, cp						

Elemental

Carbon	46.58	81.90	89.31	79.41		33.17
Hydrogen	6.38	5.56	8.22	9.62	11.11	10.49
Nitrogen	0.66	0.71	0.12	0.24		0.10
Sulfur	0.59	1.38	2.20	0.98		0.41
Oxygen (by diff)	39.49	4.20	0.15	9.75	88.89	55.83
Ash	6.30	6.25	-0-	-0-		
Total	100	100	100	100	100	100
Number of Sample Avg.	2	2	2	2	2	2

Prepared by: G. Baker

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 6/30/76

Run No: M-1C
Date: 2/5/76
Time: 0300-1500

WEIGHT PERCENT

Quantitative

Sample Description	F-2 Btms	Feed Solvent	S-8 Oil	S-8 Water	S-8 Total	Feed Gas
Sample Point	Sx-13	Sx-14	Sx-16	Sx-16	Sx-16	Sx-18
Hydrogen - H_2						7.15
Carbon Monoxide - CO						91.89
Carbon Dioxide - CO_2						
Hydrogen Sulfide - H_2S						
Methane - CH_4						
Ethane - C_2H_6						
Propane - C_3H_8						
Butane - C_4H_{10}						
Nitrogen - N_2						0.96
Ammonia - NH_3 (PPM)						
Light Oils (0-100°C @ 1.6 Torr)	4.40	3.97	19.06		15.13	
Solvent (100-230°C @ 1.6 Torr)	83.65	81.80	71.68		56.90	
Heavy Oil (230-255°C @ 1.6 Torr)	11.95	14.23	9.26		7.35	
SRL						
Coal (MAF)						
Ash						
Water				100	20.62	
Total	100	100	100	100	100	100
Specific Gravity 60/60		1.046				
Viscosity, cp						

Elemental

Carbon	88.03	88.95	88.01		69.86	39.38
Hydrogen	8.41	8.27	8.58	11.11	9.10	7.15
Nitrogen	0.19	0.22	0.29		0.23	0.96
Sulfur	2.10	2.11	2.00		1.59	
Oxygen (by diff)	1.27	0.41	1.12	88.89	19.22	52.51
Ash	-0-	0.04			-0-	
Total	100	100	100	100	100	100
Number of Sample Avg.	3	3	2	2	2	3

Prepared by: Gene BAker

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 6/30/76

Run No: M-1C
Date: 2/5/76
Time: 0300-1500

WEIGHT PERCENT

Quantitative

Sample Description	Product Gas	+S-4A Ovhd	++ K-1 Btms	J-1 Exhaust	Cold Traps	
Sample Point	Sx-20	Sx-30	Sx-31	Sx-32	Sx-33	
Hydrogen - H_2	4.76	1.44				
Carbon Monoxide - CO	48.14	18.46				
Carbon Dioxide - CO_2	40.93	72.53				
Hydrogen Sulfide - H_2S	0.45	1.25				
Methane - CH_4	3.24	2.10				
Ethane - C_2H_6	1.36	2.35				
Propane - C_3H_8		1.03				
Butane - C_4H_{10}						
Nitrogen - N_2	1.12	0.84				
Ammonia - NH_3 (PPM)						
Light Oils (0-100°C @ 1.6 Torr			3.10		34.35	
Solvent (100-230°C @ 1.6 Torr)			84.20		52.51	
Heavy Oil (230-255°C @ 1.6 Torr)			12.70		6.74	
SRL						
Coal (MAF)						
Ash						
Water					6.40	
Total	100	100	100		100	
Specific Gravity 60/60						
Viscosity, cp						

NOT AVAILABLE

Elemental

Carbon	35.31	31.99	87.50		81.25	
Hydrogen	5.87	2.69	7.50		8.36	
Nitrogen	1.12	0.84	-		0.12	
Sulfur	0.42	1.18	2.50		1.97	
Oxygen (by diff)	57.28	63.30	2.50		8.30	
Ash						
Total	100	100	100		100	
Number of Sample Avg.	2	2	1		2	

+ Gas composition estimated from M-2A data

++ Elemental composition was estimated

Prepared by: Gene Baker

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 6/30/76

Run No: M-1C
Date: 2/5/76
Time: 0300-1500

MOLE PERCENT

Quantitative

Sample Description	Feed Gas	Product Gas	+S-4A Ovhd			
Sample Point	Sx-18	Sx-20	Sx-30			
Hydrogen - H_2	51.87	44.65	21.70			
Carbon Monoxide - CO	47.63	32.25	19.80			
Carbon Dioxide - CO_2		17.45	49.50			
Hydrogen Sulfide - H_2S		0.25	1.10			
Methane - CH_4		3.80	3.95			
Ethane - C_2H_6		0.85	2.35			
Propane - C_3H_8			0.70			
Butane - C_4H_{10}						
Nitrogen - N_2	0.50	0.75	0.90			
Ammonia - NH_3 (PPM)						
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						
Total	100	100	100			
Specific Gravity 60/60						
Viscosity, cp						

Elemental

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

+ Gas Composition estimated from M-2A data

Prepared by: Gene Baker

APPENDIX A: PART 2

Process Development Unit Yield Period M-2A

February 18, 1976

In the first run of this series, M-1, insufficient solvent was recovered to remain in solvent balance. Analyses indicated there to be solvent in the light streams from the high-pressure separation system and also in the vacuum bottoms from the vacuum flash drum. Consequently, in M-2 more attention was to be given to recovering solvent from the light oil stream, and in the vacuum flash drum F-1. In practice, at the start of Run M-2, the reactors were operating at somewhat higher overall temperatures than in M-1 and with lower solvent recovery. The nominal and actual operating conditions for this yield period are shown in the table following:

	Nominal	Actual
LHSV	0.75	0.85
GHSV	160	160
Solvent/Coal Ratio	1.86	1.80
Dissolver Pressure, psig	1500	1500
Temperature, ^{o}F		
Preheater Outlet	750	733
Dissolver R-1A Lower	-	714
Dissolver R-1A Upper	-	760
Dissolver R-1B Lower	-	735
Dissolver R-1B Upper	-	752

During M-2A an overall material balance of 103.3 weight percent was realized. However, backing out the circulating solvent results in a net material balance of 105.2 weight percent. Because of the possible errors in weighing the coal in, this stream was selected for adjustment to provide 100 percent material balance. The yield structure, on a solvent free basis,

after this adjustment is shown below:

	← M-2A →		
Wt. %	Charge	Product	Net Yield (1)
Gas	23.4	31.8	16.5
MAF Coal	51.1	9.2	-81.9
Water	21.0	15.3	-11.2
Ash	4.5	3.9	- 1.2
Net Light Oil(2)		2.1	4.0
Net SRL(2)		37.7	73.8
Total	100.0	100.0	0

(1) As weight percent of MAF coal charged.
(2) Not all solvent was recovered for recycle; some was present in the light oil and SRL streams.

In this run the net gas production was about 16 percent of the MAF lignite charged and the net hydrocarbon liquid yield amounted to approximately 78 percent, as determined largely by pyridine extraction and distillation of the vacuum bottoms. The amount of reducing gases consumed, on a hydrogen equivalent basis, was about 1.7 weight percent of the MAF lignite fed. Water consumption was 27 percent of that fed, and conversion of MAF lignite to gas and liquid was 82 percent.

In this yield period solvent recovery and recycle was even lower than in the previous Run M-1, at slightly over 60 weight percent of the solvent charged. Again, unrecovered solvent was found by analyses in the F-2 distillation column overhead stream and in the F-1 vacuum flash drum bottoms.

Run Conditions
Run M-2A
2/18/76

TEMPERATURE

PREHEATER E-1

SAND BATH	784
INLET	308
OUTLET	733

DISSOLVERS

R-1A (1)	714
(2)	735
(3)	752
(4)	760
R-1B (1)	735
(2)	742
(3)	745
(4)	752

REACTOR PRODUCT SEPARATORS

S-1	562
S-2	63
S-3	723
S-4A	71
S-4B	70

PREHEATER E-11

DOWTHERM	609
INLET	546
OUTLET	583

VACUUM FLASH F-1

OVERHEAD VAPOR	538
UPPER WALL	547
LOWER WALL	609
DOWNCOMER	434
CONDENSATE ACCUMULATOR S-7	135

PREHEATER E-13

DOWTHERM	498
INLET	116
OUTLET	369

LIGHT ENDS COLUMN F-2

REBOILER	501
LOWER SECTION	324
FEED SECTION	306
OVERHEAD	235
SEPARATOR S-8	105
SEPARATOR S-9	71

SOLVENT COLUMN F-3

REBOILER	NIU
LOWER SECTION	NIU
FEED SECTION	NIU
OVERHEAD	NIU

HEAVY ENDS COLUMN F-4

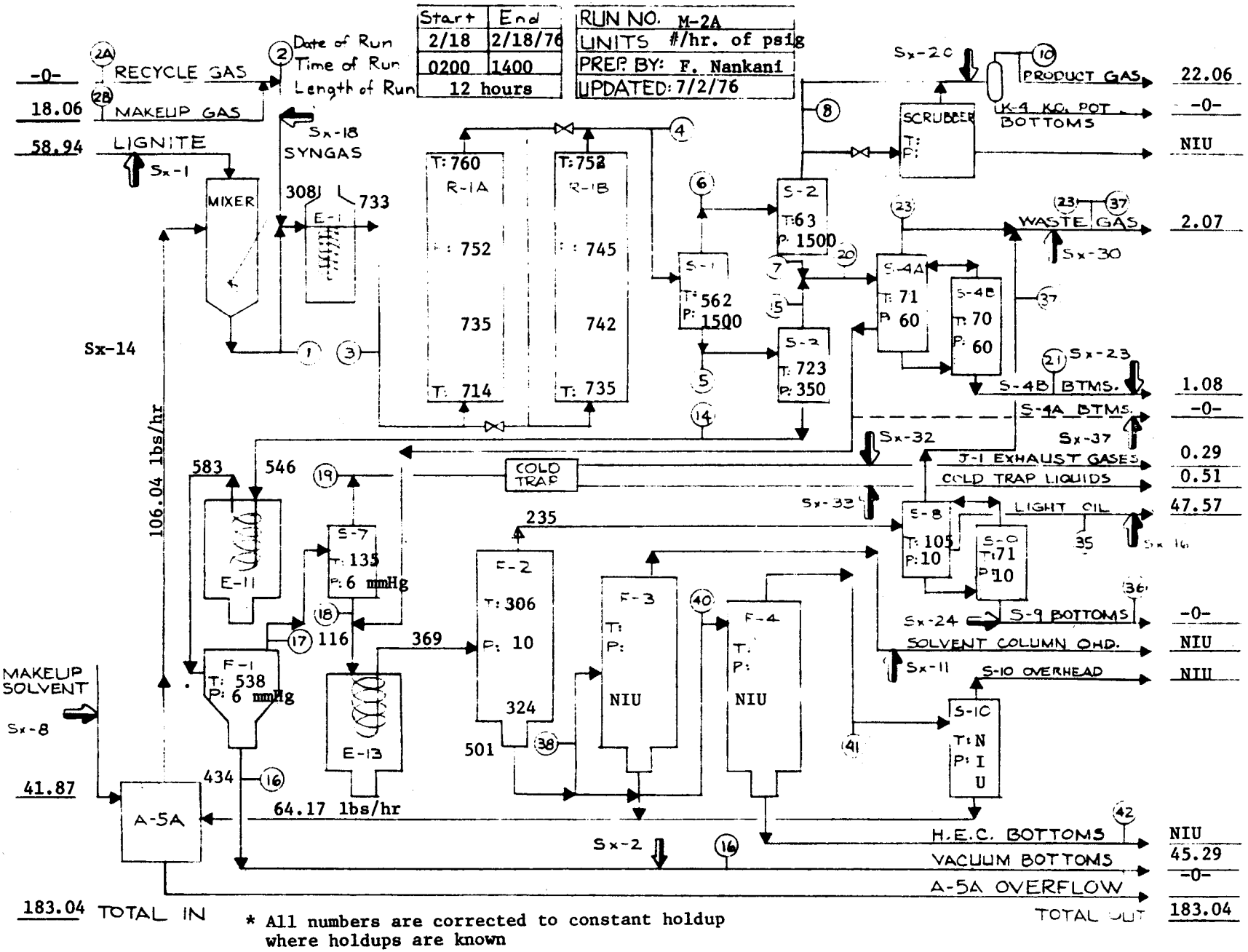
REBOILER	NIU
LOWER SECTION	NIU
FEED SECTION	NIU
OVERHEAD	NIU

PRESSURE

SEPARATORS S-1&2	1500
SEPARATORS S-3	350
SEPARATORS 4A&B	60
VACUUM FLASH F-1	6 mm Hg
L.E.C. F-2	10
COLUMN F-3	NIU
H.E.C. F-4	NIU

Adjusted Material Balance

* MASS RATES AND RUN CONDITIONS



* All numbers are corrected to constant holdup where holdups are known

PROJECT LIGNITE

ASJUSTED MATERIAL BALANCE

Updated: 7/2/76

Run No: M-2A
Date: 2/18/76
Time: 0200-1400

Summary

Temperature: 750°F
Pressure: 1500 psig
LHSV: 0.85
GHSV: 160

	Stream Description	Total Charge	Total Product	Net Yield		Wt.%of MAF Coal	
	Sample Point						
	Pressure (psig)						
	Temperature (°F)						
Quantitative	Hydrogen - H_2	1.21	0.99	-0.22			
	Carbon Monoxide - CO	16.65	10.50	-6.15			
	Carbon Dioxide - CO_2		11.75	11.75			
	Hydrogen Sulfide - H_2S		0.11	0.11			
	Methane - CH_4		0.60	0.60	6.48	16.46	
	Ethane - C_2H_6		0.31	0.31			
	Propane - C_3H_8		0.14	0.14			
	Butane - C_4H_{10}						
	Nitrogen - N_2	0.20	0.14	-0.06			
	Ammonia - NH_3						
	Light Oil (0-100°C @ 1.6 Torr)	3.50	17.89	14.39			
	Solvent (100-230°C @ 1.6 Torr)	85.52	77.40	-8.12	30.65	77.85	
	Heavy Oil (230-255°C @ 1.6 Torr)	17.02	12.34	-4.68			
	SRL		29.06	29.06			
	Coal (MAF)	39.37	7.11	-32.26		-81.94	
	Ash	3.47	2.99	-0.48		- 1.22	
	Water	16.22	11.83	-4.39		-11.15	
	Total	183.16	183.16	0.00		0	

Elemental	Carbon	129.53	132.80	3.27			
	Hydrogen	13.83	13.68	-0.15			
	Nitrogen	0.79	0.67	-0.12			
	Sulfur	2.68	2.70	+0.02			
	Oxygen	32.86	30.32	-2.54			
	Ash	3.47	2.99	-0.48			
	Total	183.16	183.16	0.00			

Prepared by:

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
(Feed Lignite Increased)
Updated: 7/2/76

Run No: M-2A
Date: 2/18/76
Time: 0200-1400

Material In

LBS/HOUR

Temperature: 750°F
Pressure: 1500 psig
LHSV: 0.85
GHSV: 160

Quantitative

Stream Description	Feed Gas	Feed Lignite	Makeup Solvent	Feed Solvent		Total In
Sample Point	Sx-18	Sx-1	Sx-8	Sx-14		
Pressure (psig)						
Temperature (°F)						
Hydrogen - H_2	1.21					1.21
Carbon Monoxide - CO	16.65					16.65
Carbon Dioxide - CO_2						
Hydrogen Sulfide - H_2S						
Methane - CH_4						
Ethane - C_2H_6						
Propane - C_3H_8						
Butane - C_4H_{10}						
Nitrogen - N_2	0.20					0.20
Ammonia - NH_3						
Light Oil (0-100°C @ 1.6 Torr)			0.77	3.50		3.50
Solvent (100-230°C @ 1.6 Torr)			31.28	85.52		85.52
Heavy Oil (230-255°C @ 1.6 Torr)			9.82	17.02		17.02
SRL						
Coal (MAF)		39.37				39.37
Ash		3.47				3.47
Water		16.22				16.22
Total	18.06	59.06	41.87	106.04		183.16

Elemental

Carbon	7.14	28.03	37.42	94.36		129.53
Hydrogen	1.21	3.82	3.48	8.80		13.83
Nitrogen	0.20	0.42	0.05	0.17		0.79
Sulfur		0.34	0.90	2.34		2.68
Oxygen (by diff)	9.51	22.98	0.02	0.37		32.86
Ash		3.47				3.47
Total	18.06	59.06	41.87	106.04		183.16

Note: Feed Solvent includes makeup solvent

Prepared by:

PROJECT LIGNITE
MEASURED AND ADJUSTED MATERIAL BALANCE

Updated: 7/2/76

Run No: M-2A
Date: 2/18/76
Time: 0200-1400

Material Out

Temperature: 750°F
Pressure: 1500 psig
LHSV: 0.85
GHSV: 160

LBS/HOUR

Quantitative

Stream Description	Product Gas	S-4A Ovhd	S-4B Btms	J-1 Exhaust	Cold Traps	S-8 Btms
Sample Point	Sx-20	Sx-30	Sx-23	Sx-32	Sx-33	Sx-16
Pressure (psig)						
Temperature (°F)						
Hydrogen - H_2	.96	0.03		0		
Carbon Monoxide - CO	10.08	0.38		0.04		
Carbon Dioxide - CO_2	9.92	1.50		0.33		
Hydrogen Sulfide - H_2S	0.06	0.03		0.02		
Methane - CH_4	0.56	0.04		0		
Ethane - C_2H_6	0.25	0.05		0.01		
Propane - C_3H_8	0.12	0.02				
Butane - C_4H_{10}						
Nitrogen - N_2	0.11	0.02		0.01		
Ammonia - NH_3						
Light Oil (0-100°C @ 1.6 Torr)			0.39		0.10	12.58
Solvent (100-230°C @ 1.6 Torr)			0.54		0.02	21.36
Heavy Oil (230-255°C @ 1.6 Torr)			0.03		0.01	2.30
SRL						
Coal (MAF)						
Ash						
Water			0.12			11.33
Total	22.06	2.07	1.08	0.41	0.51	47.57

Elemental

Carbon	7.74	0.66	0.80	0.11	0.11	29.76
Hydrogen	1.17	0.06	0.11	0.01	0.05	4.44
Nitrogen	0.11	0.02	-	0.01	-	0.05
Sulfur	0.06	0.02	0.01	0.02	0.01	0.65
Oxygen (by diff)	12.98	1.31	0.16	0.26	0.34	12.67
Ash						
Total	22.06	2.07	1.08	0.41	0.51	47.57

Prepared by:

PROJECT LIGNITE

MEASURED AND ADJUSTED MATERIAL BALANCE

Updated: 7/2/76

Run No: M-2A
Date: 2/18/76
Time: 0200-1400

Material Out

Temperature: 750°F
Pressure: 1500 psig
LHSV: 0.85
GHSV: 160

LBS/HOUR

	Stream Description	F-1 Btms	F-2 btms				Total Out
	Sample Point	Sx-2	Sx-13				
	Pressure (psig)	6mmHg					
	Temperature (°F)	579					
Quantitative	Hydrogen - H_2						0.99
	Carbon Monoxide - CO						10.50
	Carbon Dioxide - CO_2						11.75
	Hydrogen Sulfide - H_2S						0.11
	Methane - CH_4						0.60
	Ethane - C_2H_6						0.31
	Propane - C_3H_8						0.14
	Butane - C_4H_{10}						
	Nitrogen - N_2						0.14
	Ammonia - NH_3						
	Light Oil (0-100°C @ 1.6 Torr)		4.82				17.89
	Solvent (100-230°C @ 1.6 Torr)	2.51	52.97				77.40
	Heavy Oil (230-255°C @ 1.6 Torr)	3.62	6.38				12.34
	SRL	29.06					29.06
	Coal (MAF)	7.11					7.11
	Ash	2.99					2.99
	Water						11.83
	Total	45.29	64.17				183.16

Elemental	Carbon	36.99	56.63				132.80
	Hydrogen	2.45	5.39				13.68
	Nitrogen	0.36	0.12				0.67
	Sulfur	0.60	1.33				2.70
	Oxygen (by diff)	1.90	0.70				30.32
	Ash	2.99					2.99
	Total	45.29	64.17				183.16

Prepared by:

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 7/2/76

Run No: M-2A
Date: 2/18/76
Time: 0200-1400

WEIGHT PERCENT

Quantitative

Sample Description	Lignite	F-1 Btms	Makeup Solvent	F-2 Btms	Feed Solvent	S-8 Oil
Sample Point	Sx-1	Sx-2	Sx-8	Sx-13	Sx-14	Sx-16
Hydrogen - H_2						
Carbon Monoxide - CO						
Carbon Dioxide - CO_2						
Hydrogen Sulfide - H_2S						
Methane - CH_4						
Ethane - C_2H_6						
Propane - C_3H_8						
Butane - C_4H_{10}						
Nitrogen - N_2						
Ammonia - NH_3 (PPM)						
Light Oils (0-100°C @ 1.6 Torr)			1.85	7.52	3.30	34.70
Solvent (100-230°C @ 1.6 Torr)		5.55	74.70	82.54	80.65	58.95
Heavy Oil (230-255°C @ 1.6 Torr)		8.00	23.45	9.94	16.05	6.35
SRL		64.16				
Coal (MAF)	66.67	15.70				
Ash	5.87	6.59				
Water	27.46					
Total	100	100	100	100	100	100
Specific Gravity 60/60			1.048		1.041	
Viscosity, cp						

Elemental

Carbon	47.46	81.67	89.36	88.25	88.98	82.11
Hydrogen	6.47	5.41	8.32	8.40	8.30	8.76
Nitrogen	0.71	0.80	0.12	0.18	0.16	0.14
Sulfur	0.58	1.32	2.15	2.08	2.21	1.80
Oxygen (by diff)	38.91	4.21	0.05	1.09	0.35	7.19
Ash	5.87	6.59				
Total	100	100	100	100	100	100
Number of Sample Avg.	2	2	2	2	2	2

Prepared by:

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 7/2/76

Run No: M-2A
Date 2/18/76
Time: 0200-1400

WEIGHT PERCENT

	Sample Description	S-8 Water	S-8 Total	Feed Gas	Product Gas	S-4B Oil	S-4B Water
	Sample Point	Sx-16	Sx-16	Sx-18	Sx-20	Sx-23	Sx-23
Quantitative	Hydrogen - H_2			6.67	4.34		
	Carbon Monoxide - CO			92.21	45.69		
	Carbon Dioxide - CO_2				44.97		
	Hydrogen Sulfide - H_2S				0.26		
	Methane - CH_4				2.55		
	Ethane - C_2H_6				1.14		
	Propane - C_3H_8				0.55		
	Butane - C_4H_{10}				-		
	Nitrogen - N_2			1.12	0.50		
	Ammonia - NH_3 (PPM)	(86)					(144)
	Light Oils (0-100°C @ 1.6 Torr)		26.44			40.80	
	Solvent (100-230°C @ 1.6 Torr)		44.91			56.00	
	Heavy Oil (230-255°C @ 1.6 Torr)		4.84			3.20	
	SRL						
	Coal (MAF)						
	Ash						
	Water	100	23.81				100
	Total	100	100	100	100	100	100
	Specific Gravity 60/60						
	Viscosity, cp						
Elemental	Carbon		62.56	39.52	35.11	83.06	
	Hydrogen	11.11	9.32	6.67	5.32	9.80	11.11
	Nitrogen		0.11	1.12	0.50	0.12	
	Sulfur		1.37		0.25	1.14	
	Oxygen (by diff)	88.89	26.64	52.69	58.82	5.88	88.89
	Ash						
	Total	100	100	100	100	100	100
	Number of Sample Avg.	2	2	2	2	2	2

Prepared by:

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 7/2/76

Run No: M-2A
Date: 2/18/76
Time: 0200-1400

WEIGHT PERCENT

	Sample Description	S-4B Total	S-9 btms	S-4A Ovhd	J-1 Exhaust	Cold Traps	Recycle Solvent
	Sample Point	Sx-23	Sx-24	Sx-30	Sx-32	Sx-33	Sx-35
Quantitative	Hydrogen - H_2			1.44	0.65		
	Carbon Monoxide - CO			18.46	9.61		
	Carbon Dioxide - CO_2			72.53	81.68		
	Hydrogen Sulfide - H_2S			1.25	4.17		
	Methane - CH_4			2.10	0.36		
	Ethane - C_2H_6			2.35	1.34		
	Propane - C_3H_8			1.03			
	Butane - C_4H_{10}						
	Nitrogen - N_2			0.84	2.19		
	Ammonia - NH_3 (PPM)						
	Light Oils (0-100°C @ 1.6 Torr)	36.27	2.70			19.78	9.20
	Solvent (100-230°C @ 1.6 Torr)	49.78	65.20			4.52	82.95
	Heavy Oil (230-255°C @ 1.6 Torr)	2.84	32.10			0.70	7.85
	SRL						
	Coal (MAF)						
	Ash						
	Water	11.11				75.00	
	Total	100	100	100	100	100	100
	Specific Gravity 60/60						1.039
	Viscosity, cp						

Elemental	Carbon	73.83	89.60	31.99	27.74	21.75	87.61
	Hydrogen	9.94	7.92	2.69	1.26	10.48	8.36
	Nitrogen	0.11	0.06	0.84	2.19	0.05	0.15
	Sulfur	1.01	2.16	1.18	3.92	0.40	2.12
	Oxygen (by diff)	15.11	0.26	63.30	64.89	67.32	1.76
	Ash						
	Total	100	100	100	100	100	100
	Number of Sample Avg.	2	1	2	1	1	2

Prepared by:

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 7/2/76

Run No: M-2A
Date: 2/18/76
Time: 0200-1400

MOLE PERCENT

Quantitative

Sample Description	Feed Gas	Product Gas	S-4A Ovhd	* J-1 Exhaust		
Sample Point	Sx-18	Sx-20	Sx-30	Sx-32		
Hydrogen - H_2	50.00	42.90	21.70	11.6		
Carbon Monoxide - CO	49.40	32.25	19.80	12.3		
Carbon Dioxide - CO_2		20.20	49.50	66.5		
Hydrogen Sulfide - H_2S		0.15	1.10	4.4		
Methane - CH_4		3.15	3.95	0.8		
Ethane - C_2H_6		0.75	2.35	1.6		
Propane - C_3H_8		0.25	0.70			
Butane - C_4H_{10}						
Nitrogen - N_2	0.60	0.35	0.90	2.8		
Ammonia - NH_3 (PPM)		(118)				
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						
Total	100	100	100	100		
Specific Gravity 60/60						
Viscosity, cp						

Elemental

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

* Corrected for air contamination
** Analysis shown is for M-2C sample.

Prepared by:

PROJECT LIGNITE
MEASURED MATERIAL BALANCE

Updated: 7/2/76

Run No: M-2A
Date: 2/18/76
Time: 0200-1400

Material In

LBS/HOUR

Temperature: 750°F
Pressure: 1500 psig
LHSV: 0.81
GHSV: 160

Quantitative

Stream Description	Feed Gas	Feed Lignite	Makeup Solvent	Feed Solvent		Total In
Sample Point	Sx-18	Sx-1	Sx-8	Sx-14		
Pressure (psig)						
Temperature (°F)		RT	RT			
Hydrogen - H_2	1.21					1.21
Carbon Monoxide - CO	16.65					16.65
Carbon Dioxide - CO_2						
Hydrogen Sulfide - H_2S						
Methane - CH_4						
Ethane - C_2H_6						
Propane - C_3H_8						
Butane - C_4H_{10}						
Nitrogen - N_2	0.20					0.20
Ammonia - NH_3						
Light Oil (0-100°C @ 1.6 Torr)			0.77	3.50		3.50
Solvent (100-230°C @ 1.6 Torr)			31.28	85.52		85.52
Heavy Oil (230-255°C @ 1.6 Torr)			9.82	17.02		17.02
SRL						
Coal (MAF)		35.35				35.35
Ash		3.11				3.11
Water		14.56				14.56
Total	18.06	53.02	41.87	106.04		177.12

Elemental

Carbon	7.14	25.16	37.42	94.35		126.66
Hydrogen	1.21	3.43	3.48	8.80		13.44
Nitrogen	0.20	0.38	0.05	0.17		0.75
Sulfur		0.31	0.90	2.34		2.65
Oxygen (by diff)	9.51	20.63	0.02	0.37		30.51
Ash		3.11				3.11
Total	18.06	53.02	41.87	106.04		177.12

Note: Feed Solvent includes makeup solvent

Prepared by:

APPENDIX A PART 3

Process Development Unit

Yield Period M-2C

February 19, 1976

In Run M-2C conditions had been changed from the operating conditions of M-2A. Solvent recovery in M-2A was even lower than in Run M-1. Batch autoclave studies had indicated that higher solvent recoveries could be obtained at the lower temperature of 700°F, though conversion would be lower. The operating conditions for yield period M-2C were changed to those summarized below:

	Nominal	Actual
LHSV	0.75	0.71
GHSV	160	162
Solvent/Coal Ratio	1.86	2.06
Dissolver Pressure, psig	1500	1500
Temperatures, °F		
Preheater Outlet	700	698
R-1A Lower	-	663
R-1A Upper	-	699
R-1B Lower	-	675
R-1B Upper	-	690

Under these conditions the conversion dropped to less than 60 percent of the MAF coal, and solvent recovery increased only to 77 percent.

The overall material balance was 106 percent, and was again adjusted by increasing the value for the pounds of lignite charged. The overall adjusted yield structure on a solvent free basis is as follows:

Wt. %	Charge	Product	Net Yield(1)
Gas	26.2	30.2	8.2
MAF Coal	48.5	20.2	-58.4
Water	21.2	19.3	- 3.7
Ash	4.1	4.4	0.6
Light Oil		-	-
SRL		25.9	53.3
Total	100.0	100.0	0

(1) As weight percent of MAF coal.

In this run the hydrogen equivalent of the reducing gases consumed was about 1.4 weight percent of the MAF coal charged.

RUN CONDITIONS
M-2C
2/19/76

TEMPERATURE

PREHEATER E-1

SAND BATH 718
INLET 304
OUTLET 698

DISSOLVERS

R-1A (1) 663
(2) 678
(3) 692
(4) 699
R-1B (1) 675
(2) 681
(3) 686
(4) 690

REACTOR PRODUCT SEPARATORS

S-1 504
S-2 58
S-3 542
S-4A 63
S-4B 68

PREHEATER E-11

DOWTHERM 603
INLET 480
OUTLET 552

VACUUM FLASH F-1

OVERHEAD VAPOR 528
UPPER WALL 538
LOWER WALL 610
DOWNCOMER 508
CONDENSATE ACCUMULATOR S-7 130

PREHEATER E-13

DOWTHERM 503
INLET 89
OUTLET 433

LIGHT ENDS COLUMN F-2

REBOILER 507
LOWER SECTION 377
FEED SECTION 263
OVERHEAD 230
SEPARATOR S-8 102
SEPARATOR S-9 69

SOLVENT COLUMN F-3

REBOILER x
LOWER SECTION x
FEED SECTION x
OVERHEAD x

HEAVY ENDS COLUMN F-4

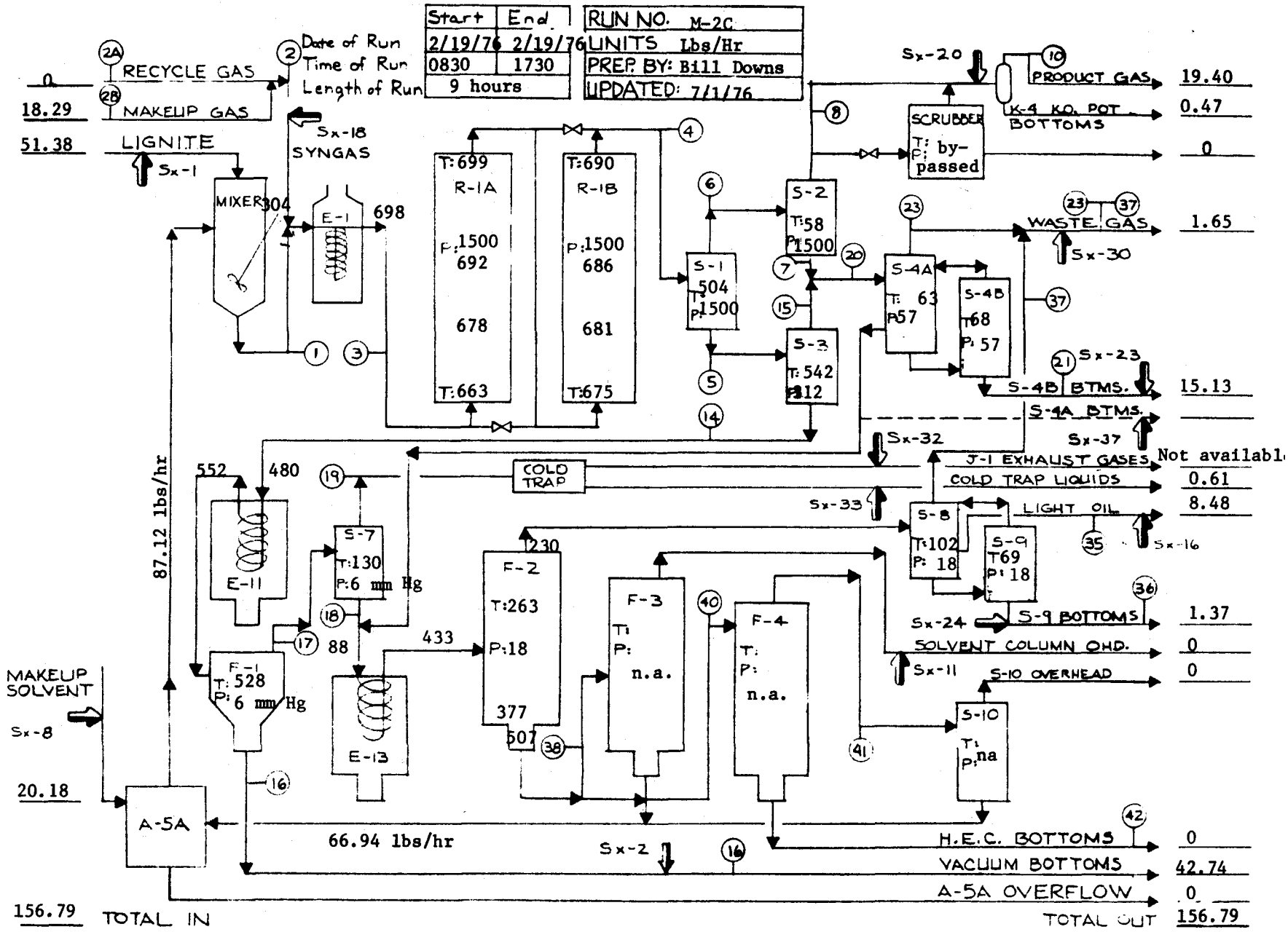
REBOILER x
LOWER SECTION x
FEED SECTION x
OVERHEAD x

PRESSURE

SEPARATORS S-1&2 1500
SEPARATORS S-3 312
SEPARATORS 4A&B 57
VACUUM FLASH F-1 6mm Hg
L.E.C. F-2 18
COLUMN F-3 x
H.E.C. F-4 x

ADJUSTED MATERIAL BALANCE

MASS RATES AND RUN CONDITIONS



PROJECT LIGNITE
Summary

Updated: 7/1/76

Run No: M-2C
Date: 2/19/76
Time: 0830-1730

ADJUSTED MATERIAL BALANCE

LBS/HOUR

Temperature: 700°F
Pressure: 1500 psig
LHSV: 0.71
GHSV: 162

Quantitative

Stream Description	Total In	Total Out	Net Yield		Wt % of MAF Coal	
Sample Point						
Pressure (psig)						
Temperature (°F)						
Hydrogen - H_2	1.17	1.01	-0.16			
Carbon Monoxide - CO	17.02	12.57	-4.45			
Carbon Dioxide - CO_2		7.00	7.00			
Hydrogen Sulfide - H_2S		0.09	0.09	2.76	8.17	
Methane - CH_4		0.20	0.20			
Ethane - C_2H_6		0.10	0.10			
Propane - C_3H_8		0.005	0.005			
Butane - C_4H_{10}						
Nitrogen - N_2	0.10	0.07	-0.03			
Ammonia - NH_3		0.005	0.005			
Light Oil (0-100°C @ 1.6 Torr)	6.53	6.64	0.11			
Solvent (100-230°C @ 1.6 Torr)	78.50	67.51	-10.99			
Heavy Oil (230-255°C @ 1.6 Torr)	2.09	7.94	5.85	18.02	53.33	
SRL		23.05	23.05			
Coal (MAF)	33.79	14.05	-19.74		-58.42	
Ash	2.84	3.06	0.22		0.65	
Water	14.75	13.49	- 1.26		- 3.73	
Total	156.79	156.79	0.00		0	

Elemental

Carbon	108.14	109.44	1.30			
Hydrogen	12.28	12.17	-0.11			
Nitrogen	0.60	0.74	0.14			
Sulfur	1.98	1.98	0.00			
Oxygen (by diff)	30.95	29.40	-1.55			
Ash	2.84	3.06	0.22			
Total	156.79	156.79	0.00			

Prepared by:

PROJECT LIGNITE
ADJUSTED MATERIAL BALANCE
(Adjusted by Decreasing Lignite Coal Charged)
Updated: 7/1/76

Run No: M-2C
Date: 2/19/76
Time: 0830-1730

Material In
LBS/HOUR

Temperature: 700°F
Pressure: 1500 psig
LHSV: 0.71
GHSV: 162

Quantitative

Stream Description	Feed Gas	Lignite	Makeup Solvent	Feed Solvent		Total In
Sample Point	Sx-18	Sx-1	Sx-8	Sx-14		
Pressure (psig)	57					
Temperature (°F)	64	RT	RT			
Hydrogen - H_2	1.17					1.17
Carbon Monoxide - CO	17.02					17.02
Carbon Dioxide - CO_2						
Hydrogen Sulfide - H_2S						
Methane - CH_4						
Ethane - C_2H_6						
Propane - C_3H_8						
Butane - C_4H_{10}						
Nitrogen - N_2	0.10					0.10
Ammonia - NH_3						
Light Oil (0-100°C @ 1.6 Torr)			0.62	6.53		6.53
Solvent (100-230°C @ 1.6 Torr)			13.56	78.50		78.50
Heavy Oil (230-255°C @ 1.6 Torr)			6.00	2.09		2.09
SRL						
Coal (MAF)		33.79				33.79
Ash		2.84				2.84
Water		14.75				14.75
Total	18.29	51.38	20.18	87.12		156.79

Elemental

Carbon	7.29	24.20	18.00	76.65		108.14
Hydrogen	1.17	3.43	1.61	7.68		12.28
Nitrogen	0.10	0.36	0.06	0.14		0.60
Sulfur	-	0.31	0.46	1.67		1.98
Oxygen (by diff)	9.73	20.24	0.05	0.98		30.95
Ash		2.84				2.84
Total	18.29	51.38	20.18	87.12		156.79

Note: Makeup solvent included in feed solvent. Prepared by:

PROJECT LIGNITE
ADJUSTED AND MEASURED MATERIAL BALANCE

Updated: 7/1/76

Run No: M-2C
Date: 2/19/76
Time: 0830-1730

Material Out

LBS/HOUR

Temperature: 700°F
Pressure: 1500 psig
LHSV: 0.66
GHSV: 162

Quantitative

Stream Description	Product Gas	K-4 Btms	S-4A Ovhd	S-4B Btms	Cold Trap	S-8 Btms
Sample Point	Sx-20	Sx-38	Sx-30	Sx-23	Sx-33	Sx-16
Pressure (psig)	4.5		60			
Temperature (°F)	71		72			
Hydrogen - H_2	0.98		0.03			
Carbon Monoxide - CO	12.15		0.42			
Carbon Dioxide - CO_2	5.86		1.14			
Hydrogen Sulfide - H_2S	0.07		0.02			
Methane - CH_4	0.19		0.01			
Ethane - C_2H_6	0.09		0.01			
Propane - C_3H_8			0.005			
Butane - C_4H_{10}						
Nitrogen - N_2	0.06		0.01			
Ammonia - NH_3			0.005			
Light Oil (0-100°C @ 1.6 Torr)		0.05		1.97	0.55	2.19
Solvent (100-230°C @ 1.6 Torr)		0.06		2.40	0.05	3.69
Heavy Oil (230-255°C @ 1.6 Torr)		0.01		0.07	0.01	0.15
SRL						
Coal (MAF)						
Ash						
Water		0.35		10.69		2.45
Total	19.40	0.47	1.65	15.13	0.61	8.48

Elemental

Carbon	7.02	0.10	0.51	3.57	0.48	5.03
Hydrogen	1.05	0.05	0.04	1.62	0.07	0.83
Nitrogen	0.06		0.015	0.01		0.02
Sulfur	0.07		0.015	0.05	0.01	0.08
Oxygen (by diff)	11.20	0.32	1.07	9.88	0.05	2.52
Ash						
Total	19.40	0.47	1.65	15.13	0.61	8.48

Prepared by:

PROJECT LIGNITE

MEASURED AND ADJUSTED MATERIAL BALANCE

Updated: 7/1/76

Run No: M-2C
Date: 2/19/76
Time: 0830-1730

Material Out

LBS/HOUR

Temperature: 700°F
Pressure: 1500 psig
LHSV: 0.71
GHSV: 162

Quantitative

Stream Description	S-9 Btms	F-1 Btms	F-2 Btms			Total Out
Sample Point	Sx-24	Sx-2	Sx-13			
Pressure (psig)						
Temperature (°F)						
Hydrogen - H_2						1.01
Carbon Monoxide - CO						12.57
Carbon Dioxide - CO_2						7.00
Hydrogen Sulfide - H_2S						0.09
Methane - CH_4						0.20
Ethane - C_2H_6						0.10
Propane - C_3H_8						0.005
Butane - C_4H_{10}						
Nitrogen - N_2						0.07
Ammonia - NH_3						0.005
Light Oil (0-100°C @ 1.6 Torr)	0.04		1.84			6.64
Solvent (100-230°C @ 1.6 Torr)	0.89	2.42	58.00			67.51
Heavy Oil (230-255°C @ 1.6 Torr)	0.44	0.16	7.10			7.94
SRL		23.05				23.05
Coal (MAF)		14.05				14.05
Ash		3.06				3.06
Water						13.49
Total	1.37	42.74	66.94			156.79

Elemental

Carbon	1.23	33.53	57.97			109.44
Hydrogen	0.11	2.56	5.84			12.17
Nitrogen	-	0.53	0.10			0.74
Sulfur	0.03	0.44	1.29			1.98
Oxygen (by diff)		2.62	1.74			29.40
Ash		3.06				3.06
Total	1.37	42.74	66.94			156.79

Prepared by:

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 7/1/76

Run No: M-2C
Date: 2/19/76
Time: 0830-1730

WEIGHT PERCENT

Quantitative

Sample Description	Feed Lignite	F-1 Btms	Makeup Solvent	F-2 Btms	Feed Solvent	S-8 Oil
Sample Point	Sx-1	Sx-2	Sx -8	Sx-13	Sx-14	Sx-16
Hydrogen - H_2						
Carbon Monoxide - CO						
Carbon Dioxide - CO_2						
Hydrogen Sulfide - H_2S						
Methane - CH_4						
Ethane - C_2H_6						
Propane - C_3H_8						
Butane - C_4H_{10}						
Nitrogen - N_2						
Ammonia - NH_3 (PPM)						
Light Oils (0-100°C @ 1.6 Torr)			3.05	2.75	7.50	36.30
Solvent (100-230°C @ 1.6 Torr)		5.67	67.20	86.65	90.10	61.25
Heavy Oil (230-255°C @ 1.6 Torr)		0.37	29.75	10.60	2.40	2.45
SRL		53.94				
Coal (MAF)	65.77	32.87				
Ash	5.53	7.15				
Water	28.70					
Total	100	100	100	100	100	100
Specific.Gravity 60/60			1.068		1.022	
Viscosity, cp						

Elemental

Carbon	47.10	78.47	89.18	86.60	87.98	83.66
Hydrogen	6.67	5.98	7.97	8.72	8.82	9.21
Nitrogen	0.70	1.23	0.28	0.16	0.16	0.30
Sulfur	0.60	1.04	2.30	1.92	1.92	1.33
Oxygen (by diff)	39.40	6.13	0.27	2.60	1.12	5.50
Ash	5.53	7.15				
Total	100	100	100	100	100	100
Number of Sample Avg.	2	3	2	2	2	2

Prepared by:

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 7/1/76

Run No: M-2C
Date: 2/19/76
Time: 0830-1730

WEIGHT PERCENT

	Sample Description	S-8 Water	S-8 Total	Feed Gas	Product Gas	*S-4B Oil	S-4B Water
	Sample Point	Sx-16	Sx-16	Sx-18	Sx-20	Sx-23	Sx-23
Quantitative	Hydrogen - H_2			6.42	5.03		
	Carbon Monoxide - CO			93.03	62.61		
	Carbon Dioxide - CO_2				30.20		
	Hydrogen Sulfide - H_2S				0.38		
	Methane - CH_4				0.97		
	Ethane - C_2H_6				0.50		
	Propane - C_3H_8						
	Butane - C_4H_{10}						
	Nitrogen - N_2			0.55	0.31		
	Ammonia - NH_3 (PPM)	(29)					(343)
	Light Oils (0-100°C @ 1.6 Torr)		25.78			44.37	
	Solvent (100-230°C @ 1.6 Torr)		43.49			53.91	
	Heavy Oil (230-255°C @ 1.6 Torr)		1.74			1.72	
	SRL						
	Coal (MAF)						
	Ash						
	Water	100	28.99				100
	Total	100	100	100	100	100	100
	Specific Gravity 60/60				0.627		
	Viscosity, cp						

Elemental	Carbon		59.41	39.87	36.20	80.28	
	Hydrogen	11.11	9.76	6.42	5.39	9.78	11.11
	Nitrogen		0.21	0.55	0.31	0.24	
	Sulfur		0.94		0.36	1.07	
	Oxygen (by diff)	88.89	29.68	53.16	57.74	8.63	88.89
	Ash						
	Total	100	100	100	100	100	100
	Number of Sample Avg.	2	2	1	1	2	2

***Corrected for water and normalized**

Prepared by:

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 7/1/76

Run No: M-2C
Date: 2/19/76
Time: 0830-1730

WEIGHT PERCENT

Quantitative

Sample Description	S-4B Total	**S-9 Btms	S-4A Ovhd	J-1 Discharge	Cold Traps	K-4 Btms
Sample Point	Sx-23	Sx-24	Sx-30	Sx-32	Sx-33	Sx-38
Hydrogen - H_2			1.81			
Carbon Monoxide - CO			25.46			
Carbon Dioxide - CO_2			69.32			
Hydrogen Sulfide - H_2S			0.90			
Methane - CH_4			0.79			
Ethane - C_2H_6			0.69			
Propane - C_3H_8			0.16			
Butane - C_4H_{10}						
Nitrogen - N_2			0.79			
Ammonia - NH_3 (PPM)			0.08			
Light Oils (0-100°C @ 1.6 Torr)	13.03	2.70			90.30	11.25
Solvent (100-230°C @ 1.6 Torr)	15.83	65.20			7.80	13.30
Heavy Oil (230-255°C @ 1.6 Torr)	0.50	32.10			1.90	0.45
SRL						
Coal (MAF)						
Ash						
Water	70.64					75.00
Total	100	100	100	100	100	100
Specific Gravity 60/60						
Viscosity, cp						

Elemental

Carbon	23.57	89.60	31.09		79.39	21.25
Hydrogen	10.72	7.92	2.24		11.35	10.64
Nitrogen	0.07	0.06	0.86		0.05	0.05
Sulfur	0.31	2.16	0.85		0.41	0.02
Oxygen (by diff)	65.33	0.26	64.96		8.80	68.04
Ash						
Total	100	100	100		100	100
Number of Sample Avg.	2	1	2		1	2

**Estimated from M-2A data

Prepared by:

PROJECT LIGNITE
SAMPLE ANALYSIS

Updated: 7/1/76

Run No: M-2C
Date: 2/19/76
Time: 0830-1730

MOLE PERCENT

Quantitative

Sample Description	Feed Gas	Product Gas	S-4A Ovhd			
Sample Point	Sx-18	Sx-20	Sx-30			
Hydrogen - H_2	49.00	45.40	25.65			
Carbon Monoxide - CO	50.70	40.40	25.80			
Carbon Dioxide - CO_2		12.40	44.70			
Hydrogen Sulfide - H_2S		0.20	0.75			
Methane - CH_4		1.10	1.40			
Ethane - C_2H_6		0.30	0.65			
Propane - C_3H_8			0.10			
Butane - C_4H_{10}						
Nitrogen - N_2	0.30	0.20	0.80			
Ammonia - NH_3 (PPM)		(244)	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						
Total	100	100	100			
Specific Gravity 60/60						
Viscosity, cp						

Elemental

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

Prepared by:

PROJECT LIGNITE
MEASURED MATERIAL BALANCE

Updated: 7/1/76

Run No: M-2C
Date: 2/19/76
Time: 0830-1730

Material In

LBS/HOUR

Temperature: 700°F
Pressure: 1500 psig
LHSV: 0.66
GHSV: 162

Quantitative

Stream Description	Feed Gas	Lignite	Makeup Solvent	Feed Solvent		Total In
Sample Point	Sx-18	Sx-1	Sx-8	Sx-14		
Pressure (psig)	57					
Temperature (°F)	64	RT	RT			
Hydrogen - H_2	1.17					1.17
Carbon Monoxide - CO	17.02					17.02
Carbon Dioxide - CO_2						
Hydrogen Sulfide - H_2S						
Methane - CH_4						
Ethane - C_2H_6						
Propane - C_3H_8						
Butane - C_4H_{10}						
Nitrogen - N_2	0.10					0.10
Ammonia - NH_3						
Light Oil (0-100°C @ 1.6 Torr)			0.62	6.53		6.53
Solvent (100-230°C @ 1.6 Torr)			13.56	78.50		78.50
Heavy Oil (230-255°C @ 1.6 Torr)			6.00	2.09		2.09
SRL						
Coal (MAF)		27.84				27.84
Ash		2.34				2.34
Water		12.15				12.15
Total	18.29	42.33	20.18	87.12		147.74

Elemental

Carbon	7.29	19.94	18.00	76.65		103.88
Hydrogen	1.17	2.82	1.61	7.68		11.67
Nitrogen	0.10	0.30	0.06	0.14		0.54
Sulfur	--	0.25	0.46	1.67		1.92
Oxygen (by diff)	9.73	16.68	0.05	0.98		27.39
Ash		2.34				2.34
Total	18.29	42.33	20.18	87.12		147.74

Note: Make up solvent included in the feed solvent. Prepared by:

SOLID-LIQUID SEPARATION

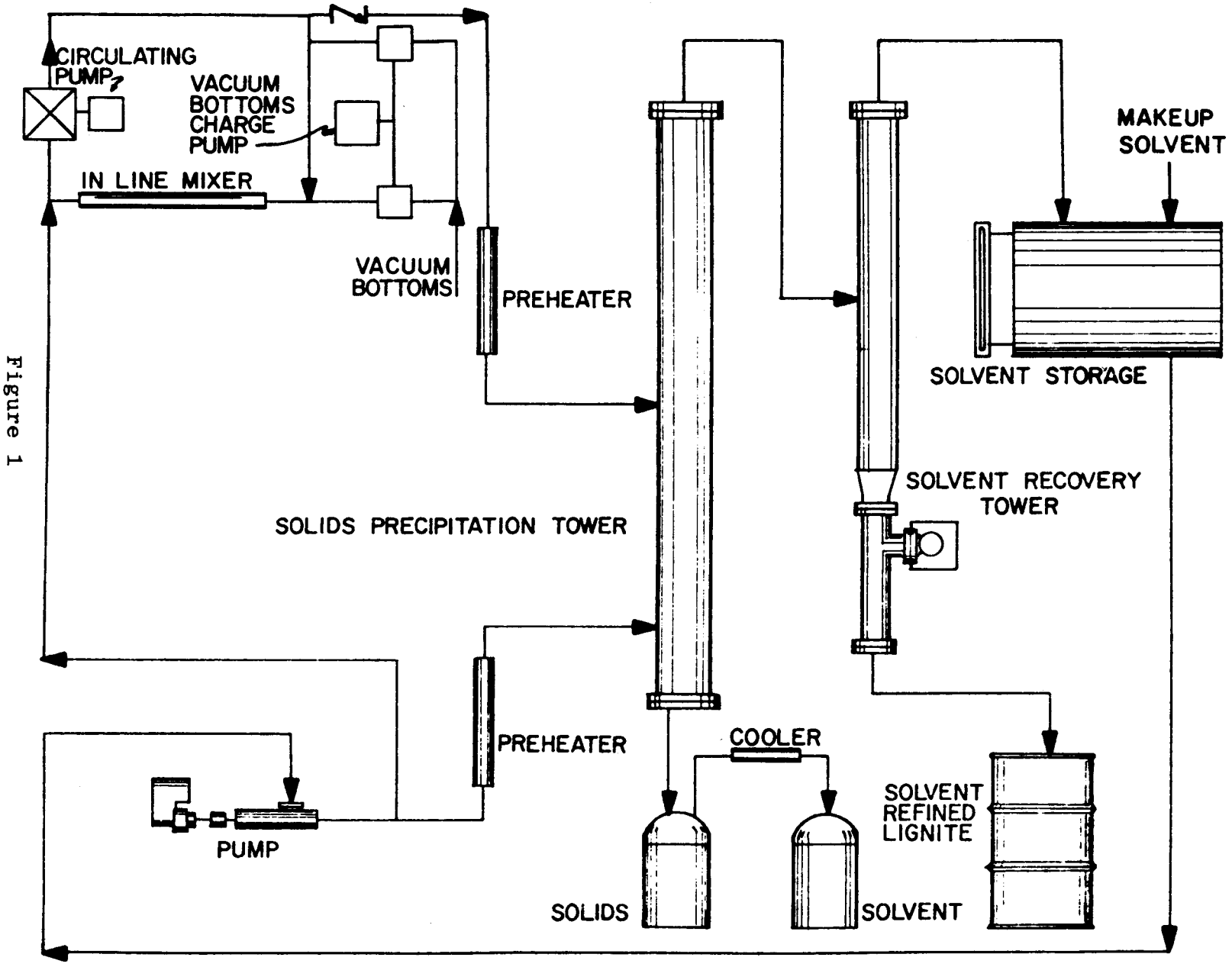


Figure 1

APPENDIX B PART 1
BATCH AUTOCLAVE OPERATIONS
TABLE 1: MATERIAL BALANCE AND YIELD DATA

Run No.	613	614	615	616	617
Test Conditions					
Time, hrs.	0.5	0.5	0.5	0.5	0.5
Avg. Temp. °F	755	752	752	752	752
Max. Press., psia	3095	3085	3095	2985	3065
Gas Charged	CO/H_2	CO/H_2	CO/H_2	CO/H_2	CO/H_2
Starting Solvent	← UNDCAO-73 →				M3A Recycle Solvent
Lignite	← NA 73-2 (B) →				PDU-H Coal
	70 wks N_2 Strg	70 wks Air Strg	70 wks N_2 Strg	70 wks H_2O Strg	
Material In, grams					
Coal	271.9	274.6	274.9	303.2	284.5
Water	51.0	48.6	47.7	27.1	33.9
Solvent	399.7	400.1	399.1	399.6	398.3
Gas	141.5	137.6	141.6	142.3	142.5
Catalyst	-	-	-	-	-
Other	-	-	-	-	-
Total	864.1	860.9	863.3	872.2	859.2
Material Out, grams					
Unfiltered Coal Prod.					500.4
Filtrate	428.9	388.9	451.7	470.4	-
Filter Cake	118.1	85.2	65.2	90.8	-
Residues	43.1	58.5	22.8	24.1	40.4
Gas	203.1	198.6	203.0	213.2	217.5
Water	86.8	87.8	93.6	84.7	80.1
Light Oil	1.2	3.5	2.3	3.3	2.9
Catalyst Gain	-	-	-	-	-
Other	-	-	-	-	-
Total	881.2	822.5	838.6	886.5	841.3
% Recovery	102	95.5	97.1	101.6	97.9
Yields, 100% Rec. Basis, Products, grams					
Light Oil	33.3	41.0	39.3	44.7	3.0
Solvent	365.6	354.7	231.6	350.5	0.0
Vac. Btms	136.3	119.7	150.8	137.9	496.2
Total Liquid	535.2	515.4	521.7	533.1	499.2
Net Liquids	135.5	115.3	122.6	133.5	100.9
Net Gas	57.7	70.3	67.4	67.5	79.7
Net Water	-14.8	-8.0	-3.4	-16.7	-17.8
Net Ash	-1.8	-2.3	0.0	0.9	-1.7
Unconverted Coal (MAF)	23.5	24.9	13.3	14.8	30.3

APPENDIX B PART 1

TABLE 1 CONT.: MATERIAL BALANCE AND YIELD DATA

Run No.	613	614	615	616	617
Yields - Wt.% MAF Coal					
Net Gas	28.8	35.1	33.7	33.8	40.0
Net Liquid	67.7	57.6	61.4	66.8	54.5
Unconverted	11.8	12.4	6.7	7.4	15.2
Net H_2O & Ash	-8.3	-5.1	-1.8	-8.0	-9.7
Total	100.0	100.0	100.0	100.0	100.0
Filtrate Composition, (Wt.%)					
Light Oil	6.0	7.3	7.2	7.8	-
Solvent	68.5	69.3	63.8	66.2	-
Vac. Btms.	25.5	22.4	29.0	26.0	-
Net Liquid Composition					
Net Light Oil	16.7	20.5	19.7	22.3	1.5
Net SRL	51.1	37.1	41.7	44 ¼	53.0
Solvent Recovery					
Wt.%	93.3	84.7	80.7	89.2	-
Wt.% (100% Rec.)	91.5	88.7	83.1	87.7	-

APPENDIX B PART 1

BATCH AUTOCLAVE OPERATION

TABLE 2: ANALYTICAL DATA

Run No.	613	614	615	616	617
Test Conditions					
Time, hrs.	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	755	752	752	752	752
Max. Press., psia	3095	3085	2095	2985	3065
Gas Charged	CO/H_2	CO/H_2	CO/H_2	CO/H_2	CO/H_2
Starting Solvent	← UNDCAO-73 →				M3A Recycle Solvent
Lignite	← NA 73-2 (B) →				PDU-H Coal
	70 wks N_2 Strg	70 wks Air Strg	70 wks N_2 Strg	70 wks H_2O Strg	
Analytical Data					
1. Gas Analysis, mol%					
H_2	44.1	43.8	47.7	43.1	42.9
CH_4	1.0	0.8	0.8	1.5	1.4
CO	24.3	30.0	16.3	16.5	20.0
C_2H_6	0.3	0.2	6.8	0.4	0.5
CO_2	29.9	24.8	28.2	37.2	34.9
C_3H_8	-	-	-	-	-
H_2S	0.44	0.4	0.35	1.4	0.3
Gas Specific Gravity @RT	0.7356	0.7330	0.7378	0.8190	0.7746
2. Input Coal					
Volatile Matter, Wt.%	36.75	36.90	36.79	34.32	36.64
Ash, Wt.%	8.43	8.40	8.37	10.04	6.84
Moisture, Wt.%	17.98	18.67	18.95	24.05	23.09
Fixed Carbon, Wt.%	36.84	36.03	36.89	31.59	33.43
Carbon, Wt.% (1)	65.68	63.19	61.61	61.90	65.34
Hydrogen, Wt.% (1)	4.08	4.18	4.41	4.38	4.18
Sulfur, Wt.% (1)	0.98	0.93	1.00	2.51	-
Nitrogen, Wt.% (1)	0.86	0.85	0.84	0.79	0.85
Oxygen, Wt.% (diff) (1)	18.12	20.52	21.81	17.20	-
3. Input Solvent					
Ash, Wt.%	0.0	0.0	0.0	0.0	1.95
Carbon, Wt.%	91.12	91.12	91.12	91.12	85.21
Hydrogen, Wt.%	6.13	6.13	6.13	6.13	7.50
Sulfur, Wt.%	0.55	0.55	0.55	0.55	1.74
Nitrogen, Wt.%	0.98	0.98	0.98	0.98	0.06
Oxygen, Wt.% (diff)	1.22	1.22	1.22	1.22	3.54
Specific Gravity, 60/60 F	1.106	1.106	1.106	1.106	1.115
Brookfield Viscosity, cp, RT	22.4	22.4	22.4	22.4	30.91
IR Ratio	1.67	1.67	1.67	1.67	-
4. Coal - Solvent Slurry					
Brookfield Visc., cp, RT	1900.0	709.0	613.8	474.3	
5. Cake					(3)
Ash, Wt.%	16.98	19.24	15.61	29.76	4.63
Carbon, Wt.%	72.62	70.04	73.57	62.43	81.22
Hydrogen, Wt.%	4.83	4.74	4.88	4.40	7.15
Sulfur, Wt.%	1.27	1.69	1.32	3.96	1.47
Nitrogen, Wt.%	0.82	0.86	0.86	0.94	0.62
Oxygen, Wt.% (diff)	3.48	3.43	3.76	-	4.91
Pyridine Solubles, Wt.% (2)	79.00	72.97	80.77	80.94	94.24

APPENDIX B PART 1

TABLE 2 CONT. ANALYTICAL DATA

Run No.	613	614	615	616	617
6. Filtrate					
Ash, Wt.%	0.28	0.16	1.60	0.1	
Carbon, Wt.%	88.99	88.64	88.34	88.06	
Hydrogen, Wt.%	6.28	6.18	6.19	6.80	
Sulfur, Wt.%	0.44	0.52	0.57	0.59	
Nitrogen, Wt.%	1.20	1.40	0.94	1.20	
Oxygen, Wt.% (diff)	2.81	3.10	2.36	3.75	
Specific Gravity, 60/60 F	1.136	1.0379	1.1208	1.1205	
Brookfield Viscosity, cp, @RT	1156.7	809.2	1675	1092	
Blackness, abs/g./100ml @550 mu	9.49	8.60	8.43	3.67	
7. Vacuum Bottoms					
Ash, Wt.%	0.20	0.27	6.40	0.45	
Carbon, Wt.%	85.80	85.87	79.99	84.22	
Hydrogen, Wt.%	6.60	5.91	5.44	6.57	
Sulfur, Wt.%	0.27	0.36	0.68	0.38	
Nitrogen, Wt.%	1.74	1.75	1.53	1.59	
Oxygen, Wt.% (diff)	5.39	5.84	5.96	6.79	
Melting Point, °F	310.1	318.1	273.9	228.1	
8. Residues					
Ash, Wt.%	2.92	5.65	14.70	17.47	
Carbon, Wt.%	85.76	84.40	73.91	67.42	
Hydrogen, Wt.%	6.80	5.85	5.24	4.80	
Sulfur, Wt.%	0.75	0.67	1.75	3.79	
Nitrogen, Wt.%	0.75	0.47	0.71	0.99	
Oxygen, Wt.% (diff)	3.02	2.96	4.40	5.53	
Pyridine Solubles, Wt.% (2)	91.90	90.64	87.89	85.50	
9. Light Oil					
Carbon, Wt.%	87.49	88.31	88.87	-	
Hydrogen, Wt.%	8.06	8.08	8.18	-	
Sulfur, Wt.%	-	0.32	0.57	0.51	
Nitrogen, Wt.%	-	0.53	0.57	1.08	
Oxygen, Wt.% (diff)	-	2.76	1.81	-	
10. Product Solvent					
Ash, Wt.%	0.00	0.01	0.05	0.00	
Carbon, Wt.%	90.64	90.75	90.65	90.38	
Hydrogen, Wt.%	6.22	6.24	6.30	6.50	
Sulfur, Wt.%	0.62	0.49	0.53	0.53	
Nitrogen, Wt.%	0.13	0.15	0.15	0.19	
Oxygen, Wt.% (diff)	2.39	2.36	2.30	2.40	
Specific Gravity, 60/60 F	1.1027	1.0984	1.1047	1.1004	
Brookfield Visc., cp, @RT	28.4	31.2	36.0	35.2	
IR Ratio	0.97	0.97	1.13	0.84	

(1) Moisture - free basis
(2) Ash - free basis
(3) Unfiltered Coal Product

TABLE 3: DEASHING BY SOLVENT EXTRACTION AND SETTLING

Run No.	BEX-19	BEX-20
Extraction Solvent	Toluene	Toluene
Coal Feedstock	Vac Btms from CLR-7 & CLR-8	Wilsonville SRC
Temperature, °F	500	500
Pressure, PSIG	550	550
Mixer Type	double-blade	double-blade
Mixing Time, hrs	0.5	0.5
Settling Time, hrs	2.5	2.5

Material In, grams		
Coal Feedstock	194.9	195.0
Extraction Solvent	780.8	780.9
Total	975.7	975.9

Material Out, grams	Total	Solvent	Vac.Dist.	Vac.Btms.	Total	Solvent	Vac.Dist.	Vac.Btms.
Upper Layer No. 1	445.9	392.3	2.9	50.7	459.5	397.6	4.9	57.0
Upper Layer No. 2	191.9	168.2	3.3	20.4	167.8	144.4	2.6	20.8
Upper Layer No. 3	70.6	61.6	0.3	8.7	99.2	84.7	1.1	13.4
Upper Layer No. 4	12.6	11.7	0.2	0.7	30.3	26.1	1.4	2.8
Pot Residue	208.7	95.6	113.1		152.7	65.2	87.5	
Total	929.7	729.4	200.3		909.5	718.0	191.5	
% Recovery	95.29	93.42	102.77		93.20	91.95	98.21	

Corrected to 100% Recovery, grams	Extraction Solvent	Vac.Dist. Heavy Oil	Vac.Dist. Solid	Extraction Solvent	Vac.Dist. Heavy Oil	Vac.Dist. Solid
Upper Layer No. 1	420.0	2.8	49.3	432.4	5.1	58.0
Upper Layer No. 2	180.1	3.2	19.9	157.1	2.6	21.2
Upper Layer No. 3	65.9	0.3	8.5	92.1	1.1	13.6
Upper Layer No. 4	12.5	0.2	0.7	28.4	1.4	2.9
Pot Residue	102.3	110.0		70.9	89.1	
Total	780.8	194.9		780.9	195.0	

Coal Feedstock Components, Grams	Vac.Dist. Heavy Oil	Vac.Btms. Solid	Unconverted	Ash	Vac.Dist. Heavy Oil	Vac.Btms. Solid	Unconverted	Ash
Upper Layer No. 1	2.8	49.15	-	0.15	5.1	57.99	-	0.01
Upper Layer No. 2	3.2	19.80	-	0.10	2.6	21.17	-	0.03
Upper Layer No. 3	0.3	8.40	-	0.10	1.1	13.53	-	0.07
Upper Layer No. 4	0.2	0.63	-	0.07*	1.4	2.61	-	0.29*
Pot Residue	71.10		18.64	20.26	16.71		49.45	22.94
Total	155.58		18.64	20.68	122.21		49.45	23.34
Wt% in Upper 4 Layers	54.30		-	2.73	86.33		-	1.71
Coal Feedstock by Analysis, gms	153.51		18.24	23.15	148.80		20.73	25.47
Wt% of Input Material Extracted	55.03		-	1.90	70.90		-	1.57

Ash Content, Wt%		
V.B. Solid from Upper Layer No. 1	0.30	0.01
V.B. Solid from Upper Layer No. 2	0.48	0.14
V.B. Solid from Upper Layer No. 3	1.12	0.52
V.B. Solid from Upper Layer No. 4	-	-
Pot Residue (Solvent-free)	18.42	25.75
Coal Feedstock	11.88	13.06
Pyridine Solubles, Wt% (Ash-free)		
Pot Residue	79.25	25.24
Coal Feedstock	89.38	87.77

*Estimated for material balance calculations.