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THE EFFECT OF
HYDROGEN PARTIAL PRESSURE ON LIGNITE LIQUEFACTION IN A TWO-STAGE HOT-CHARGE BATCH AUTOCLAVE SYSTEM

by
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Bachelor of Science in Chemistry in Education
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A Thesis

Submitted to the Graduate Faculty

of the

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for the degree of

Master of Science

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1984

This thesis submitted by Robert O. Ness, Jr. in partial fulfillment of the requirements for the degree of Master of Science from the University of North Dakota is hereby approved by the faculty advisory committee under whom the work has been done.

Thomas C. Owens

R. Hasan.

This thesis meets the standards for appearance and conforms to the style and format requirements of the Graduate School of the University of North Dakota, and is hereby approved.

A. William Johnson 11-15-84

Title: The Effect of Hydrogen Partial Pressure on Lignite Liquefaction in a Two-Stage Hot-Charge Batch Autoclave System

Department Chemical Engineering

Degree Master of Science

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Signature Robert O Ness Jr.

Date Oct 11, 1984

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And last I wish to dedicate this thesis to my parents for their patience and support through my years of education.

ABSTRACT

The effect of varying hydrogen partial pressure in a two-stage liquefaction process on conversion of North Dakota lignite to THF solubles was studied using the UND hot-charge, time sample, batch autoclave system. Approximately one hundred and fifty grams of moisture- and ash-free (MAF) Zap lignite, three hundred grams of AO4 (Anthracene oil) solvent and seventy five grams of water were reacted in the autoclave unit. Carbon monoxide was used as feed gas for the first stage and varying amounts of hydrogen and carbon monoxide were utilized in the second stage. First stage temperatures ranged from 358°C to 370°C while the second stage temperatures were 427°C to 444°C. Residence times in the first and second stages were 20 and 60 minutes, respectively. Liquid and gas samples were taken at one, three, six, ten, twenty, forty, and sixty minutes during the second stage run period.

Overall conversion ranged from 68.7 to 85.0 percent while the distillate yield varied from -7.9 to 25.0 percent based on the MAF coal charge. The soluble residuum yield rose with increasing hydrogen partial pressure from 20.1 to 39.3 percent. Light and middle oil production increased directly with hydrogen partial pressure. When the second stage feed gas consisted of pure carbon monoxide, the heavy oil yield was -15.2 percent. With increasing hydrogen content of the feed gas, the heavy oil yield ranged from -15.2 to 3.2 percent and when 100 percent hydrogen feed gas was used in the second stage, a 3.9 percent yield was produced. Conversion reached a maximum after approximately ten minutes in the second stage. Results indicated that syngas may be a viable feed gas if yields can be increased, perhaps by adding hydrogen sulfide or using a hydrogen donating solvent.

Chapter I

INTRODUCTION

With the world wide availability and convenience of crude oil, coal use in the United States has substantially decreased over the past several decades. However, coal has recently become of interest as a source of energy and feedstock material due to the possibility of oil shortages caused by embargoes, political unrest, and diminishing reserves (1)[1]. One area of coal research is the conversion of coal to liquid fuel which, like crude oil, is more desirable than solid fuel because of convenience and high energy density.

The first practical method for converting coal to liquids was achieved by Bergius and colleagues (2). His work involved reacting pulverized coal-oil slurries with gaseous hydrogen at high temperatures and pressures. The main objective was to raise the H/C ratio from approximately 0.7 to 1.2, which is the ratio for petroleum. Overall conversion from the Bergius process ranged in the area of 50 percent conversion to middle oils and 40 percent to heavy oils. With further studies and research, "second generation" liq-

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[1] The underlined numbers in parenthesis refer to references cited at the end of this report.

uefaction processes have shown great promise for reducing cost and achieving greater efficiency (3). Overall conversion of coal to oils and C_1-C_3 hydrocarbon gases is better than 90 percent using less harsh conditions than those of the Bergius process. Processes have been under development by Exxon, Gulf, Lummus, Consolidation Coal, and others to provide a cost effective method of liquefaction.

Another process developed by the Pittsburgh Energy Technology Center (DOE) is the COSTEAM process, which uses the water-gas shift reaction (WGSR) as a source of hydrogen. The cost of the feed gas for the process is reduced by using syngas instead of pure hydrogen. Overall conversion is also assisted by the inherent moisture of the coal.

The basis for this study of hydrogen partial pressure effects on liquefaction lies in using a modification of the COSTEAM process. A low temperature stage using pure carbon monoxide as the reductant was followed by a higher temperature second stage using a carbon monoxide/hydrogen atmosphere. The composition of the second stage atmosphere was the primary variable of the study. By utilizing the two stage system, the reaction time should be reduced from 60 minutes as in the Bergius method to less than 30 minutes. The shorter reaction time may be attributed to carbon monoxide stabilizing the reactive intermediates which were produced by thermal decomposition of lignite at low temperatures (370°C). The intermediates were then thermally cracked and "capped" with hydrogen at high temperatures (440°C). Reducing the reaction time and using syngas may reduce operating costs and may make lignite liquefaction a more economically viable process.

Chapter II

HISTORY OF PREVIOUS WORK

Present coal technology has descended from work, called hydroliquefaction, developed by Bergius in Germany starting around the beginning of World War I, in which hydrogen was added directly from the gas phase (2). Direct liquefaction may be modeled, simplistically, by the relation: (4)

$$C + .8H_2 = CH_{1.6} \quad (1)$$

At liquefaction temperatures, this reaction is slightly exothermic so thermal efficiency is high; however, the reaction proceeds at a very slow rate. The hydrogen that must be supplied accounts for an important fraction of the energy consumed and, thus, the cost of the process increases in proportion to the hydrogen used in the process. Hydrogen not only is used in the liquefaction process but is also consumed in reducing oxygen, sulfur, and nitrogen in the coal. Because hydrogen is used as a reductant, the overall thermal efficiency is below thermal neutrality, the point where heat must be added for the reaction to occur.

Besides hydrogen consumption, the reaction time of the coal is an important variable in liquefaction. It is generally quite long, often in excess of one hour. Conversion of coal to heavy asphaltenes is very rapid. Long residence times may reduce asphaltene yields, but they may also reduce oil yields because of thermal decomposition.

The processes currently receiving attention are primarily "second generation" systems (3,5). Temperatures range from 450°C to 475°C and pressures from 10 to 20 MPa with some up to 30 MPa (1400 to 4250 psia) are used. These high pressures are necessary to prevent excessive production of char and gas. Conversions range from 90 to 95 percent of MAF coal charged. Distillate constitutes 50 to 60 percent of the product. Earlier processes, such as that of Bergius, required pressures of 70 MPa and produced mostly distillate.

There are several methods presently used to achieve liquefaction. One of these is the H-Coal process which centers around a unique ebullated bed of cobalt molybdate on alumina (3,5). Hydrogenation takes place at approximately 455°C and 2500 psi. Overall conversion of coal to oils and C_1-C_3 hydrocarbons exceeds 90 percent.

The Exxon Corporation has developed the EDS (Exxon Donor Solvent) process which liquefies coal by noncatalytic hydrogen transfer from recycle oil and regenerates the donor by hydrotreating and returning a portion of the coal liquids (3,5). The main reaction takes place at approximately 425°C to 465°C. The pressure ranges from 10.5 to 14.0 MPa (1500-2000 psi).

Other processes include SRC-II (Solvent Refined Coal), CCL (Catalytic Coal Liquids), both developed by Gulf Oil, the CFFC (Clean Fuels from Coal), researched by Lummus, and CSF (Consolidated Synthetic Fuels) sponsored by Consolidation Coal Company (3,5). All of these processes use hydrogen as the feed gas and process bituminous or sub-bituminous coals.

In 1968, the Pittsburgh Energy Technology Center (PETC) began testing a process to liquefy low rank coals (3,5). In place of hydrogen as the main component in the feed gas, carbon monoxide and water were substituted. Results of the study indicated the reaction time could be reduced from 60 minutes to 10 minutes and operating temperatures could be reduced to the range of 380°C to 400°C (6). It was postulated that the increased rates of conversion and enhanced reactivity of the coal were due to:

1. hydrogenation with activated hydrogen produced by the water-gas shift reaction,
2. introduction of alkyl groups; and,
3. the unique ability of carbon monoxide to cleave certain types of bonds or to inhibit condensation reactions leading to benzene-insoluble materials (7).

Later work done at UNDERC (University of North Dakota Energy Research Center) has indicated that carbon monoxide provides stability to free radicals and prevents undesirable condensates (8). Possible radical intermediates that occur in thermal decomposition are the formate ion, which reduces carbonyl groups to alcohol, the formic acid group, isoformate, and the carbon monoxide radical anions. Carbon dioxide also shows a tendency to attack cross linkage bonds (8).

Studies using synthesis gas were initiated at PETC to reduce cost and improve physical and molecular weight characteristics of the product slurry. Temperatures of 450°C and pressures of 3000 psi were found to be desirable (9). A continuous process unit (CPU) was then developed using 0.7 H_2:1.0 CO and 3.0 H_2:1.0 CO mole ratios of synthesis gas (10). Increasing the temperature above 400°C was reported to adversely affect distillate yields using the 0.7 H_2:1.0 CO synthesis gas. However, distillate yields increased with increasing temperature using the 3.0 H_2:1.0 CO synthesis gas.

Work on solvent-hydrogenation of lignite at the University of North Dakota started in 1965 under the sponsorship of the Great Northern Railway Company (now Burlington Northern) (11). From 1965 to 1970, batch autoclave runs using carbon monoxide and hydrogen with various catalysts and solvents were carried out to provide data for the construction of a process development unit (PDU). A five year contract between the UND Department of Chemical Engineering and the U.S.D.I. Office of Coal Research was negotiated and a comprehensive research program on lignite technology was developed (11). This program, called Project Lignite, operated a 0.6 ton coal/day continuous PDU. The process used a continuous donor solvent in a carbon monoxide and/or hydrogen atmosphere to produce low sulfur, low ash fuels (12,13). The distillate bottoms were known as SRL (Solvent Refined Lignite) and could be upgraded to premium liquid fuel or used directly as boiler fuel.

Research on the COSTEAM process has continued using a single stage batch autoclave, time-sample system (14,15). Runs have been made using carbon monoxide and hydrogen over the temperature range of 350°C to 480°C (16). The research for this thesis was based on the previous work done on the single stage autoclave system.

Chapter III

EXPERIMENTAL PROCEDURE AND CALCULATIONS

3.1 MATERIALS

The lignite used in the experiment was from the Indian Head Mine of the North American Coal Company located near Zap, North Dakota. The size distribution, proximate analysis, and ultimate analysis are shown in Table 1. The solvent used in the feed slurry was an anthracene oil from batch number four (AO4) obtained from Crowley Tar and Chemical Company of New York. Analysis of the solvent is shown in Table 2.

The carbon monoxide, hydrogen, and syngas were obtained in 1700 psig cylinders from Dow Supply Company. Analysis of the feed gas stock is given in Table 3.

3.2 EQUIPMENT

The equipment used was UND's two-stage, hot-charge, time sample, batch autoclave system. The system was a modified form of the single-stage system described by Rindt, Severson, and Souby (17).

TABLE 1

Analysis of Lignite

Size Distribution

U.S. Screen Size	Percent Passing
60 mesh	100.0
100 mesh	97.5
150 mesh	87.3
170 mesh	75.7
200 mesh	62.2

Proximate Analysis[a]

Constituent	Percent
Volatile Matter	32.18
Moisture	29.08
Fixed Carbon	30.47
Ash	8.27
Total	100.00

[a] as received basis

Ultimate Analysis[b]

Constituent	Percent
Carbon	61.52
Hydrogen	3.78
Nitrogen	0.61
Sulfur	0.74
Oxygen	20.95
Ash	11.10

[b] moisture free basis

TABLE 2

Analysis of Solvent AO4

Solvent	AO4[a]
ASTM D-1160 Distillation at 5 torr	
IBP, °C	94
Vol. % off at, °C	
10	146
20	163
30	175
40	185
50	195
60	205
70	217
80	231
90	251
95	276
Max. Temp., °C	288
Vol. % off at Max. Temp.	97
Calculated from ASTM D-1160	
IBP - 120°C Fraction Wt. %	3.1
120 - 260°C Fraction Wt. %	85.1
260 - Max. Temp. Fraction Wt. %	7.6
Vacuum Bottoms, Wt. %	4.3
Density, gm/ml at RT	1.1
Element Analysis	
Carbon, Wt. %	90.66
Hydrogen, Wt. %	6.38
Nitrogen, Wt. %	0.78
Sulfur, Wt. %	0.59
Oxygen, Wt. %	1.59
H/C Ratio	0.79

[a] AO4 is as received anthracene oil from Crowley Tar & Chemicals.

TABLE 3

Analysis of Syngas

Component	Mole Percent
H_2	50.195
CO	49.805

Figure 1 shows a simplified schematic of the two stage system. Figure 2 shows a photograph of the autoclave system.

The slurry charge vessel, rated at 10,000 psig at room temperature, was a one gallon stainless steel modified accumulator equipped with a movable piston. The feed gas system was made of two 2.5 gallon piston accumulators also rated at 10,000 psig. The first stage reactor was a one liter stainless steel autoclave rated at 5800 psia at 343°C. The second stage reactor was a one gallon vessel rated at 5100 psia at 510°C. Both autoclaves were equipped with magnetic stirrers and have the capability of sampling liquid and gas from the system during the run. A one gallon autoclave served as a quench vessel for the system. A Perkin-Elmer Gas Chromatograph was used to analyze gas samples. All major valve operations made during the run were controlled using a Gould P-180 computer.

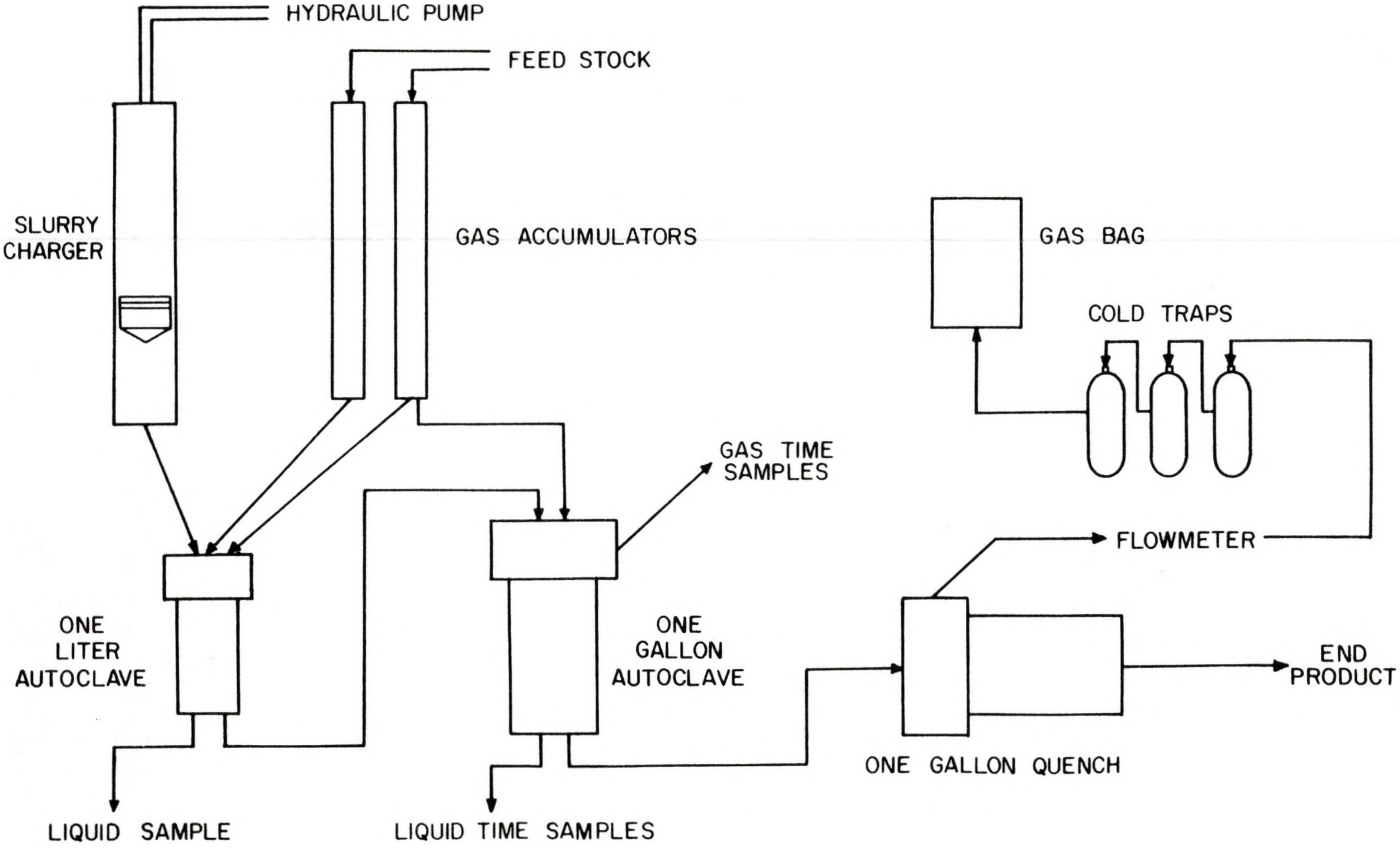
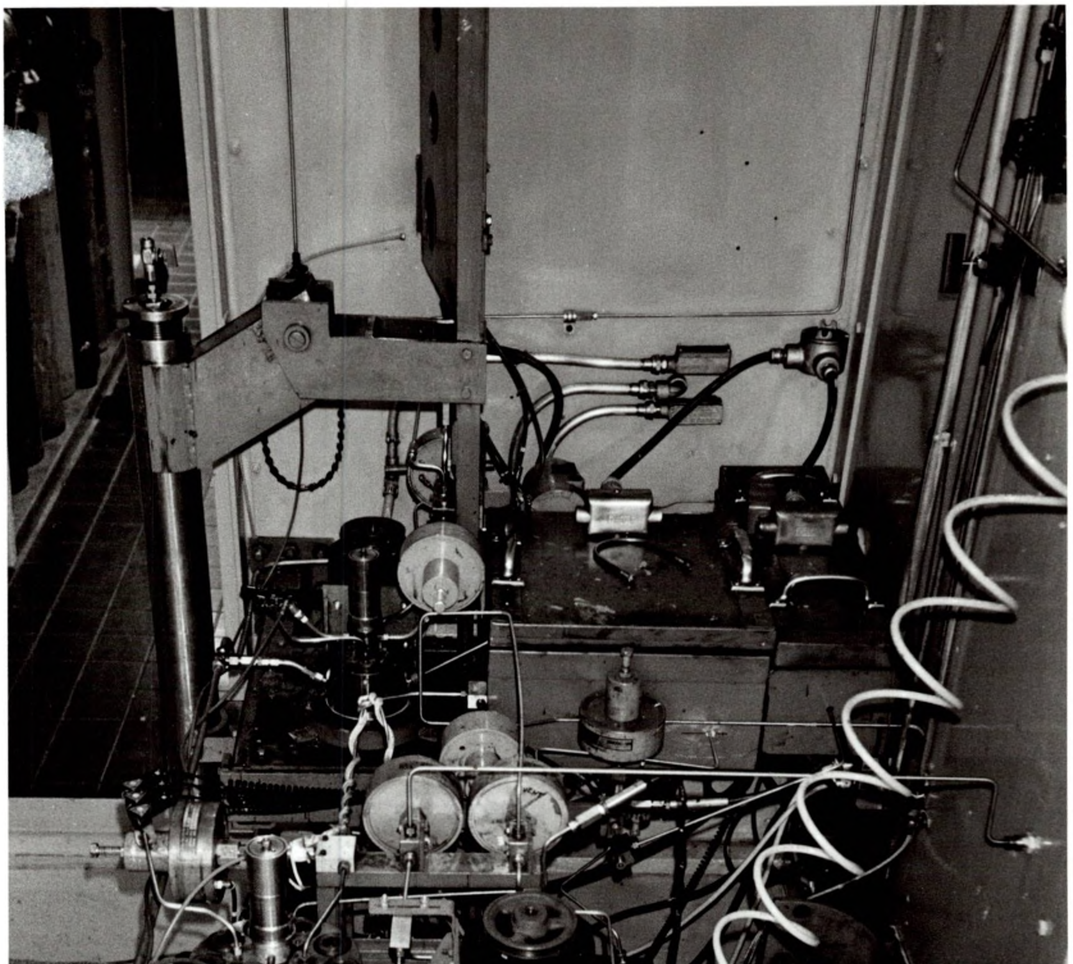


Figure 1: Block Diagram of Two-Stage System

Figure 2: Photograph of Two-Stage System



3.3 SLURRY PREPARATION

Because the moisture content of the coal depended on the relative humdity of the air, a moisture test was done before the slurry was made up. The procedure is found in Appendix A. Ash composition for the slurry was also calculated (See Appendix B). The A04 was heated prior to adding coal and water to ensure that all components were in liquid form. The A04 storage can was shaken by hand to thoroughly mix all of the constituents. The slurry was prepared in the ratio of 4:2:1 by weight with respect to the solvent, lignite, and water. The slurry makeup was nominally 310 grams A04, 155 grams MAF lignite, and 77.5 grams H_2O.

3.4 FEED GAS

Each of the two autoclaves used a separate accumulator to supply feed gas to the system. Both accumulators were evacuated with the pistons at the top of the cylinders to remove all residual gases. Gas was fed into each cylinder from feed gas stock until the piston had been pushed to the bottom of the cylinder and a predetermined pressure had been reached. The pressure in the first stage accumulator was 100 psig and the pressure in the second stage accumulator was 350 psig. Under room temperature and the accumulator pressures approximately 10.5 moles of gas was charged into the system. Calculations were done using the "GASCALC" program found in Appendix C. This program took into account the compressibility of the feed gas plus correction factors for the void volume of the lines and cylinders. These correction factors were found by Rindt (18). All of the first stage gas and fifty percent of the second stage gas was charged prior to the start of the run.

3.5 PROCEDURE FOR STAGED OPERATION

The first stage autoclave was preheated to 393°C, 33° above the run temperature so the cold slurry would reach the reaction temperature in a relatively short time. Fifty percent of the second stage feed gas was charged in the second stage reactor and preheated to 451°C. The slurry was then injected into the first stage reactor at 5,000 psig along with the first stage feed gas. The reaction time began when the liquid temperature was within 20°C of the desired run temperature. The residence times for the first and second stages were 20 and 60 minutes, respectively. Temperatures were maintained using on-off controllers that supplied current to the autoclave heaters. Manual control was necessary until the liquid reached a stable heating cycle. At the end of the 20 minute first stage reaction period, liquid and gas samples were taken and the reaction product was released into the second stage reactor. The final portion of the second stage feed gas was supplied by injection through the first stage reactor. The first stage reactor was thus used as a preheater so the second stage run temperature could be achieved. Previous runs on the two stage system had charged 9.5 moles of gas (19). In order to provide the amount of gas used in previous runs and include the loss due to the void volume of the first stage reactor, 10.5 moles of gas were injected. Gas and liquid samples were taken at one, three, six, ten, twenty, forty, and sixty minutes during the run. The gas sample volume was measured by an inline temperature compensated flowmeter. After the final sample was taken, the liquid product was released into the quench vessel. The gas from each stage was withdrawn and measured using the inline flowmeter. Three cold traps were also placed inline to condense water and light oils from the exiting gas. The first of the cold traps was immersed in an ice bath while the next two were placed in an isopropanol-dry ice mixture.

3.6 MATERIAL RECOVERY

3.6.1 Gas Recovery

The amount of feed gas loaded into the two accumulators was measured using pressure transducers. After the run, a two liter sample was taken from the first stage reactor. The second stage reactor gas was collected in a gas bag after the gas passed through the flowmeter. A product gas composition was calculated using the program "PRODGAS". The product gas calculation program took into account residual gas left in the autoclave and added in an amount based on the reactor temperature and volume. "PRODGAS" can be found in Appendix C.

3.6.2 Liquid Recovery

All containers that transported the slurry charge or end product were weighed before and after use. The difference in weight was added to the weight of the material lost from the system during the run.

After completing a run, the charger vessel was cleaned with disposable wipes. After all surfaces were cleaned with the wipes, cotton tip swabs were used to remove any residual matter. All material that did not enter the first stage autoclave was subtracted from the initial slurry charge. The two autoclaves were then cleaned with the wipes. All connecting lines, plus lines to the pressure transducers were flushed with THF (Tetrohydrofuran). The material collected was added to the liquid product weight.

The quench vessel was tilted upright and the bottom valve opened to allow liquid to drain into a preweighed can. The sides were then scraped with a spatula and a plunger. All product material was placed into the end product sample can. The sides of the quench vessel were then cleaned with disposable wipes and the net weight was added to the end product weight. The cold traps from the gas lines were cleaned and weighed also.

3.7 ANALYTICAL PROCEDURE

3.7.1 Product Gas Analysis

A Hewlett Packard F and M Scientific 700 Laboratory Chromatograph with Porapak Q and 5 A Mole Sieve columns was used to analyze the product gas. The product gas composition included the following gases: H_2, N_2, O_2, CO, CO_2, CH_4, C_2H_6, and C_3H_8. H_2S was determined using a modified Nessler's method (20). Specific gravity of the gas was determined by the Reanult method using a gas density bulb (21). All of the gas analytical procedures are detailed in Appendix E.

3.7.2 Liquid Product Analysis

The liquid product was stirred to insure a uniform sample was obtained for analysis. Appendix F shows the procedures that were used to determine the distillate, soluble residuum, moisture, and ash content of the product. A standard vacuum distillation test (ASTM D-1160) was used to determine the weight fraction of distillate in the liquid product. The soluble residuum was determined by a THF solubility test. A Karl Fischer Water Determination (PLAM I-3-76T, II-14-76T, and V-A-3-77T) was done to find the moisture content of the liquid product. The ash content of the product was found by using a procedure (PLAM I-4-76) that heats a sample in a muffle furnace at 750°C for four hours.

3.8 MATERIAL BALANCE AND TIME SAMPLE CALCULATIONS

All material balance calculations were done in four parts. First, the mass of each component that was charged into the system was calculated. Next, the mass leaving the system was determined. In the third step, the material balance closure was calculated as a ratio of mass leaving the system to the mass entering the system. The mass that was not recovered was then added to either the gases or liquids or a combination of the two. The mass was added to each individual component in the gas, liquid or a combination of both, in an amount equal to the ratio of the weight of each component to the total weight of the gas or liquid. For example, if ten grams of mass were not recovered, ten grams would be added either to the gas or liquid components or an amount to each so the total was ten grams. For example, six grams could be added to the gas and four grams to the liquid. Of the four grams added to the liquid each component would receive a proportional amount. If the component contained 25 percent of the liquid mass, the component would be increased by 25 percent of four grams or one gram. The other three grams would be distributed accordingly to the remaining constituents of the liquid product. The remaining six grams would be distibuted similarly to the gas components. The proportion of mass assigned to the liquid or gas was determined by elemental balances, or data from previous runs. The fourth step entailed normalizing the weights to a

100 gram MAF coal charge basis. Material balance sample calculations are shown in Appendix D.

The time sample calculations determine the product yields based on the analysis of gas and liquid samples taken during the run period. A correction factor was included in the calculations so the mass of each constituent of the time sample taken at the end of the run equaled the mass of the constituent determined by analysis of the end product. For example, the analysis of the sample indicated the mass of soluble residuum to be 50 grams. Analysis of the end product showed the soluble residuum to be 45 grams. Each of the soluble residuum time samples was then multiplied by the ratio 45/50. A correction factor was calculated for each constituent of the gas and liquid product. The correction factor would include experimental error and error introduced by data obtained from a nonrepresentative sample. The liquid sample may not have been representative of the liquid product because the sample may have been contaminated with liquid from an earlier sample or a homogenous mixture may not have existed throughout the reactor at the time of the sample. Similar problems may occur with the gas samples so correction factors are also included in the gas calculations. The time sample calculations are shown in Appendix G. The programs used for the time sample calculations called "TIMSAM7" and "NORMAN" are found in Appendix C.

Chapter IV

DISCUSSION AND RESULTS

Earlier liquefaction processes, such as the COSTEAM process developed by the PETC for lignite, used pure carbon monoxide while others, such as the Bergius process, used pure hydrogen for higher rank coals. A liquefaction process using syngas, a mixture of approximately 50 mole percent hydrogen and 50 mole percent carbon monoxide, may be more economical because of the higher cost of pure carbon monoxide or of pure hydrogen. The process using syngas, in order to have a lower production cost than a process using pure hydrogen, must either have lower equivalent hydrogen consumption (lower feed gas requirements) and/or have a higher yield of THF-soluble materials, mostly in the form of distillates. Runs using pure carbon monoxide, pure hydrogen, and syngas were made in order to compare overall lignite conversion, equivalent hydrogen consumption, net yields of gas, distillate, THF soluble residuum, moisture, and insoluble organic matter (IOM) and determine the feasibility of using a syngas feed. Appendix D describes the overall material balance calculations while Appendix G shows the time sample material balance calculations. The computer programs used in the data reduction process are shown in Appendix C.

Results of calculations for overall reaction products and for time sample data are shown in Appendix H.

4.1 EXPERIMENTAL CONDITIONS

The pressure, temperature, solvent, run time, time of sampling, and hydrogen content of the feed gas for each run are listed in in Table 4. The hydrogen content of the second stage feed gas was varied while holding the remaining variables constant. The hydrogen content ranged from 0 to 100 percent in 25 percent increments. The first stage reactor utilized a pure carbon monoxide feed gas with the exception of Run N-180 in which pure hydrogen was used. This run was used as a control to verify the importance of carbon monoxide in the feed gas as suggested by previous work (22). One set of duplicate runs, N-174 and N-178, was made. Temperatures for the first stage ranged from 358°C to 371°C with the pressure varying from 3910 psi to 4365 psi. Second stage temperatures varied from 427°C to 444°C, and the pressure ranged from 2275 psi to 2750 psi.

4.2 STATISTICAL ANALYSIS OF OPERATING CONDITIONS

Table 5 summarizes the results and important parameters of the seven experimental runs. A student t-test was performed on the "constant" operating conditions to determine if the operating parameters were similar. The sample calculations for the analysis are given in Appendix I.

TABLE 4

Operating Parameters for Runs N-174 to N-180

Parameters	N-175	N-177	N-179	N-176	N-174	N-178	N-180
Hydrogen in 2nd Stage feed gas (Mole %)	0	25	50	75	100	100	100
Average Temperature (°C)							
1st Stage	365	366	358	365	370	361	359
2nd Stage	440	440	441	440	439	427	440
Maximum Temperature (°C)							
1st Stage	367	368	360	369	371	367	360
2nd Stage	441	444	444	441	440	429	443
Pressure (psi)							
1st Stage	4000	4090	3970	3910	4000	3940	4365
2nd Stage	2385	2555	2525	2365	2275	2490	2750
Solvent	A04	A04	A04	A04	A04	A04	A04
Run Time (Minutes)							
1st Stage	20	20	20	20	20	20	20
2nd Stage	60	60	60	60	60	60	60
Time of Sampling (Minutes)							
2nd Stage	1, 3, 6, 10, 20, 40, 60 for all runs						

Table 5.

Results and Important Parameters of Experimental Runs

RUN #	N175	N177	N179	N176	N174	N178	N180
COAL	ZAP	ZAP	ZAP	ZAP	ZAP	ZAP	ZAP
SOLVENT	A04-A04	A04-A04	A04-A04	A04-A04	A04-A04	A04-A04	A04-A04
ADDITIVE	NONE	NONE	NONE	NONE	NONE	NONE	NONE
RUN TIME (MIN)	20-60	20-60	20-60	20-60	20-60	20-60	20-60
AVG TEMP	365-440	366-440	358-441	365-440	370-439	361-427	359-440
MAX PRESS (PSIG)	4000-2385	4090-2555	3970-2525	3910-2365	4000-2275	3940-2490	4365-2750
% H2 FEED GAS	.0	18.3	38.0	57.1	73.3	76.1	100.0
MOISTURE (GMS)	72.9	73.6	76.1	74.0	73.3	74.1	73.1
NET YIELDS (WT% 100 GRAMS MAF COAL CHARGED)							
CO	-56.3	-37.5	-38.3	-37.7	-29.7	-28.6	8.2
H2	1.7	.9	- 1.1	- .0	- 2.5	- .3	- 3.3
CO2	109.5	80.3	83.1	69.2	64.4	62.9	40.9
C1-C3	4.6	5.0	5.0	6.4	7.2	2.7	6.7
H2S, NH3	1.2	.5	.9	1.1	.4	.8	1.2
H2O	-19.3	-13.2	-15.5	- 6.4	- 6.1	- 4.3	4.7
GAS + H2O	41.4	36.1	34.0	32.6	33.7	33.2	58.4
LT OIL	- .0	2.4	3.1	6.4	5.8	- 1.8	4.7
MID OIL	26.5	33.0	30.5	26.5	11.1	10.5	2.6
HVY OIL	-15.2	- 9.9	-15.2	-15.2	2.3	3.9	-15.2
C4+ DIST	11.3	25.4	18.4	17.8	19.2	12.6	- 7.9
SOL RESIDUUM	26.4	20.1	24.1	29.5	30.6	39.3	18.2
TOTAL LIQUID	37.6	45.5	42.6	47.3	49.9	51.9	10.3
IOM	21.0	18.4	23.4	20.2	16.4	15.0	31.3
H2 EQUIV CONSUMP (Moles)	1.7	1.3	2.9	2.0	3.4	1.7	2.0
IOM/GRAM ASH	8.1	8.1	8.1	8.1	8.2	7.5	8.1
CONVERSION	79.0	81.6	76.6	79.8	83.6	85.0	68.7
MAT BAL CLOSURE	92.3	95.7	92.4	91.1	95.3	94.7	93.0

Runs N-174, N-179, and N-180 were rejected by the analysis for not having similar temperatures in the first stage. Run N-178 was rejected because the second stage temperature was low. Therefore, temperature effects had to be considered in the data interpretation.

Material closures for the runs ranged from 91.1 percent to 95.7 percent.

4.3 EQUIVALENT HYDROGEN CONSUMPTION

Equivalent hydrogen consumption, based on the total grams of MAF coal charged to the system, is defined by the following equation:

$$\text{Equivalent Hydrogen Consumption (Moles)} = \frac{\overset{CO}{\text{(g. consumed)}}}{\text{MW CO}} + \frac{\overset{H_2}{\text{(g. consumed)}}}{\text{MW } H_2} \qquad (2)$$

Figure 3 shows the equivalent hydrogen consumption with time for each run. Run N-175, pure carbon monoxide feed gas, consumed the largest quantity of gas as shown in Figure 3. The consumption dropped considerably with the introduction of a 25 percent hydrogen feed gas in the second stage. Statistical analysis of the runs using 25, 50, 75, and 100 percent hydrogen in the second stage feed gas indicated the equivalent hydrogen consumption increased with increasing hydrogen partial pressure. Appendix I contains the calculations for the statistical analysis done on the equivalent

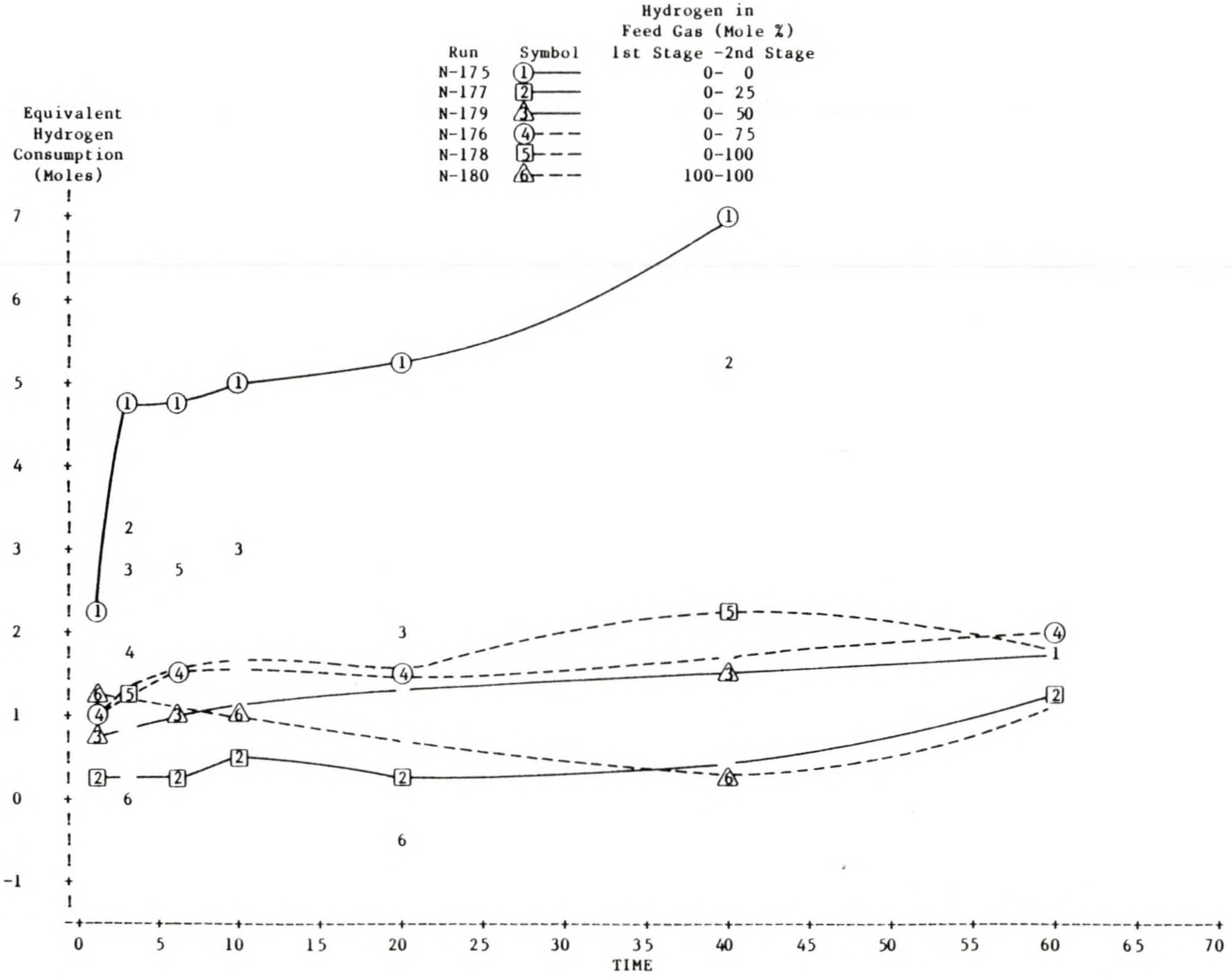


Figure 3. The Effect of Hydrogen Partial Pressure on Hydrogen Equivalent Consumption

hydrogen consumption. Run N-180, having pure hydrogen in both stages, may have been affected by the low first stage temperature. The low temperature could account for the drop in consumption early in the run and the subsequent gradual increase. The most efficient liquefaction process will raise the hydrogen/carbon ratio from approximately 0.7 to 1.2 and keep the equivalent hydrogen consumption to a minimum. From Figure 3, runs, N-176 and N-178, using syngas (50 percent hydrogen) and a 1:1 ratio of syngas to hydrogen (75 percent hydrogen) respectively, show a decrease in the equivalent hydrogen consumption when compared with the pure hydrogen run, N-180. If the THF-soluble yields are not significantly lowered, syngas would be a better feed gas than hydrogen because of the decreased equivalent hydrogen consumption. The proportion of syngas to hydrogen to be used in the feed gas would depend mainly on the product yields desired and the cost of syngas and pure hydrogen.

4.4 EFFECT ON CONVERSION

Conversion is defined as 100 minus the ash-free, THF insoluble material as a weight percent of MAF lignite charge:

$$\text{Conversion} = \frac{\text{MAF Lignite In} - \text{MAF-Insoluble Material Out}}{\text{MAF Lignite In}} \times 100 \quad (3)$$

Overall conversion can be broken into three parts: yields of THF-soluble residuum, distillate, and gas, which includes water. Figure 4 shows overall conversions and yields for the seven experimental runs in this study. Distillate yields can also be broken into three fractions: light, middle, and heavy oils. Figure 5 shows distillate yields for the seven runs for varying hydrogen partial pressures in the reactor. The two bar graphs show that distillate yields increased with the introduction of hydrogen in the second stage reactor. Overall conversion remained fairly constant but gas and soluble residuum decreased to compensate for the increased distillate. The distillate bar graph, Figure 5, shows an increase in light and middle oil production and a considerable decrease in heavy oil consumption with increasing hydrogen partial pressure. The consumption would indicate the heavy oil component of the solvent was being thermally cracked and then "capped" with hydrogen before the reaction products could recombine to form heavy oils, soluble residuum, or even IOM. A negative yield of distillate or oil means that component of the solvent is being converted to either a lighter or heavier molecular weight material.

Increasing the first stage hydrogen content from 25 to 50 percent caused the conversion to drop from 82 to 77 percent. The gas production remained relatively constant with a small increase in soluble residuum at the expense of the

```
         Conversion
(Wt % MAF Lignite Charged)

                 !                                    GGG       GGG       GGG
              80 +              GGG       GGG       GGG       GGG       GGG       GGG
                 !    GGG       GGG       GGG       GGG       GGG       GGG       GGG
                 !    GGG       GGG       GGG       GGG       GGG       GGG       GGG
                 !    GGG       GGG       GGG       GGG       GGG       GGG       GGG
              60 +    GGG       GGG       GGG       GGG       GGG       GGG       GGG
                 !    GGG       GGG       GGG       GGG       GGG      00000      GGG
                 !    GGG       GGG       GGG      00000     00000     00000      GGG
                 !    GGG      00000     00000     00000     00000     00000      GGG
              40 +    GGG      00000     00000     00000     00000    RRRRRRR     GGG
                 !   00000     00000     00000     00000     00000    RRRRRRR     GGG
                 !   00000     00000     00000    RRRRRRR   RRRRRRR   RRRRRRR     GGG
                 !  RRRRRRR    00000    RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR     GGG
              20 +  RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR
                 !  RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR
                 !  RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR
                 !  RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR   RRRRRRR
               0 +-----------------------------------------------------------------------
                 !                                                               00000
                 !                                                               00000
                 ------------------------------------------------------------------------
Percent Hydrogen     0-0       0-25      0-50      0-75     0-100     0-100    100-100
Gas (Mole %)
1st Stage - 2nd Stage

Run                  N175      N177      N179      N176      N174      N178      N180

G = Gas + Water            R = THF-Soluble Residuum                 0 = Distillate
```

Figure 4. The Effect of Hydrogen Partial Pressure on Conversion

```
        Distillate Yield
   (Wt % MAF Lignite Charged)
                 !
                 !               LLL       LLL
                 !              MMMMM      LLL       LLL
              30 +              MMMMM     MMMMM      LLL
                 !              MMMMM     MMMMM      LLL
                 !    MMMMM     MMMMM     MMMMM     MMMMM
                 !    MMMMM     MMMMM     MMMMM     MMMMM
                 !    MMMMM     MMMMM     MMMMM     MMMMM
              20 +    MMMMM     MMMMM     MMMMM     MMMMM      LLL
                 !    MMMMM     MMMMM     MMMMM     MMMMM      LLL
                 !    MMMMM     MMMMM     MMMMM     MMMMM      LLL
                 !    MMMMM     MMMMM     MMMMM     MMMMM     MMMMM     MMMMM
                 !    MMMMM     MMMMM     MMMMM     MMMMM     MMMMM     MMMMM
              10 +    MMMMM     MMMMM     MMMMM     MMMMM     MMMMM     MMMMM
                 !    MMMMM     MMMMM     MMMMM     MMMMM     MMMMM     MMMMM
                 !    MMMMM     MMMMM     MMMMM     MMMMM     MMMMM     MMMMM      LLL
                 !    MMMMM     MMMMM     MMMMM     MMMMM     MMMMM    HHHHHHH     LLL
                 !    MMMMM     MMMMM     MMMMM     MMMMM    HHHHHHH   HHHHHHH    MMMMM
               0 +----------------------------------------------------------------------
                 !     LLL     HHHHHHH   HHHHHHH   HHHHHHH               LLL     HHHHHHH
                 !   HHHHHHH   HHHHHHH   HHHHHHH   HHHHHHH                       HHHHHHH
                 !   HHHHHHH   HHHHHHH   HHHHHHH   HHHHHHH                       HHHHHHH
                 !   HHHHHHH   HHHHHHH   HHHHHHH   HHHHHHH                       HHHHHHH
             -10 +   HHHHHHH   HHHHHHH   HHHHHHH   HHHHHHH                       HHHHHHH
                 !   HHHHHHH             HHHHHHH   HHHHHHH                       HHHHHHH
                 !   HHHHHHH             HHHHHHH   HHHHHHH                       HHHHHHH
                 !   HHHHHHH             HHHHHHH   HHHHHHH                       HHHHHHH
                 !   HHHHHHH
                 -------------------------------------------------------------------------
Percent Hydrogen       0-0       0-25      0-50      0-75     0-100     0-100    100-100
in Feed Gas
1st Stage - 2nd Stage

Run                    N175      N177      N179      N176      N174      N178      N180

L = Light Oils                   M = Middle Oils                       H = Heavy Oils
```

Figure 5. The Effect of Hydrogen Partial Pressure on Distillate Yield

distillate. Light and middle oil yields were constant but the heavy oil yield dropped from -10 to -15 percent.

Increasing the hydrogen content from 50 to 75 percent further increased the soluble residuum yield while keeping gas and oil yields relatively constant. The distillate showed a higher yield of light oil and a lower yield of middle oil.

Using 100 percent hydrogen in the second stage showed no increase in gas yields but again a large quantity of the distillate was converted into soluble residuum. The distillate yield bar graph, Figure 5, indicated a two and one-half fold decrease in middle oil production, a net loss of light oils, and an increase in heavy oils. This would indicate that with a reduced quantity of carbon monoxide present, the ability of hydrogen to "cap" the intermediate radicals and form light distillates is reduced. These effects, in Run N-178, can not be entirely attributed to hydrogen partial pressure because the first stage temperature was statistically lower than the first stage mean temperature for the other runs.

The control run, N-180, using pure hydrogen in both stages, showed a gas yield of 55 percent with a net consumption of oils, and a small yield of soluble residuum. The distillate bar graph, Figure 5, when compared with that of Run N-178, showed a small gain in light and middle oil and a drop in heavy oil yields from 3.9 percent to -15.2 percent.

With increasing hydrogen partial pressure, Figures 4 and 5 show an increase in soluble residuum and light oil at the expense of the heavy oil. There was also a trend toward lower middle oil production with gas yields remaining fairly constant over the entire range of gas feeds. This would indicate that "capping" material such as activated molecular hydrogen was able to stabilize some of the intermediate reaction products but not entirely prevent condensation of material to form preashaltenes and asphaltenes (soluble residuum). Hydrogen is either added directly as activated molecular hydrogen or added indirectly through the coal liquids. The coal liquids serve as a medium that "accepts" hydrogen and then "donates" the hydrogen to an "active" site. Studies have indicated the hydrogen donating solvent predominates the hydrogen transfer mechanism (23). Studies have also shown hydrogen donating solvents produce increased distillate yields while decreasing soluble residuum and gas yields (24). The use of ammonia as a catalyst has also shown beneficial results (25). Overall conversion is slightly less but the reduced conversion is due to decreased gas yields. Distillate yields were enhanced by the ammonia catalyst which is thought to function similarly to the carbon monoxide molecule by "tying" up the active site of the intermediate reaction products until hydrogenation is favorable. With increasing hydrogen partial pressure, distillate yields would be expected to continually increase at the expense of soluble residuum because of the increased available hydrogen. However, this was not the case as soluble residuum increased with higher hydrogen partial pressures. This would indicate that carbon monoxide is also necessary to the two stage liquefaction process. Previous work has shown carbon monoxide acts as a reductant that prevents condensation or repolymerization reactions from occuring (26). With decreasing carbon monoxide partial pressure, the condensation reactions become more prevalent producing increased quantities of soluble residuum.

The trend for the series of runs indicates that with increasing hydrogen partial pressure, soluble residuum increases while distillate and gas yields decrease. However, Run N-180 with pure hydrogen in both stages, exhibited low soluble residuum yields, a net loss of distillates, and a substantial increase in gas yields. The results from Run N-180 may be attributed to the "thermal shock" of injecting the mixture from the first stage reactor (260°C) into the second stage reactor (440°C). The University of North Dakota Energy Research Center reported that the thermal shock observed in the two stage system decreased soluble residuum and distillate yields and increased gas yields (27).

4.5 CONVERSION VERSUS TIME AND HYDROGEN PARTIAL PRESSURE

Figure 6 shows the overall conversion of MAF coal for the seven experimental runs runs except for Run N-174, pure hydrogen in the second stage, for which data were not available. Runs N-177, N-179, and N-176 which used 25 percent, 50 percent, and 75 percent hydrogen in the second stage, respectively, showed conversions slightly lower than the pure hydrogen run, N-180. Conversion for runs with both carbon monoxide and hydrogen in the second stage feed gas increased at similar rates in the early portion of each run. The maximum conversion for these runs was approached after ten minutes in the second stage reactor. During this time, the high molecular weight substances such as IOM, soluble residuum, and distillate were thermally cracked. After ten minutes, thermal cracking of the high molecular weight reaction products ceased because of the reduced quantity of reaction products or the cracking proceeded at a rate equal to condensation reactions which reformed IOM, soluble residuum, and distillate.

For the pure carbon monoxide and hydrogen runs, the conversion decreased rapidly with the introduction of slurry into the second stage and then slowly increased over the period of the run. With only carbon monoxide present, the intermediate reaction products would probably be stabilized

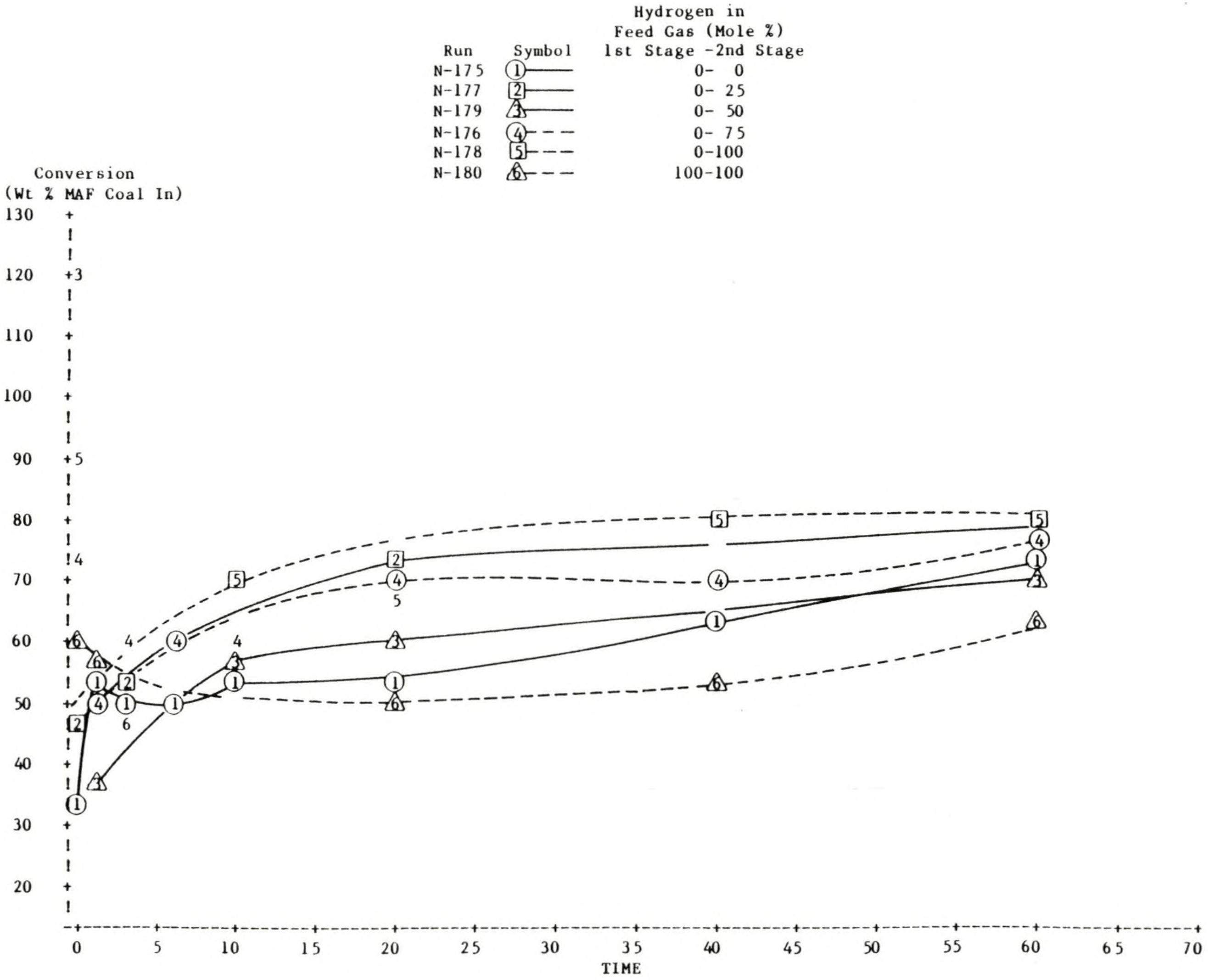


Figure 6. The Effect of Residence Time on Conversion

but available hydrogen supplied by the water gas shift reaction would be insufficient to "cap" a major portion of the active sites. Without hydrogen present the active sites were not "capped" and the reaction products recombined to form soluble residuum. The slow decrease in overall conversion, as seen by Figure 6, for the pure hydrogen run was caused by thermal decomposition of the reaction products. As the products were broken down, hydrogenation did not occur as extensively as in the other runs because of the absence of the stabilizing effect of carbon monoxide. The reaction products recombined to form IOM which would explain the decrease in conversion. Shorter residence times with one stage would alleviate this problem because overall conversion is the highest when the reactants enter the second stage (28).

4.6 WATER GAS SHIFT VERSUS TIME AND HYDROGEN PARTIAL PRESSURE

The water gas shift reaction defined by the following equation; (29)

$$CO + H_2O = H_2 + CO_2 \quad (4)$$

plays an important role in coal liquefaction because it is a relatively inexpensive source of hydrogen. Therefore, a shift to the right of the equation is desirable. An approximation of the hydrogen that is produced by the water gas shift can be calculated by assuming one mole of hydrogen is produced for every mole of carbon dioxide. Carbon dioxide is not lost in the system because it is relatively unreactive. The approximation of hydrogen using this method will be high since some carbon dioxide is given off by the coal during thermal decompostion. Appendix J contains graphs showing the moles of carbon monoxide, water, hydrogen, and carbon dioxide for each run. Table 6 shows the approximate fraction of hydrogen that was produced by the water gas shift reaction.

TABLE 6

The Effect of Hydrogen Partial Pressure on Hydrogen Production

Run #	H_2 in Feed Gas (Moles)	Moles Produced	Available Hydrogen Produced by Shift Reaction (Mole %)
N-175	0.00	8	100
N-177	1.75	1	36
N-179	3.50	3.5	50
N-176	5.25	2	28
N-178	7.00	1.75	20
N-180	Not Available		

Run N-175, pure carbon monoxide, produced the largest quantity of carbon dioxide for the six runs indicating a high water gas shift. Approximately 8 moles of hydrogen were produced during the run. This would be expected because the higher partial pressure of carbon monoxide would drive the reaction to the right. With the introduction of hydrogen, the yields greatly decreased. However, as the hydrogen partial pressure increased and subsequently, the carbon monoxide partial pressure decreased, the amount of carbon dioxide produced did decrease except for run N-179. This decrease would be expected because as the partial pressure of carbon monoxide decreased and the partial pressure of hydrogen increased, the reaction conditions were not favorable. Run N-179, 50 percent hydrogen in the second stage, did not follow the general pattern of the other runs possibly because of the lower first stage temperature. The hydrogen produced during the runs with hydrogen in the feed gas ranged from 36 to 20 mole percent except in Run N-179.

4.7 DISTILLATE YIELD VERSUS TIME AND HYDROGEN PARTIAL PRESSURE

The distillate yield is calculated by adding the yields of the vapor and liquid components of the distillate. The liquid component of the distillate is calculated by comparing a ratio of liquid/ash in to liquid/ash out. Since the mass of the ash in is assumed to be constant except for material losses in the system, a simple ash balance can be done. The vapor component of the distillate is calculated as a difference in the total mass charged to the system and the mass of the liquids and gases (not including the vapor component) at the time of the sample. For example, if the system had a charge of 450 grams and at the time of the sample the mass of the gas and liquid was 350 grams. The mass of vapor component of the distillate would be 100 grams. If the mass of the water was not known, the vapor distillate could not be determined. Figure 7 shows the distillate for the six runs. As can be seen from Figure 7, the distillate yield remained fairly constant for the all runs over the entire run period. Runs N-175, pure carbon monoxide, and N-177, 25 percent hydrogen, are not shown because water yields were not available.

Figures 8 and 9 show the two components, vapor and liquid distillate, respectively. The vapor component of the distillate remained fairly constant with only a slight increase after the introduction of hydrogen. However, the liquid component of the distillate (heavy oil) was broken down for the first ten minutes of the second stage reaction time and then cracking of the liquid component of the distillate leveled off. With increasing hydrogen partial pressure, the vapor component of the distillate did not vary to any great degree but the liquid component of the distillate yield decreased slightly. The total distillate yields for the runs remained fairly constant during the entire second stage run period.

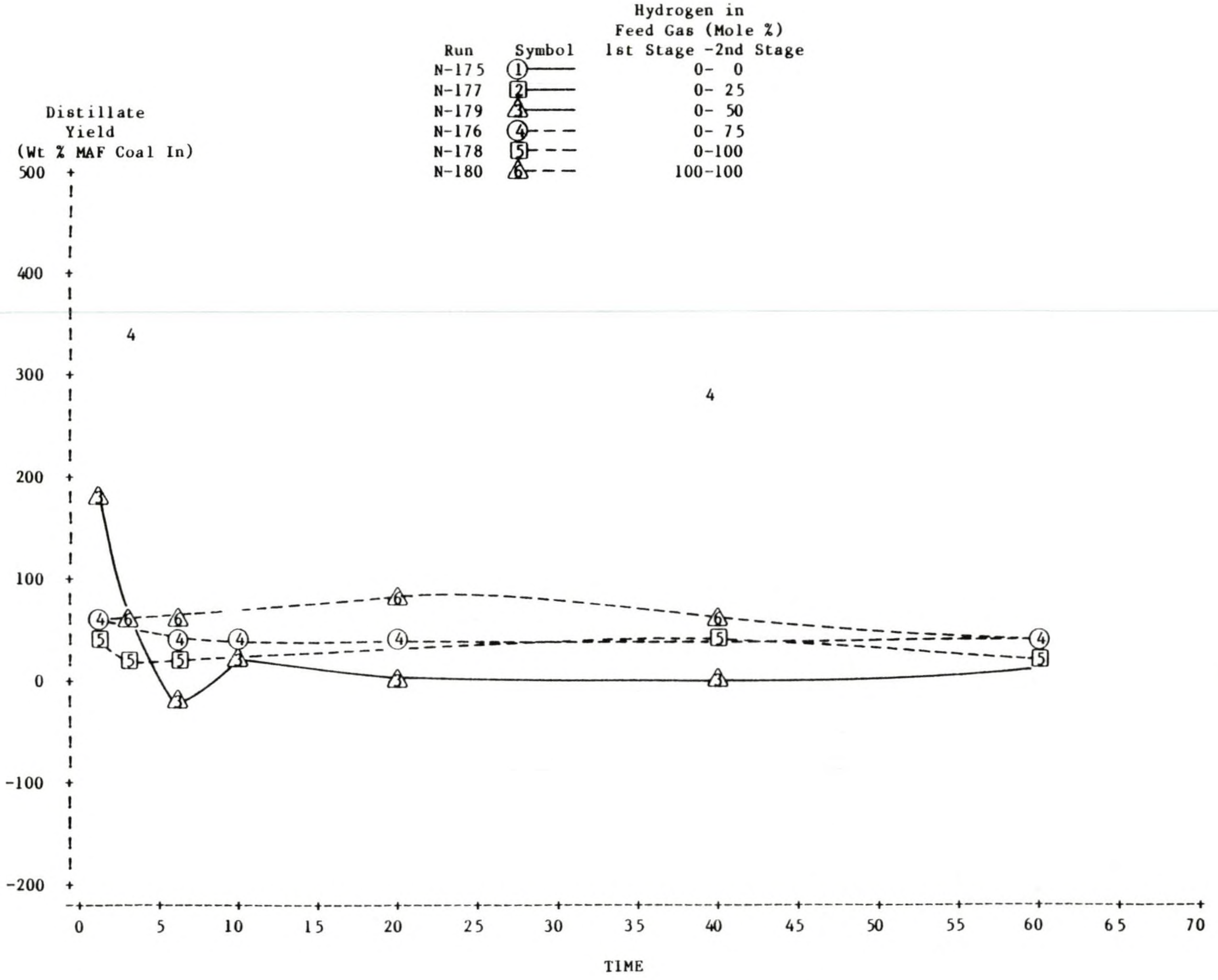


Figure 7. The Effect of Residence Time on Distillate Yield

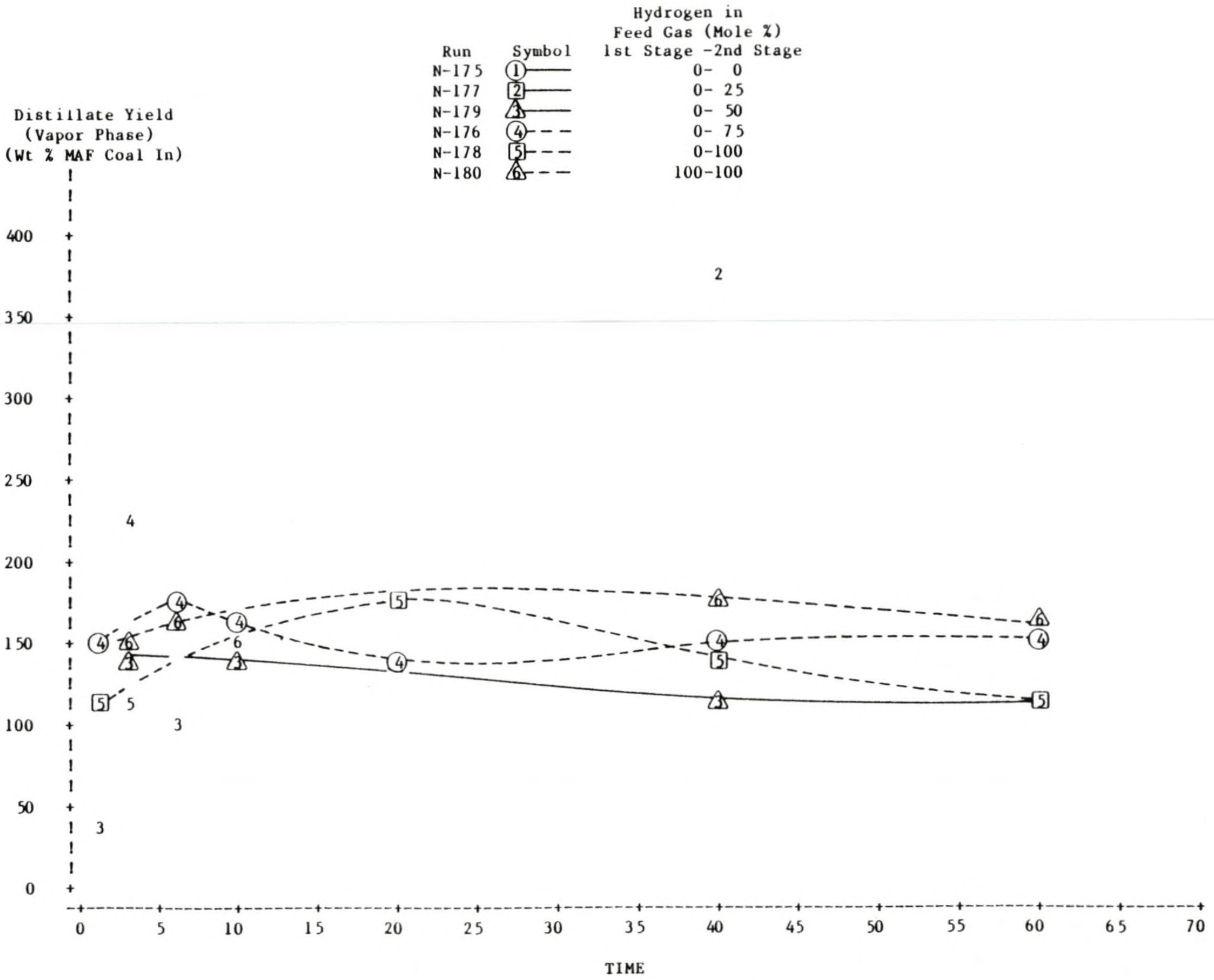


Figure 8. The Effect of Residence Time on Distillate Yield (Vapor Phase)

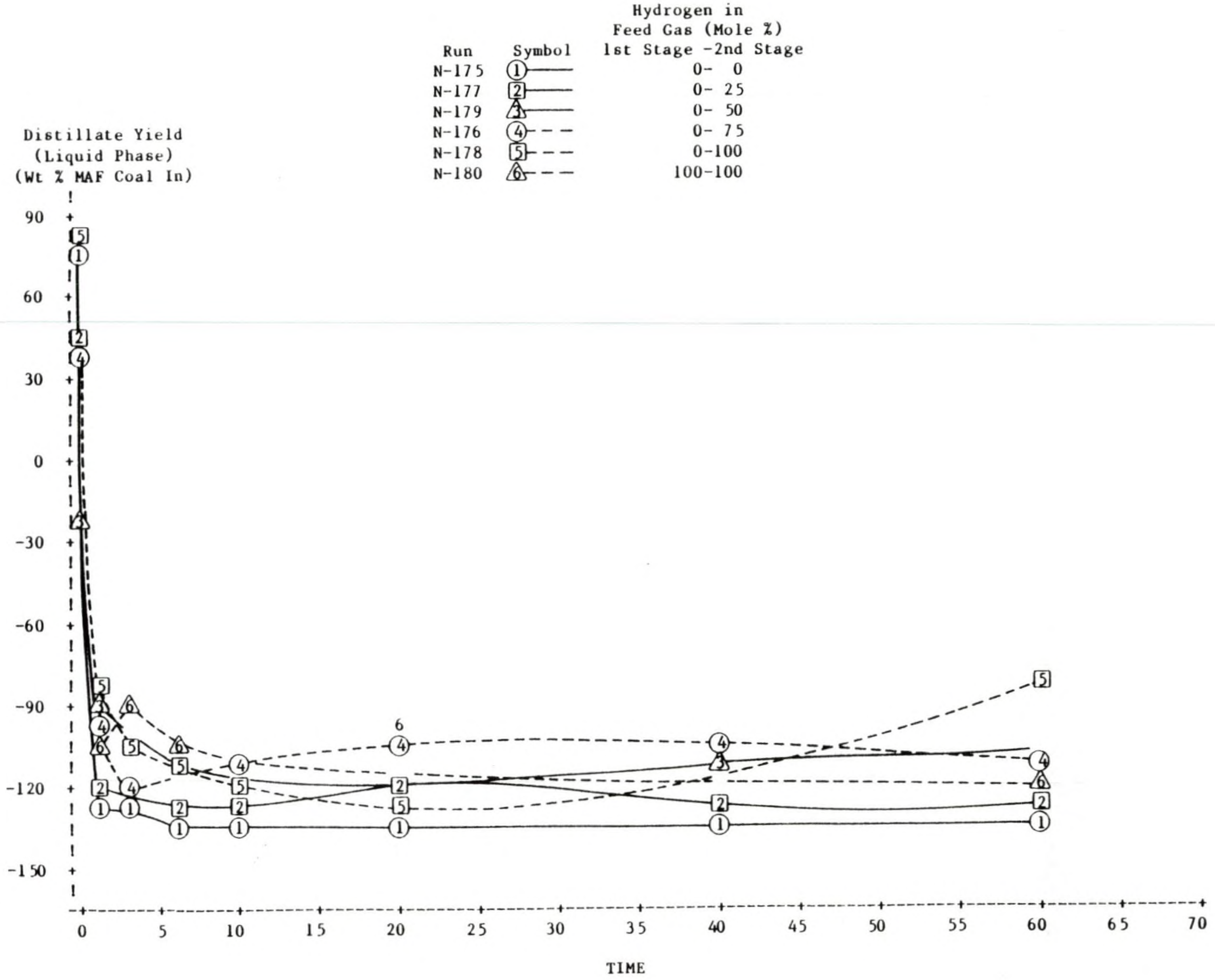


Figure 9. The Effect of Residence Time on Distillate Yield (Liquid Phase)

4.8 SOLUBLE RESIDUUM VERSUS TIME AND HYDROGEN PARTIAL PRESSURE

Soluble residuum yields increased slightly with time for all runs. Figure 10 shows the increase of soluble residuum with reaction time for each of the runs. The increase during the run period was probably due to the declining amount of hydrogen that was present as the run progressed. It also appears that the yield of soluble residuum increased slightly with increasing hydrogen partial pressure but the increase could also have been caused by lower first stage temperatures in Runs N-179 and N-180 and the lower second stage temperature of Run N-178. Previous studies have shown soluble residuum yields are enhanced by reduced temperatures (30). Figure 11 shows the soluble residuum yield for the same hydrogen partial pressure but with increased reaction temperature. Using Figure 11 and referring to Figures 4 and 5, the lower temperature run, N-178 (358°C), has higher soluble residuum yield and a net loss of light oils as compared to run N-174 (370°C). This would indicate that at lower first stage temperatures condensation reactions are not as inhibited by carbon monoxide and the thermal cracking that occurs in the second stage is not sufficient to break down the increased soluble residuum yields.

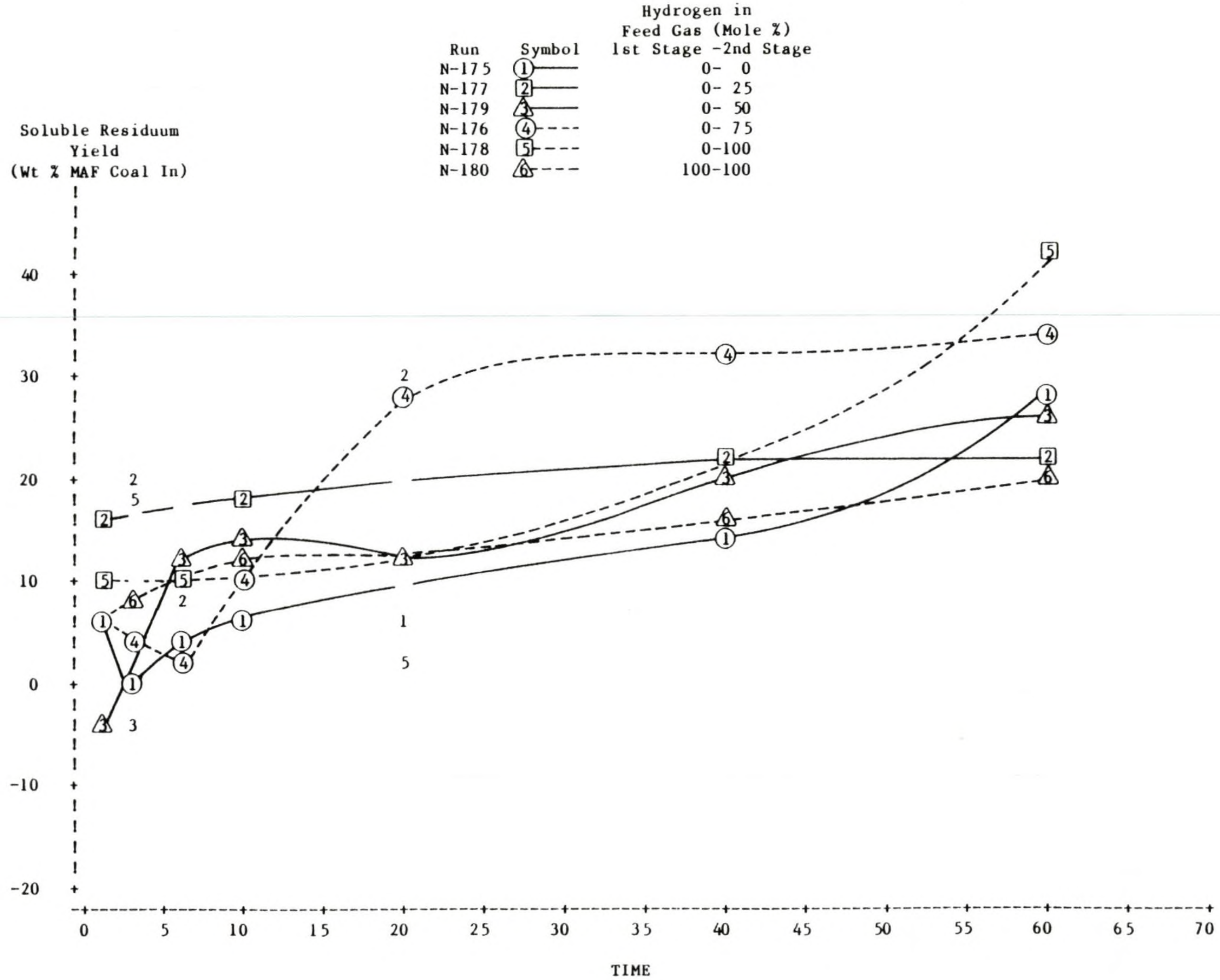


Figure 10. The Effect of Residence Time on Soluble Residuum

```
SOLUBLE RESIDUUM YIELD
 (% MAF COAL CHARGED)
        40 +      RRRRRRR
           !      RRRRRRR
           !      RRRRRRR
           !      RRRRRRR
           !      RRRRRRR
        30 +      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
        20 +      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
        10 +      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
           !      RRRRRRR     RRRRRRR
           -----------------------------
  1st Stage
  Temp. Degree C     358         370

  Run                N178        N174
```

Figure 11. The Effect of Temperature on Soluble Residuum Yield

Chapter V

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

1. A combination of syngas with pure carbon monoxide or pure hydrogen reduced equivalent hydrogen consumption below that used by a pure hydrogen feed in the second stage. A lighter slate of distillates was produced using a feed richer in hydrogen. Equivalent hydrogen consumption was relatively high when using a pure carbon monoxide feed gas. The consumption reached a minimum with a 25 percent hydrogen feed stock and increased slightly with increasing hydrogen partial pressure.
2. Syngas may be an economical feed gas to be used in the second stage. The higher overall conversion in the second stage was due to a higher yield of soluble residuum which might have been caused by a low reaction temperatures. The highest conversion was produced when the hydrogen partial pressure was in the 50 to 100 percent range. Lighter oils were produced with high concentrations of hydrogen but a higher overall conversion was produced in the 50 to 75 percent range.

3. The reaction time for the second stage can be reduced because conversion rose sharply during the first 10 minutes of the second stage residence time and then leveled off to a constant value. Reaction time for the second stage can therefore be reduced from 60 minutes to less than 10 minutes.
4. The water gas shift reaction produced a sufficient quantity of hydrogen to merit further investigation into the role the reaction plays in hyrogenation of coal liquids. There was a decrease in hydrogen produced by the shift reaction with increasing hydrogen partial pressure in the second stage feed gas. With hydrogen present in the feed gas, hydrogen production from the shift reaction ranged from 28 to 36 mole percent of the total hydrogen available for the hydroliqeufaction reactions.
5. A step to upgrade the heavy oils in the distillate may be necessary in the two stage system if a short reaction time (ten minutes) is used in the second stage. The step may be necessary because the light and middle oil production (vapor component of the distillate) continued for the entire 60 minute reaction in the second stage while the thermal cracking of the heavy oils (liquid component of the distillate) decreased or stopped after approximately ten minutes in the second stage. The use of hydrogen do-

nating solvents or catalysts may speed up the light and middle oil production making the upgrading step unecessary.

5.2 RECOMMENDATIONS

The first stage temperature should be approximately 370°C and should not fall below 365°C. This will insure better light and middle oil yields without consuming heavy oil. Soluble residuum yields will also decrease. The run period can be decreased to a maximum of ten minutes for the second stage. Further studies should then be conducted to find the optimum reaction time for the second stage. Studies of conversion of lignite to THF solubles could include using syngas coupled with hydrogen sulfide, iron catalysts, or a hydrogen donating solvent as was suggested by Cassidy et al (31). Using catalysts or hydrogen donating solvent may provide a better mechanism for supplying hydrogen to the intermediate reactants than does gaseous hydrogen. Studies that would help understand the makeup of the reactants or the reacting mixture might include: temperature studies of the reactants in the autoclaves to better understand the liquid and gas makeup of the mixture, radioactive tracers placed in a hydrogen donating solvent or on hydrogen sulfide to determine transfer mechanisms, further examination of the water gas shift, and studies on equivalent hydrogen consumption using varying gas compositions. Further studies on the water gas shift reaction could include using a deuterium tracer placed in the water to determine if the hydrogen produced by the shift reaction is readily transfered to the coal liquids or is used to produce hydrogen gas. These studies may lead to a better understanding of the hydrogen transfer system.

Appendices

Appendix A

DETERMINATION OF LIGNITE MOISTURE CONTENT

Procedure

1. Preheat oven to 110 ± 2^{o}C
2. Dry crucibles in oven for 15 minutes
3. Cool crucibles in desiccator for 10 minutes
4. Weigh empty crucibles and then add two grams of lignite
5. Dry in oven for 60 minutes
6. Cool in desiccator for 10 minutes and weigh

Sample Calculation:

	Sample	
	A	B
Weight of empty crucible:	13.1159	13.0960
Weight of crucible + coal:	15.1157	14.5625
Weight of Coal:	1.9998	1.9998
Weight of crucible + coal:	15.1157	14.5625
Weight of crucible + coal after drying:	14.5827	14.5625
Weight of Moisture Lost:	0.5330	0.5333
Weight of Moisture Lost:	0.5330	0.5333
Weight of Coal:	1.9998	1.9998
Percent Moisture in Coal:	26.65%	26.67%
Average Percent Moisture in Coal:	26.66%	

Appendix B

DETERMINATION OF LIGNITE ASH CONTENT

General Calculation:

$$\frac{(100 - \text{Moisture Content})*(\text{Experimentally found Ash Content})}{(100 - \text{Moisture Content At Time of Ashing})}$$

Sample Calculation:

$$\frac{(100 - 26.66) * (7.52)}{(100 - 31.76)}$$

= 8.08 % Ash in Sample

Appendix C

COMPUTER PROGRAMS AND PROGRAM VARIABLES USED FOR MASS BALANCE AND OTHER CALCULATIONS

Computer Program Variables

Character Variables:

A$ - Low number of runs to be printed

B$ - High number of runs to be printed

D$ - Highest run number to be found in a particular search file

E$ - Search file name

F$ - Run number

G$ - Coal

H$ - Type of solvent

I$ - Type of additive

J$ - Date

K$ - Run time, minutes

L$ - Time sample, yes or no

M$ - Average temperature, ^{O}C

N$ - Search variable choice, 1-10

O$ - Maximum temperature of run, ^{O}C

P$ - Maximum pressure of run, psig

Q$ - Internal program variable used for subject search in files

R$(1-14) - Title variables for table printout

Y$ - H_2S added in autoclave, yes or no

Z$ - Search variable in coal data

Number Variables:

A1 - Average temperature of run, $^{\circ}$C

A2 - Endpoint for balance code

A3 - Endpoint for balance code

B1 - Mole % H_2 in feed gas

B2 - Mole % CO in feed gas

B3 - Mole % H_2S in feed gas

B4 - Number of gram moles in feed gas

C1 - Cubic ft of product gas

C2 - Barometric pressure, mmHg

C3 - Temperature of gas (60°F)

D(1) - Mole % CO product gas

D(2) - Mole % C_2H_6 product gas

D(3) - Mole % C_3H_8 product gas

D(4) - Mole % H_2 product gas

D(5) - Mole % CH_4 product gas

D(6) - Mole % CO product gas

D(7) - Mole % H_2S product gas

D(8) - Mole % NH_3 product gas

E1 - Flag used in balance code

E2 - Location variable for print format statement

E3 - Location variable for print format statement

F1 - Grams of light oil from microdistillation

F2 - Grams of middle oil from microdistillation

F3 - Grams of heavy oil from microdistillation

F4 - Wt % water in end product

F5 - Grams of sample used in microdistillation (alliquot)

F6 - Grams of nondistillables in microdistillation

F7 - Grams of condensed liquids from product gas stream

F8 - Grams of light oil in the quench cold traps

F9 - Grams of water in the quench cold traps

G1 - Wt % ash in end product

G2 - Wt % THF solubles in end product

H1 - Total wt end product, gms

H2 - Wt of product left in system, gms (THF flush)

H3 - Wt of slurry in, gms (charge)

I1 - Wt of coal in charge, gms

I2 - Wt of water in charge, gms

I3 - Wt of addition in charge, gms

I4 - Wt of solvent in charge, gms

I5 - Wt % ash in charge

I6 - Wt % water in charge

I7 - Wt % of L.O. in charge, gms

I8 - Wt % of H.O. in charge, gms

I9 - Wt % of solid Residue in charge, gms

J1 - Wt loss in process for 100% mat. bal. closure

K(1) - Grams CO in

K(2) - Grams H_2 in

K(5) - Grams H_2S in

K(7) - Grams H_2O in

K(8) - Grams additive in

K(9) - Grams ash in

K(10) - Grams MAF coal in

K(11) - Grams solid residue in

K(12) - Grams H.O. in

K(13) - Grams M.O. in

K(14) - Grams L.O. in

K(15) - Grams solvent in

L1 - Total grams feed gas

L2 - Moles product gas

L3 - Correction factor for void volume of reactors

L4 - Correction factor for void volume of reactors

L5 - Total moles after correction factor

M(1) - CO_2 molecular wt

M(2) - C_2H_6 molecular wt

M(3) - C_3H_8 molecular wt

M(4) - H_2 molecular wt

M(5) - CH_4 molecualr wt

M(6) - CO molecular wt

M(7) - H_2S molecular wt

M(8) - NH_3 molecular wt

N(1-8) - Grams out of gas, (individual)

N9 - Total weight gas out

O1 - Grams water in alliquot

O2 - Grams light oil in alliquot and oil traps

O3 - Total gms oil (water, soluble residuum free) in alliquot

O4 - Total gms oil in alliquot

O5 - Ratio of oil in end product to oil in alliquot

O6 - Total gms of oil in end product

O7 - Grams water and oil in end product

O8 - Grams light oil in alliquot and cold traps

O9 - Total gms of nondistillables

P(1) - Grams CO out

P(2) - Grams H_2 out

P(3) - Grams CO_2 out

P(4) - Grams C_1-C_3 out

P(5) - Grams H_2S out

P(6) - Grams NH_3 out

P(7) - Grams H_2O out

P(8) - Grams additive out

P(9) - Grams ash out

P(10) - Grams IOM out

P(11) - Grams solid residue out

P(12) - Grams H.O. out

P(13) - Grams M.O. out

P(14) - Grams L.O. out

Q1 - Total gms liquid product out

R1 - Total wt slurry, gms

R2 - Grams coal in

R3 - Total wt gas, normalized based on MAF coal in, gms

R4 - Total wt liquid, normalized based on MAF coal charged, gms

R5 - Total wt MAF coal in, gms

R6 - Wt of gas in process unaccounted for, gms

R7 - Grams gas + liquid out

R8 - Wt of liquid in process unaccounted for, gms

R9 - Total normalized coal in, gms

S(1-14) - Grams out with loss factor added

T(1-10) - Grams out/MAF coal in

U(1-14) - Normalized grams out/MAF coal in

(V's are based on 100g MAF coal charged)

V1 - Total grams in

V2 - Material balance closure

V3 - Grams distillate oils in

V4 - Grams total THF soluble and soluble residuum

V5 - Grams of gas

V6 - Wt % MAF coal converted to THF solubles, (Conversion)

V7 - Total oils out / total oils in, (Solvent Recovery)

W1 - Counter flag

Z1 - Weight loss assigned to liquid

Name: COMMAND1
Language: Basic
Description: Constructs programs for various data processing

```
 10 LIN 130
 20 MESSAGE BLOCK
 30 TRANS OP
 40 &TYPE WELCOME TO THE UNDERC HOT CHARGE DATA PROCESSNG
    LIBRARIES
 50 &TYPE
 60 &TYPE
 70 &TYPE         LIBRARY OPTIONS
 80 &TYPE
 90 &TYPE         1---MATERIAL BALANCE PROGRAMS
100 &TYPE         2---TIME SAMPLING PRORAMS
110 &TYPE         3---TUBING BOMB PROGRAMS
120 &TYPE
130 &TYPE  ENTER OPTION
140 &READ 1
150 &IF &1 EQ 1 &GOTO MATBAL
160 &IF &1 EQ 2 &GOTO TIMSAM
170 &IF &1 EQ 3 &GOTO TUBEBAL
180 MATBAL:&TYPE               MATERIAL BALANCE OPTIONS
190 &TYPE             1---INPUT MATERIAL BALANCE DATA
200 &TYPE             2---MATERIAL BALANCE CALCULATIONS AND
                          RESULTS
210 &TYPE             3---MASTER LIST OF RUNS
220 &TYPE             4---COMPARE SPECIFIC PARAMETERS
230 &TYPE             5---COMPARE COAL ANALYSIS
231 &TYPE             6---FEED GAS CALCULATION
232 &TYPE             7---PRODUCT GAS CALCULATIONS
240 &TYPE
250 &TYPE     INPUT OPTION--ENTER 6 FOR INSTRUCTIONS ELSE 0
    (EX. 2,6)
260 &READ 2,3
270 &IF &2 EQ 1 &GOTO MDATA
280 &IF &2 EQ 2 &GOTO MBCALC
290 &IF &2 EQ 3 &GOTO MASTER
300 &IF &2 EQ 4 &GOTO SPECPAR
310 &IF &2 EQ 5 &GOTO COAL
311 &IF &2 EQ 6 &GOTO GASCALC
312 &IF &2 EQ 7 &GOTO PRODGAS
320 MDATA: FILE TEMPA
330 RUN MDATA
340 LOAD RUNA
350 LIST
360 &TYPE INPUT HIGHEST SEARCH FILE NAME
370 &READ 8
380 PROTECT &8 WRITE
390 LOAD &8
400 LIST 1
410 &TYPE ENTER NUMBER OF RUNS IN FILE
420 &READ 9
430 &IF &9 EQ 12 450 &GOTO NEWFLE
```

```
440 &TYPE ENTER THE NEW NUMBER OF FILES
450 &READ 10
460 CH 1 '&9' '&10' T
470 MERGE TEMPA
480 RENUM 1 1 1
490 SAVE &8
500 PURGE TEMPA
510 PROTECT &8 NOWRITE
520 LOAD RUNA
530 LIST
540 PROTECT RUNA WRITE
550 &TYPE
560 &TYPE INPUT LINE WITH HIGHEST RUN#,THE RUN#, AND THE NEW
    HIGH RUN NUMBER
570 &READ 5,6,7
580 CH &5 '&6' '&7'T
590 SAVE RUNA
600 PROTECT RUNA NOWRITE
610 &EXIT
620 NEWFLE: PROTECT &8 NOWRITE
630 &TYPE INPUT NEW SEARCH FILE NAME
640 &READ 12
650 FILE &12
660 RUN NEW
670 PROTECT &12 NOWRITE
680 PURGE TEMPA
690 PROTECT RUNA WRITE
700 LOAD RUNA
710 LIST
720 &TYPE
730 &TYPE INPUT LAST LINE NUMBER AND RUN#
740 &READ 13,14
750 CH &13 '' '''&14              '' ''&12             '''T
760 SAVE RUNA
770 PROTECT RUNA NOWRITE
780 &EXIT
790 MBCALC: LOAD DIM
800 &IF  EQ 6 MERGE MINSTRUC OVERLAY
810 MERGE BCIN OVERLAY
820 MERGE GLWGT OVERLAY
830 MERGE MBPRINT OVERLAY
840 RUN
850 &EXIT
860 MASTER: LOAD MASTERL
880 MERGE BCIN OVERLAY
880 DELETE 10:170
890 RUN
891 &EXIT
900 SPECPAR: LOAD DIM
910 MERGE BCIN OVERLAY
930 DELETE 10:190
940 DELETE 290:390
950 MERGE GLWGT OVERLAY
960 MERGE SPECPAR OVERLAY
```

```
970 RUN
980 &EXIT
990 COAL: LOAD DIM
1000 MERGE BCIN OVERLAY
1010 DELETE 10:190
1020 DELETE 290:390
1030 MERGE SPECPAR OVERLAY
1040 MERGE GLWGT OVERLAY
1050 MERGE COAL OVERLAY
1060 RUN
1070 &EXIT
1180 GASCALC: LOAD GASCALC
1190 RUN
1200 &EXIT
1210 PRODGAS: LOAD PRODGAS
1220 RUN
1230 &EXIT
```

Name: DIM
Language: Basic
Description: Dimensions Variables

```
1 DIM D(8)
2 DIM K(15)
3 DIM M(8)
4 DIM N(8)
5 DIM P(14)
6 DIM S(15)
7 DIM T(14)
8 DIM U(14)
9 DIM R$(14)
```

Name: BCIN
Language: Basic
Description: Allows balance code choices and loads data from files

```
10 PRINT'DO YOU WISH TO CHOOSE BALANCE CODE?'
15 INPUT N$
20 IF N$='N' THEN GOTO 180
25 PRINT '                SELECT BALANCE CODE'
30 PRINT '                       1---0/100  LIQUID/GAS'
35 PRINT '                       2---50/50  LIQUID/GAS'
40 PRINT '                       3---100/0  LIQUID/GAS'
45 PRINT '                       4---ALL OF THE ABOVE'
50 PRINT '                       5---CHOOSE YOUR OWN RATIO'
55 PRINT '                       6---LIQ/(LIQ+GAS)'
60 INPUT N1
65 IF N1=1 THEN LET Z1=0
70 IF N1=2 THEN LET Z1=50
75 IF N1=3 THEN LET Z1=100
80 IF N1=4 THEN LET E1=1
85 IF N1=5 THEN PRINT'          INPUT % IN LIQUID ONLY'
90 IF N1  5 THEN GOTO 180
95 INPUT Z1
100 PRINT '      INPUT SEARCH ID#,LOW VALUE,HIGH VALUE
    (N###)'
105 INPUT N3,A$,B$
110 GET '4009 RUNA',D$,E$
115 IF D$ A$ THEN GOTO 110
120 GET E$ ,N4
125 FOR A=1 TO N4
130 GET E$, F$,G$,H$,I$,J$,K$,L$,M$,O$,P$
135 IF F$ B$ THEN STOP
140 GET E$ , A1,B1,B2,B3,B4,C1,C2,C3,D(1),D(2)
145 GET E$ , D(3),D(4),D(5),D(6),D(7),D(8),F1,F2,F3,F4
150 GET E$ , F5,F6,F7,F8,F9,G1,G2,H1,H2,H3
155 GET E$ , I1,I2,I3,I4,I5,I6,I7,I8,I9,J1
160 IF F$ A$ THEN GOTO 2675
165 IF N$='N' THEN LET Z1=J1
170 IF N3=1 THEN LET Q$=F$
175 IF N3=2 THEN LET Q$=G$
180 IF N3=3 THEN LET Q$=H$
185 IF N3=4 THEN LET Q$=I$
190 IF N3=5 THEN LET Q$=J$
195 IF N3=6 THEN LET Q$=K$
200 IF N3=7 THEN LET Q$=L$
210 IF N3=8 THEN LET Q$=M$
215 IF N3=9 THEN LET Q$=O$
220 IF N3=10 THEN LET Q$=P$
```

Name: GLWGT
Language: Basic
Description: Material balance calculations and normalization to 100% MAF coal

```
499 REM THIS ROUTINE IS FOR CALCUALTION OF PRODUCT AND
    FEED GAS WEIGHTS
500 K(1)=B4*B2/100*28.011
510 K(2)=B4*B1/100*2.016
520 K(5)=B4*B3/100*34.032
530 L1=K(1)+K(2)+K(5)
540 L2=C2*C1*453.6/(21.85*(C3+460))
545 IF F$ 'N001' .OR. F$ 'N160' THEN GOTO 570
550 L3=(2*3.7853)/(301*.082057)
560 L4=(3.7853)/((A1+273)*.082057)
570 L5=L2+L3+L4
580 M(1)=44.011
590 M(2)=30.07
600 M(3)=44.097
610 M(4)=2.016
620 M(5)=16.04
630 M(6)=28.011
640 M(7)=34.032
650 M(8)=17.024
660 FOR B=1 TO 8
670 N(B)=D(B)/100*L5*M(B)
680 NEXT B
690 N9=N(1)+N(2)+N(3)+N(4)+N(5)+N(6)+N(7)+N(8)
700 REM THIS ROUTINE IS FOR LIQUID PRODUCT DISTRIBUTION
    CALCULATIONS
710 O1=F5*F4/100
720 O2=F1+F7-O1
730 O3=O2+F2+F3
740 O4=F5-F6-O1
750 O5=O4*H1/(O3*F5)
760 P(14)=O2*O5+F8
770 P(13)=F2*O5
780 P(12)=F3*O5
790 O6=P(12)+P(13)+P(14)
800 P(7)=H1*F4/100+F9
810 O7=P(7)+O6
820 P(9)=G1*H1/100
830 P(11)=(H1*G2/100)-(O7-F8-F9)
840 P(10)=((H1*(100-G2))/100)-370 P(9)
850 O8=H2*100/(100-G2)*F6/F5
860 O9=P(9)+P(10)+P(11)
870 P(9)=P(9)+O8*P(9)/O9
880 P(10)=P(10)+O8*P(10)/O9
890 P(11)=P(11)+O8*P(11)/O9
900 Q1=O7+P(9)+P(10)+P(11)
901 Z2=Q1/(Q1+N9)*100
902 IF N1=6 THEN LET Z1=Z2
909 REM THIS ROUTINE CALCULATES LIQUID CHARGE DISTRIBUTION
```

```
910 R1=I1+I2+I3+I4
920 R2=I1*H3/R1
930 K(9)=R2*I5/100
940 K(7)=R2*I6/100+I2*H3/R1
950 K(8)=I3*H3/R1
960 K(10)=R2*(100-I5-I6)/100
965 K(15)=I4*H3/R1
970 K(11)=K(15)*I9/100
980 F(12)=K(15)*I8/100
990 K(14)=K(15)*I7/100
1000 K(13)=K(15)-(K(11)+K(12)+K(14))
1030 REM THIS ROUTINE CALCULATES NORMALIZATION FOR MATERIAL
     BALANCE
1040 R3=0
1050 R4=0
1060 R5=0
1070 R9=0
1090 P(1)=N(6)
1100 P(2)=N(4)
1110 P(3)=N(1)
1120 P(4)=N(2)+N(3)+N(5)
1130 P(5)=N(7)
1140 P(6)=N(8)
1150 R7=P(1)+P(2)+P(3)+P(4)+P(5)+P(6)+P(7)+P(9)+P(10)+P(11)
     P(12)+P(13)+P(14)
1160 R6=(1-Z1/100)*((H3+L1)-(Q1+N9))
1170 R8=Z1/100*((H3+L1)-(Q1+N9))
1180 FOR B=1 TO 6
1190 S(B)=P(B)+R6*P(B)/N9
1200 R3=R3+S(B)
1210 NEXT B
1220 FOR B=7 TO 14
1230 S(B)=P(B)+R8*P(B)/Q1
1240 R4=R4+S(B)
1250 NEXT B
1260 FO R B=1 TO 14
1270 IF B=8 THEN GOTO 1330
1280 IF B  9 THEN GOTO 1310
1290 T(9)=S(9)-K(9)-K(8)
1300 GOTO 1320
1310 T(B)=S(B)-K(B)
1320 R5=R5+T(B)
1330 NEXT B
1340 S(15)=S(13)+S(12)
1350 IF T(14) =0 THEN GOTO 1380
1360 S(15)=S(15)+K(14)
1370 GOTO 1390
1380 S(15)=S(15)+S(14)
1390 R5=R5-T(9)-T(10)
1400 T(9)=0
1410 T(8)=0
1420 T(10)=S(10)+(S(9)-K(9))
1430 R5=R5+T(10)-S(8)
1440 FOR B=1 TO 14
```

```
1450 U(B)=T(B)/R5*100
1460 R9=R9+U(B)
1470 NEXT B
1480 U1=K(1)+K(2)+K(5)+K(6)+K(7)+K(8)+K(9)+K(10)+K(11)
     K(12)+K(13)+K(14)
1490 V2=R7/U1*100
1500 V3=U(12)+U(13)+U(14)
1510 V4=U(11)+V3
1520 V5=U(1)+U(2)+U(3)+U(4)+U(5)+U(6)
1525 V6=100-U(10)
1530 IF K(15)=0 THEN GOTO 1550
1540 V7=S(15)/K(15)*100
```

Name: MBPRINT
Language: basic
Description: Contains formats and instructions for the print out of data

```
231 IF N1=4 THEN LET Z1=0
2000 REM THIS SUBROUTINE PRINTS RESULT. S OF THE MATERIAL
     BALANCE CALCULATIONS
2010 PRINT TAB(1);' ';TAB(2);' ';TAB(3);' '
2020 PRINT '....................................
     ..................'
2030 PRINT ' '
2040 PRINT USING 2041, 'RUN #',F$,'(',Z1,'% LOSS ASSIGNED TO
     LIQUID)'
2041 FORM C,POS30,C,POS45,C,PIC(ZZZ.#),C,SKIP
2050 PRINT 'COAL';TAB(30);G$
2060 PRINT 'SOLVENT';TAB(30);H$
2070 pRINT 'ADDITIVE';TAB(30);I$
2080 PRINT 'DATE';TAB(30);J$
2090 PRINT 'CONDITIONS'
2100 PRINT 'RES TIME (MIN)';TAB(30);K$
2110 PRINT 'TIME SAMPLE ?';TAB(30);L$
2120 PRINT 'AVG TEMP (C)';TAB(30);M$
2130 PRINT 'MAX TEMP (C)';TAB(30);O$
2140 PRINT 'MAX PRESS, PSIG';TAB(30);P$
2150 PRINT USING 2160,'% H2 IN FEED GAS',B1
2160 FORM C,POS30,PIC(ZZZ.##),SKIP
2170 PRINT USING 2180,'NET YIELDS  NET YIELDS'
2180 FORM POS48,C,SKIP
2190 PRINT TAB(50);'GMS OF     WT % MAF'
2200 PRINT 'COMPONENT';TAB(24);'GRAMS IN';TAB(36);'GRAMS OUT
     MAF COAL    '
2230 I8=I8+1
2240 R$(1)= 'CO'
2250 R$(2)= 'H2'
2260 R$(3)= 'CO2'
2270 R$(4)= 'C1-C3'
2280 R$(5)= 'H2S'
2290 R$(6)= 'NH3'
2300 R$(7)= 'H20'
2310 R$(8)= 'ADDITIVE'
2320 R$(9)= 'ASH'
2330 R$(10)= 'MAF IN-IOM OUT'
2340 R$(11)= 'SOLUBLE RESID'
2350 R$(12)= 'HO'
2360 R$(13)= 'MO'
2370 R$(14)= 'LO'
2375 FOR B=1 TO 14
2380 PRINT USING 2390,R$(B),K(B),P(B),T(B),U(B)
2390 FORM C,POS23,PIC(-ZZZZ.##),POS36,PIC(-ZZZ.##),
     POS48,PIC(-ZZZ.##),
2391 CONTINUE POS60,PIC(-ZZZ.##),SKIP
2400 IF B=6 THEN PRINT '  '
```

```
2430 NEXT B
2440 PRINT ' '
2450 PRINT USING 2390, 'TOTAL',U1,R7,R5,R9
2460 PRINT ' '
2470 PRINT 'MISC. NET YIELDS, WT % MAF COAL IN'
2480 PRINT ' '
2490 PRINT USING 2660 ,'DISTILLABLE OILS',V3
2500 PRINT USING 2660 ,'SOLUBLE RESIDUUM',U(11)
2510 PRINT USING 2660 ,'TOTAL THF SOLUBLES',V4
2520 PRINT USING 2660 ,'UNCONVERTED COAL',U(10)
2530 PRINT USING 2660 ,'WATER',U(7)
2540 PRINT USING 2660 ,'GAS',V5
2550 PRINT USING 2660 ,'% CONVERSION',V6
2560 IF K(15)=0 THEN GOTO 2580
2570 GOTO 2600
2580 PRINT USING 2669, 'SOLVENT RECOVERY,WT %','....'
2590 GOTO 2610
2600 PRINT USING 2660, 'SOLVENT RECOVERY, WT %',V7
2610 PRINT USING 2660, 'MAT BAL CLOSURE, WT%',V2
2620 PRINT '.........................................
     ..................'
2630 PRINT ' '
2640 PRINT ' '
2650 PRINT ' '
2660 FORM POS5,C,POS60,PIC(-ZZZ.##),SKIP
2669 FORM POS5,C,POS63,C,SKIP
2670 IF E1=1 .AND. Z1=100 THEN GOTO 2675
2671 IF E1=1 .AND. Z1=Z2 THEN LET Z1=100
2672 IF E1=1 .AND. Z1=50 THEN LET Z1=Z2
2673 IF E1=1 .AND. Z1=0 THEN LET Z1=50
2674 IF E1=1 THEN GOTO 500
2675 NEXT A
2677 IF F$=B$ THEN STOP
2680 GOTO 200
2690 END
```

Name: SPECPAR
Language: Basic
Description: Allows specific parameters to be compared from run to run

```
10 DIM F$(7,10)
11 DIM L(6)
20 DIM I(28,10)
30 DIM A$(10)
40 DIM Z$(10)
100 PRINT '# OF RUNS IN TABLE'
110 INPUT C
111 IF C  1 THEN GOTO 121
112 PRINT 'DO YOU WISH TO CHOOSE BALCODE'
113 INPUT Y$
114 IF Y$='N' THEN GOTO 121
115 PRINT 'INPUT RANGE OF BALCODES (0-50)'
116 INPUT A2,A3
117 L(1)=A2
118 FOR B=2TO6
119 L(B)=L(B-1)+(A3-A2)/5
120 NEXT B
121 FOR D=1 TO C
140 PRINT USING 150,'INPUT RUN #',D
150 FORM C,PIC(ZZZ),SKIP
160 INPUT Z$(D)
161 A$(D)=Z$(D)
162 NEXT D
163 FOR F=1 TO C
164 FOR D=2 TO C
165 E=D-1
166 IF A$(E) A$(D) THEN GOTO 170
167 W$=A$(E)
168 A$(E)=A$(D)
169 A$(D)=W$
170 NEXT D
171 NEXT F
180 B$='N999'
181 IF Y$ = 'Y' THEN LET C=6
190 D=1
210 IF D$ A$(D) THEN GOTO 200
290 IF F$  A$(D) THEN GOTO 2675
300 Z1=J1
301 IF Y$ .NE. 'Y' THEN GOTO 500
301 FOR G=1 TO 6
303 Z1=L(G)
2430 F$(1,D)=F$
2440 F$(2,D)=G$
2450 F$(3,D)=H$
2460 F$(4,D)=I$
2470 F$(5,D)=K$
2480 F$(6,D)=M$
2490 F$(7,D)=P$
```

```
2500 I(8,D)=B1
2510 I(9,D)=K(7)
2515 I(10,D)=U(1)
2520 I(11,D)=U(2)
2530 I(12,D)=U(3)
2540 I(13,D)=U(4)
2550 I(14,D)=U(5)+U(6)
2560 I(15,D)=U(7)
2570 I(16,D)=U(1)+U(2)+U(3)+U(4)+U(5)+U(6)+U(7)
2580 I(17,D)=U(14)
2590 I(18,D)=U(13)
2600 I(19,D)=U(12)
2610 I(20,D)=V3
2620 I(21,D)=U(11)
2625 I(22,D)=V3+U(11)
2630 I(23,D)=U(10)
2640 I(24,D)=V7
2650 I(25,D)=(-T(1)/28.011)-(T(2)/2.016)
2660 I(26,D)=K(10)/K(9)
2661 I(27,D)=V6
2662 I(28,D)=V2
2663 IF Y$ .NE. 'Y' THEN GOTO 2670
2664 D=D+1
2665 NEXT G
2666 GOTO 2929
2670 IF D=C THEN GOTO 2929
2672 IF A$(D)=F$ THEN LET D=D+1
2700 GOTO 200
2929 PRINT USING 3403
2930 PRINT USING 3380,'.............................'
2931 FOR D=1 TO C
2932 E3=D*13+18
2933 PRINT USING 3402,'............'
2934 NEXT D
2940 PRINT '  '
2941 PRINT '  '
2950 FOR B=1 TO 28
2960 IF B=1 THEN PRINT USING 3380,'RUN #'
2970 IF B=2 THEN PRINT USING 3380,'COAL'
2980 IF B=3 THEN PRINT USING 3380,'SOLVENT'
2990 IF B=4 THEN PRINT USING 3380,'ADDITIVE'
3000 IF B=5 THEN PRINT USING 3380,'RUN TIME (MIN)'
3010 IF B=6 THEN PRINT USING 3380,'AVG TEMP'
3020 IF B=7 THEN PRINT USING 3380,'MAX PRESS (PSIG)'
3030 IF B=8 THEN PRINT USING 3380,'% H2 FEED GAS'
3040 IF B=9 THEN PRINT USING 3380,'MOISTURE (GMS)'
3050 IF B=10 THEN PRINT '  '
3060 IF B=10 THEN PRINT 'NET YIELDS (WT% MAF COAL CHARGED)'
3070 IF B=10 THEN PRINT '  '
3080 IF B=10 THEN PRINT USING 3380,'CO'
3090 IF B=11 THEN PRINT USING 3380,'H2'
3100 IF B=12 THEN PRINT USING 3380,'CO2'
3110 IF B=13 THEN PRINT USING 3380,'C1-C3'
3120 IF B=14 THEN PRINT USING 3380,'H2S, NH3'
```

```
3130 IF B=15 THEN PRINT USING 3380,'H20'
3140 IF B=16 THEN PRINT USING 3380,'GAS + H20'
3150 IF B=17 THEN PRINT USING 3380,'LT.  OIL'
3160 IF B=18 THEN PRINT USING 3380,'MID OIL'
3170 IF B=19 THEN PRINT USING 3380,'HVY OIL'
3180 IF B=20 THEN PRINT USING 3380,'C4+ DIST'
3190 IF B=21 THEN PRINT USING 3380,'SOL RESIDUUM'
3195 IF B=22 THEN PRINT USING 3380,'TOTAL LIQUID'
3200 IF B=23 THEN PRINT USING 3380,'IOM'
3210 IF B=24 THEN PRINT '  '
3220 IF B=24 THEN PRINT USING 3380,'SOLVENT REC'
3230 IF B=25 THEN PRINT USING 3380,'H2 EQUIV CONSUMP'
3240 IF B=26 THEN PRINT USING 3380,'IOM/GRAM ASH'
3250 IF B=27 THEN PRINT USING 3380,'CONVERSION'
3260 IF B=28 THEN PRINT USING 3380,'MAT BAL CLOSURE'
3270 FOR E=1 TO C
3280 E2=E*13+18
3290 FOR D=1 TO C
3291 IF Y$='Y' .AND. E .NE. D THEN GOTO 3330
3300 IF Z$(E) .NE. A$(D) THEN GOTO 3330
3310 IF B .LT. 8 THEN PRINT USING 3390, F$(B,D)
3320 IF B .GT.7 THEN PRINT USING 3400, I(B,D)
3330 NEXT D
3340 NEXT E
3350 PRINT '  '
3360 NEXT B
3361 IF Y$='Y' THEN PRINT USING 3380,'BALCODE'
3362 IF Y$ .NE. 'Y' THEN GOTO 3370
3363 FOR B=1 TO 6
3364 E2=B*13+18
3365 PRINT USING 3400,L(B)
3366 NEXT B
3367 PRINT '  '
3370 PRINT USING 3380,'..............................'
3371 FOR D=1 TO C
3372 E3=D*13+18
3374 PRINT USING 3402,'............'
3375 NEXT D
3376 PRINT USING 3404
3380 FORM C
3390 FORM POS E2,C
3400 FORM POS E2,PIC(ZZZ.#)
3402 FORM POS E3,C
3403 FORM SKIP8
3404 FORM SKIP 15
3410 END
```

Name: PRODGAS
Language: Basic
Desciption: Incorporates void volume into first and and second stage product gas compositions

```
10  PRINT 'INPUT NUMBER OF STAGES'
20  INPUT F
30  IF F=1 THEN LET Q=2                REM 2 GALLON QUENCH
40  IF F=2 THEN LET Q=1                REM 1 GALLON QUENCH
50  IF F=1 THEN GOTO 120
60  PRINT '1st STAGE'
70  S=1
80  PRINT 'INPUT AVERAGE RUN TEMPERATURE'
90  INPUT A1
100 GOTO 150
110 PRINT '2nd STAGE'
120 PRINT 'INPUT AVERAGE RUN TEMPERATURE'
130 INPUT A1
140 S=2
150 PRINT 'INPUT %CO2, C2H6, C3H8, H2, CH4, CO, H2S, NH3'
160 FOR B=1 TO 8
170 INPUT D(B)
180 NEXT B
190 PRINT 'INPUT CU. FT.'
200 INPUT A
210 IF S=1 THEN LET A=A+(1)/((A1+273)*.082057)        REM
    LITER AUTOCLAVE
220 IF S=1 .OR. S=2 THEN LET A=A+(3.7853)/((A1+273)*.082057)
230 A=A+(Q*3.7853)/(301*.082057)        REM 1 OR 2 GALLON
    QUENCH AT 28C
240 FOR B=1 TO 8
250 E(B)=E(B)+D(B)*A
260 NEXT B
270 E=E+A
271 IF S=2 THEN GOTO 281
280 IF F=2 GOTO 110
281 FOR B=1 TO 8
282 E(B)=E(B)/E
283 NEXT B
290 PRINT
300 PRINT
310 PRINT 'COMPOSITE PRODUCT GAS ANALYSIS'
320 E$(1)='%CO'
330 E$(2)='%C2H6'
340 E$(3)='%C3H8'
350 E$(4)='%H2'
360 E$(5)='%CH4'
370 E$(6)='%CO'
380 E$(7)='%H2S'
390 E$(8)='%NH3'
400 E$(9)='TOTAL GAS (CU. FT.)'
410 FOR B=1 TO 8
420 PRINT USING 430,E$(B),E(B)
430 FORM POS5,C,POS15,PIC(ZZ.##),SKIP
```

```
440 NEXT B
450 PRINT USING 460,E$(9),E
460 FORM SKIP,POS5,C,POS30,PIC(ZZ.###),SKIP5
470 END
```

Time Sample Calculations

Character Variables:

A$ - Temporary storage of file name

A$(C) - Array of file names

C$ - Residence time

D$ - Low time sample value

E$ - High time sample value

F$ - Input variable for balance code

H$ - Run number search variable

J$ - Maximum pressure

M$ - Maximum temperature

P$ - Run number

Q$ - Coal

R$ - Solvent

S$ - Additive

T$ - Date

Number Variables:

B - Number of Files

B2 - Number of runs in file

B3 - Loop variable

B4 - Loop variable

B7 - Flag

B9 - Loop variable

C(G3,G4) - Response factor * Area under curve

D(B) - Total of response factors

E1 - Number of time samples

E2 - Balance code selection

E3 - flag

E4 - flag

E5 - Input for number of variable combinations

E(G7,G8) - Mole percent of individual gases

F(B4) - Weight percent ash

G2 - Loop variable

G3 - Loop variable

G4 - Loop variable

G7 - Loop variable

G8 - Loop variable

G(B9) - Array for output selection

H3 - Percent hydrogen in feed gas

H(B4) - Microdistillation Residue

J(B) - Time at which sample was taken

K1 - Total grams in

K2 - Total liquids out

K(B4) - Percent THF soluble

M7 - Loop variable

N - Variable for output selection

N7 - Number of time samples taken

N(1-5) - Molecular weights of gases

Q(B4) - Total moles of individual gases at each time sample

R1 - Ratio of MAF/IOM out

R2 - Ratio of soluble residuum in/soluble residuum out

R3 - Difference in Grams of H_2 in and out

R4 - Difference in Grams of H_2 per time sample

Grams Input into System

R(1,1) - H_2

R(2,1) - CO

R(6,1) - H_2O

R(7,1) - H_2O

R(8,1) - Total Gas

R(9,1) - Ash

R(10,1) - Slurry

R(11,1) - MAF coal

R(12,1) - Soluble residuum

R(13,1) - Distillable oils

Total Grams Out of System

R(1,N7) - H_2

R(2,N7) - CO

R(3,N7) - CH_4

R(4,N7) - CO_2

R(5,N7) - C_2H_6

R(6,N7) - H_2O

R(7,N7) - H_2S

R(8,N7) - Total Gas

R(9,N7) - Ash

R(10,N7) - Slurry

R(11,N7) - IOM out

R(12,N7) - Soluble Residuum

R(13,N7) - Distillable Oils

S - Difference in grams of Ash in/out

S8 - Average Run Temperature

V(B,C) - Area of under curve from gas chromatograph analysis

W(1-6) - Total moles of individual gases

X - Number of time samples plus 2

Y - Number of time samples

Y(1-6) - Calculated response factors

Y(G2) - Ratio of total moles/total area under G.C. curve

Z - Number of time samples plus 1

Z1 - Balance code

Z3 - Change in grams of ash per time sample

Z9 - Balance Code

Timsam7
Language: Basic
Description: Program does material balance and tabulations for time sample data

```
 10 DIM J(9)
 20 DIM G(6)
 30 DIM V(8,9)
 40 DIM A$(32)
 50 DIM W(10)
 60 DIM Y(10)
 70 DIM C(7,8)
 80 DIM D(10)
 90 DIM E(7,8)
100 DIM Q(10)
110 DIM N(10)
120 DIM N$(17)
130 DIM R(17,9)
140 DIM R$(17,9)
150 DIM F(10)
160 DIM H(10)
170 DIM K(10)
180 DIM X$(13)
190 B7 = 0
200 E3 = 0
210 E2 = 0
220 E5 = 5
230 Q = 0
240 PRINT 'SELECT FUNCTION'
250 PRINT '   1.  GRAM TAB'
260 PRINT '   2.  MOL/GRAM PLOT'
270 PRINT '   3.  DATA PRINT'
280 PRINT '   4.  MAF TAB'
290 PRINT'   5.  MAF PLOT'
300 PRINT'   6.  ALL OF THE ABOVE'
310 PRINT'   7.  COMBINATIONS OF ABOVE'
320 INPUT N
330 IF N LT. 6 THEN GOTO 450
340 IF N=7 THEN GOTO 390
350 FOR J = 1TO5
360 G(J) = J
370 NEXT J
380 IF N  LT.  7 THEN GOTO 450
390 PRINT' NUMBER OF COMBINATIONS'
400 INPUT E5
410 PRINT'INPUT THE VARIABLES'
420 FOR B9 = 1TOE5
430 INPUT G(B9)
440 NEXT B9
450 PRINT 'DO YOU WISH TO CHOOSE BALANCE CODE?'
460 INPUT F$
470 IF F$ = 'N' THEN GOTO 530
480 PRINT'SELECT BALANCE CODE'
```

```
490 PRINT'         1'
500 PRINT'         2'
510 PRINT'         3--BOTH 1 AND 2'
520 INPUT E2
530 IF E2 = 1 THEN LET Z9 = 1
540 IF E2 = 2 THEN LET Z9 = 2
550 IF E2 = 3 THEN LET Z9 = 1
560 IF N GT. 5 THEN LET B7 = 1
570 PRINT 'INPUT TIME SAMPLE RUN NUMBERS,
    LOW AND HIGH VALUES.'
580 INPUT D$,E$
590 GET 'TSFILE',B
600 FOR C=1 TO B
610 GET 'TSFILE', A$(C)
620 NEXT C
630 FOR B1=1 TO B
640 A$=A$(B1)
650 GET A$,B2
660 FOR B3=1 TO B2
670 GET A$, H$
680 J(1) = 0
690 GET A$, E1
700 Y = E1
710 Z = E1 + 1
720 X = E1 + 2
730 IF E1 = 5 GOTO 740 ELSE 800
740 REM J(B) TIME FOR EACH TIME SAMPLE
741 IF E1 = 5 .AND. H$  LT.  'N138' THEN GOTO 750
    ELSE GOTO 791
750 J(2) = 4
760 J(3) = 8
770 J(4) = 12
780 J(5) = 16
790 J(6) = 20
791 J(2) = 1
792 J(3) = 3
793 J(4) = 6
794 J(5) = 10
795 J(6) = 20
800 IF E1 = 7 GOTO 810 ELSE 870
810 J(2) = 1
820 J(3) = 3
830 J(4) = 6
840 J(5) = 10
850 J(6) = 20
860 J(7) = 40
870 J(8) = 60
880 T = 98/Z
890 FOR B=1 TO 6
900 FOR C = 2 TO Z
910 GET A$, V(B,C)
920 NEXT C
930 NEXT B
940 GET A$, W(1),W(2),W(3),W(4),W(5),W(6)
```

```
 950 FOR B4=2 TO Z
 960 GET A$, Q(B4)
 970 NEXT B4
 980 FOR B4=2 TO Z
 990 GET A$, F(B4),H(B4),K(B4)
1000 NEXT B4
1010 GET A$, R(1,1),R(2,1),R(6,1),R(7,1),
     R(8,1),R(9,1),R(10,1),R(11,1),R(12,1),R(13,1)
1020 FOR M7 = 1 TO 13
1030 N7 = X
1040 GET A$,R(M7,N7)
1050 NEXT M7
1060 GET A$, Q2,P$,Q$,R$,S$,T$,C$,S8,M$,J$,H3,Z1
1070 GET A$,Y(1),Y(2),Y(3),Y(4),Y(5),Y(6)
1080 IF F$ = 'N' THEN GOTO 1100
1090 GOTO 1110
1100 GET 'FORCECOD',Z9
1110 IF H$ GT. =D$ & H$ LT. =E$ THEN GOTO 1130
1120 GOTO 1350
1130 IF B7 = 0 THEN GOTO 1160
1140 FOR B9 = 1TOE5
1150 N = G(B9)
1160 IF N=1 THEN GOSUB 1410
1170 IF N=1 THEN GOSUB 2490
1180 IF N=1 THEN GOSUB 3230
1190 IF N=2 THEN GOSUB 1410
1200 IF N=2 THEN GOSUB 4480
1210 IF N=3 THEN GOSUB 6160
1220 IF N=4 THEN GOSUB 1410
1230 IF N=4 THEN GOSUB 2490
1240 IF N=4 THEN GOSUB 3230
1250 IF N=5 THEN GOSUB 1410
1260 IF N=5 THEN GOSUB 2490
1270 IF N=5 THEN GOSUB 4480
1280 NEXT B9
1290 IF B7 = 0 .AND. E3 = 3 THEN GOTO 1310
1300 IF E2 LT. 3 GOTO 1350
1310 IF E2 = 3 .AND. Z9 = 2 GOTO 1340
1320 IF E2 = 3 .AND. Z9 = 1 THEN LET Z9 = 2
1330 GOTO 1140
1340 IF E2 = 3 .AND. Z9=2 THEN LET Z9=1
1350 NEXT B3
1360 NEXT B1
1370 PRINT ' '
1380 PRINT ' '
1390 PRINT ' '
1400 STOP
1410 IF Z1=1 THEN GOTO 1440
1420 IF Z1=2 THEN GOTO 1590
1430 IF Z1=3 THEN GOTO 1530
1440 FOR G2=1 TO 6
1450 IF V(G2,Z) = 0 THEN GOTO 1480
1460 LET Y(G2) = W(G2)/V(G2,Z)
1470 GOTO 1510
```

```
1480 IF V(5,Z) = 0 THEN LET Y(5)=(.393)*(Y(1)/.1)
1490 GOTO 1510
1500 LET Y(G2)=0
1510 NEXT G2
1520 GOTO 1590
1530 LET Y(1)=.1000
1540 LET Y(2)=1.583
1550 LET Y(3)=.410
1560 LET Y(4)=1.461
1570 LET Y(5)=.393
1580 LET Y(6)=.9896
1590 FOR G3=1 TO 6
1600 FOR G4=2 TO Z
1610 LET C(G3,G4)=Y(G3)*V(G3,G4)
1620 NEXT G4
1630 NEXT G3
1640 FOR B=2 TO Z
1650 LET D(B)=0
1660 FOR C3=1 TO 6
1670 LET D(B)=D(B)+C(C3,B)
1680 NEXT C3
1690 NEXT B
1700 FOR G7=1 TO 6
1710 FOR G8=2 TO Z
1720 IF D(G8)=0 THEN GOTO 1750
1730 LET E(G7,G8)=(C(G7,G8)/D(G8))*100
1740 GOTO 1760
1750 LET E(G7,G8)=0
1760 NEXT G8
1770 NEXT G7
1780 LET N(1)=2.016
1790 LET N(2)=28.011
1800 LET N(3)=16.043
1810 LET N(4)=44.011
1820 LET N(5)=30.07
1830 LET N(6)=18.016
1840 FOR C=1TO6
1850 FOR B=2TOZ
1860 LET R(C,B) = (E(C,B)*Q(B)/100)*N(C)
1870 NEXT B
1880 NEXT C
1890 LET R3=R(7,1)-R(7,X)
1900 LET R4 = R3/Y
1910 FOR B=2TOZ
1920 C3 =B-1
1930 LET R(7,B) = R(7,1)-R4*C3
1940 NEXT B
1950 FOR B=2 TO Z
1960 LET R(8,B)=R(1,B)+R(2,B)+R(3,B)+R(4,B)
+R(5,B)+R(6,B)+R(7,B)
1970 NEXT B
1980 LET S = R(9,1)-R(9,X)
1990 LET Z3=S/Y
2000 FOR B=2TOZ
```

```
2010 C3 = B-1
2020 LET R(9,B) = R(9,1)-Z3*C3
2030 NEXT B
2040 FOR B =2 TO Z
2050 IF F(B)=0 THEN GOTO 2110
2060 LET R(11,B)=(R(9,B)/F(B))*H(B)*(K(B)/100)-R(9,B)
2070 LET R(12,B)=(R(9,B)/F(B))*H(B)*((100-K(B))/100)
2080 IF Z9=2 THEN GOTO 2170
2090 LET R(13,B)=((R(9,B))/(F(B)/100))*(1-(H(B)/100))
2100 GOTO 2170
2110 LET R(9,B)=0
2120 LET R(10,B)=0
2130 LET R(11,B)=0
2140 LET R(12,B)=0
2150 IF Z9=2 THEN GOTO 2170
2160 LET R(13,B)=0
2170 NEXT B
2180 IF Z9=1 THEN GOTO 2320
2190 IF R(11,Z)=0 THEN Z9=1
2200 IF R(12,Z)=0 THEN Z9=1
2210 IF Z9=1 THEN GOTO 2040
2220 LET R1=R(11,X)/R(11,Z)
2230 LET R2=R(12,X)/R(12,Z)
2240 FOR B = 2 TO Z
2250 IF F(B)=0 THEN GOTO 2300
2260 LET R(11,B)=R(11,B)*R1
2270 LET R(12,B)=R(12,B)*R2
2280 LET R(13,B)=((R(9,B))/(F(B)/100))*(1-(H(B)/100))
2290 GOTO 2310
2300 LET R(11,B)=0
2310 NEXT B
2320 FOR C=2TOX
2330 K1=0
2340 K2=0
2350 FOR B = 1TO13
2360 IF N=1 THEN GOTO 2380
2370 IF B=10 THEN GOTO 2410
2380 IF B GT. =8 THEN GOTO 2390 ELSE 2410
2390 K1 =K1+R(B,1)
2400 K2 = K2+R(B,C)
2410 NEXT B
2420 R(14,C) = K1-K2
2430 IF N=1 GOTO 2450
2440 R(14,C) =R(14,C)/R(11,1)*100
2450 R(15,C) = R(14,C)+R(13,C)
2460 NEXT C
2470 RETURN
2480 PRINT ' '
2490 REM THIS SUBROUTINE IS FOR CALCULATIONG
     NUMBERS FOR TABLES AND GRAPHS
2500 FOR B=1 TO 15
2510 FOR C = 1 TO X
2520 LET R$(B,C)='0.00'
2530 NEXT C
```

```
2540 NEXT B
2550 FOR B = 2 TO Z
2560 IF R(1,B)=0 THEN GOTO 2580
2570 GOTO 2610
2580 FOR C=1 TO 8
2590 LET R$(C,B)='.....'
2600 NEXT C
2610 NEXT B
2620 FOR B = 2 TO Z
2630 IF R(6,B)=0 THEN GOTO 2650
2640 GOTO 2660
2650 LET R$(6,B)='.....'
2660 NEXT B
2670 FOR B=1 TO 6
2680 FOR C = 2 TO Z
2690 IF V(B,C)=0 THEN GOTO 2710
2700 GOTO 2720
2710 LET R$(B,C)='.....'
2720 NEXT C
2730 NEXT B
2740 FOR B = 2 TO Z
2750 IF R(7,1)=0 THEN GOTO 2770
2760 GOTO 2780
2770 LET R$(7,B)='.....'
2780 NEXT B
2790 FOR B = 2 TO Z
2800 IF R(6,B) = 0 THEN GOTO 2810 ELSE 2880
2810 LET R$(14,B) = '.....'
2820 LET R$(15,B) = '.....'
2830 IF R(13,B) LT. =0 .OR. R(11,B) GT. R(11,1) THEN GOTO
     2850
2840 GOTO 2880
2850 FOR C=9 TO 15
2860 LET R$(C,B)='.....'
2870 NEXT C
2880 NEXT B
2890 FOR B = 2 TO Z
2900 IF R(10,1)=0 THEN GOTO 2920
2910 GOTO 2930
2920 LET R$(10,B)='.....'
2930 NEXT B
2940 IF N GT. 3 THEN GOTO 2950 ELSE 3220
2950 FOR C=1 TO 15
2960 FOR B = 1 TO X
2970 IF C=10 THEN LET R$(10,B)='..'
2980 IF C=7 THEN GOTO 3210
2990 IF C=8 THEN GOTO 3210
3000 IF C=9 THEN GOTO 3210
3010 IF B=1 THEN GOTO 3180
3020 IF B = X THEN GOTO 3180
3030 IF R$(C,B)='.....' THEN GOTO 3180
3040 IF C=1 THEN LET R(1,B)=((R(1,B)-R(1,1))/R(11,1))*100
3050 IF C=2 THEN LET R(2,B)=((R(2,B)-R(2,1))/R(11,1))*100
3060 IF C=3 THEN LET R(3,B)=R(3,B)/R(11,1)*100
```

```
3070 IF C=4 THEN LET R(4,B)=(R(4,B)/R(11,1))*100
3080 IF C=5 THEN LET R(5,B)=R(5,B)/R(11,1)*100
3090 IF C=6 THEN LET R(6,B)=((R(6,B)-R(6,1))/R(11,1))*100
3100 IF C=10 GOTO 3110 ELSE GOTO 3140
3110 IF F(B)=0 THEN LET R$(10,B)='.....' ELSE GOTO 3130
3120 GOTO 3030
3130 IF C=10 THEN LET R(10,B)=((R(9,B)/(F(B)/100))
     -(R(10,1)))/R(11,1)*100
3140 IF C=11 THEN LET R(11,B)=((R(11,1)
     -R(11,B))/R(11,1))*100
3150 IF C=12 THEN LET R(12,B)=((R(12,B)-R(12,1))
     /R(11,1))*100
3160 IF C=13 THEN LET R(13,B)=(R(13,B)-R(13,1))/R(11,1)*100
3170 IF C=15 THEN LET R(15,B)=R(14,B)+R(13,B)
3180 IF B=1 THEN LET R(10,1)=(R(11,1)+R(12,1)+R(13,1))
3190 IF B=X THEN LET R(10,X) = (R(11,X)+R(12,X)+R(13,X))
3200 NEXT B
3210 NEXT C
3220 RETURN
3230 REM THIS SUBROUTINE IS FOR PRINTING A NUMERICAL TABLE
3240 PRINT ' '
3250 PRINT ' '
3260 PRINT TAB(10);'.........................'
3270 PRINT ' '
3280 PRINT ' '
3290 PRINT ' '
3300 PRINT ' '
3310 PRINT TAB(5);'RUN #';TAB(25);P$
3320 PRINT TAB(5);'COAL';TAB(25);Q$
3330 PRINT TAB(5);'SOLVENT';TAB(25);R$
3340 PRINT TAB(5);'ADDITIVE';TAB(25);S$
3350 PRINT TAB(5);'DATE';TAB(25);T$
3360 PRINT TAB(5);'CONDITIONS'
3370 PRINT TAB(5);'RES TIME (MIN)';TAB(25);C$
3380 PRINT TAB(5);'AVG TEMP (C)';TAB(24);S8
3390 PRINT TAB(5);'MAX TEMP (C)';TAB(25);M$
3400 PRINT TAB(5);'MAX PRESS, PSIG';TAB(25);J$
3410 PRINT USING 3420, '% H2 IN FEED GAS',H3
3420 FORM POS5,C,POS25,PIC(ZZZ.##), SKIP
3430 PRINT ' '
3440 IF N=4 THEN PRINT TAB(70);'NET YIELDS (WT% MAF)'
     ELSE PRINT TAB(60);
3450 IF N=4 THEN GOTO 3470 ELSE PRINT USING 3460,' '
3460 FORM C,SKIP1
3470 H1 = 3*T-5
3480 IF N = 4 THEN GOTO 3500
3490 H1 = 2*T-5
3500 H = X*T
3510 GOSUB 6770
3520 Q=0
3530 FOR J = 1 TO H
3540 Q = Q+1
3550 IF J LT. H1 THEN PRINT USING 3570,' '
3560 IF J GT. =H1 THEN PRINT USING 3570,'.'
```

```
3570 FORM POSQ,C
3580 NEXT J
3590 PRINT ' '
3600 H = 2*T-4
3610 GOSUB 6770
3620 IF N=4 THEN PRINT USING 3630,'GRAMS @','GRAMS @'
3630 FORM  POSH,C,POS123,C,SKIP1
3640 PRINT USING 3650,'COMPONENTS'
3650 FORM POS1,C
3660 FOR B = 1 TO Z
3670 H = (T*B) + (T-4) +1
3680 GOSUB 6770
3690 Q = H+2
3700 PRINT USING3710,'T=';J(B)
3710 FORM POSH,C,POSQ,PIC(##)
3720 NEXT B
3730 PRINT USING  3740,'TERMINATION'
3740 FORM POS120,C,SKIP2
3750 LET N$(1)='H2'
3760 LET N$(2)='CO'
3770 LET N$(3)='CH4'
3780 LET N$(4)='CO2'
3790 LET N$(5)='C2H6'
3800 LET N$(6)='H2O'
3810 LET N$(7)='H2S'
3820 LET N$(8)='TOTAL GAS'
3830 LET N$(9)='ASH'
3840 IF N=4 THEN N$(10)='SLURRY' ELSE N$(10)='ADDITIVE'
3850 IF N=4 THEN N$(11)='MAF' ELSE N$(11)='MAF/IOM'
3860 LET N$(12)='SOL RESID'
3870 LET N$(13)='DISTILLATES (LIQ)'
3880 LET N$(14)='DISTILLATES (VAP)'
3890 LET N$(15)='DISTILLABLES'
3900 LET N$(16)='CONVERSION'
3910 IF N=4 THEN LET Q7=16 ELSE Q7=15
3920 IF N=4 THEN GOSUB 6980
3930 FOR C=1 TO Q7
3940 FOR B=1TOX
3950 IF C=16 .AND. B=1 THEN PRINT ' '
3960 IF N=4 THEN GOTO 3970 ELSE GOTO 4010
3970 IF C=7 THEN GOTO 4330
3980 IF C=8 THEN GOTO 4330
3990 IF C=9 THEN GOTO 4330
4000 IF C GT. 13 .AND. B=X THEN GOTO 4330
4010 IF B=1 THEN GOTO 4050
4020 IF B GT. =2 .AND. B LT. X THEN GOTO 4080
4030 IF B = X THEN GOTO 4170
4040 GOTO 4320
4050 IF C=8 THEN PRINT '  '
4060 PRINT USING 4070,N$(C)
4070 FORM POS1,C
4080 H = (T*B) + (T-7) +2
4090 IF C GT. 13 .AND. B=1 THEN GOTO 4320
4100 GOSUB 6770
```

```
4110 IF R$(C,B)='.....' THEN GOTO 4210
4120 IF C GT. 13 .AND. B=Z THEN PRINT USING 4300, R(C,B)
4130 IF C GT. 13 .AND. B=Z THEN GOTO 4330
4140 PRINT USING 4150,R(C,B)
4150 FORM POSH,PIC(ZZZZ.##)
4160 GOTO 4320
4170 PRINT USING 4180,R(C,B)
4180 FORM POS 121,PIC(ZZZZ.##),SKIP
4190 IF C=8 THEN PRINT '  '
4200 GOTO 4320
4210 H = H+2
4220 IF C GT. 13 .AND. B= Z THEN PRINT USING 4230, R$(C,B)
4230 FORM POSH,C,SKIP
4240 IF C GT. 13 .AND. B=Z THEN GOTO 4330
4250 IF C=8 .AND. B=1 THEN PRINT '  '
4260 PRINT USING 4270,R$(C,B)
4270 FORM POSH,C
4280 GO TO 4320
4290 PRINT USING 4300 , R(C,B)
4300 FORM POSH,PIC(ZZZZ.##),SKIP
4310 GO TO 4330
4320 NEXT B
4330 NEXT C
4340 PRINT ' '
4350 PRINT ' '
4360 PRINT USING 4370,'Z1=',Z1
4370 FORM POS5,C,POS9,PIC(Z),SKIP1
4380 PRINT USING 4390,'Z9=',Z9
4390 FORM POS5,C,POS9,PIC(Z),SKIP1
4400 PRINT TAB(10);'........................'
4410 IF N=1 THEN LET N1=5 ELSE N1=8
4420 PRINT USING 4430,'  '
4430 FORM C, SKIP N1
4440 RETURN
4450 PRINT ' '
4460 PRINT ' '
4470 PRINT ' '
4480 REM THIS ROUTINE IS FOR PRINT A POINT GRAPH
     OF ANY VARIABLE IN TIME SAMPLE DATA
4490 IF N=5 THEN LET I3=3
4500 IF N=2 THEN LET I3=1
4510 IF I3=3 THEN GOTO 4520 ELSE GOTO 4530
4520 FOR I2=1 TO2
4530 J=10
4540 I9=0
4550 I1=0
4560 I7=1
4570 I8=0
4580 B = 1
4590 PRINT TAB(5);'RUN #';TAB(25);P$
4600 PRINT TAB(5);'COAL';TAB(25);Q$
4610 PRINT TAB(5);'SOLVENT';TAB(25);R$
4620 PRINT TAB(5);'ADDITIVE';TAB(25);S$
4630 PRINT TAB(5);'AVG TEMP, C';TAB(25);S8
```

```
4640 PRINT TAB(5);'MAX PRESS, PSIG';TAB(25);J$
4650 PRINT USING 4660, '% H2 IN FEED GAS',H3
4660 FORM POS5,C,POS25,PIC(ZZZ.##), SKIP
4670 X$(1)='H'
4680 X$(2)='C'
4690 C$(3)='M'
4700 X$(4)='2'
4710 X$(5)='E'
4720 X$(6)='W'
4730 X$(7)='O'
4740 X$(8)='TG'
4750 X$(9)='A'
4760 X$(10)='L'
4770 X$(11)='I'
4780 X$(12)='S'
4790 X$(13)='D'
4800 GOTO 4840
4810 I7=1
4820 B = 1
4830 G = 0
4840 IF I3=1 THEN PRINT USING 4850,'GMOL'
4850 FORM POS65,C,SKIP2
4860 IF I3=3 THEN PRINT USING 4850,'NET YIELDS (MAF)'
4870 IF I3=2 THEN PRINT USING 4850,'GRAMS'
4880 IF I3=1 THEN PRINT USING 4920,'0','1.0','2.0','3.0',
     '4.0','5.0','6.0','7.0','8.0','9.0','10.0'
4890 IF I3=2 THEN PRINT USING 4920,'00','20','40','60',
     '80','100','120','140','160','180','120'
4900 IF I3=3 .AND. I2=1 THEN PRINT USING 4920,'-280',
     '-240','-200','-160','-120','-80','-40','00','40',
     '80','120'
4910 IF I3=3 .AND. I2=2 THEN PRINT USING 4920,'-50',
     '-40','-30','-20','-10','00','10','20','30','40','50'
4920 FORM POS15,C,POS25,C,POS35,C,POS45,C,POS55,C,POS65,
     C,POS75,C,POS85,C,POS95,POS105,C,POS115,C,SKIP1
4930 PRINT '......';
4940 PRINT USING 4920,'▮','▮','▮','▮','▮','▮','▮','▮','▮
4950 PRINT TAB(15);'....................................'
4960 I7=0
4970 FOR I4=1 TO Z
4980 IF I3=1 THEN GOTO 4990 ELSE GOTO 5190
4990 LET A1=15+((R(1,I4)/N(1))*10)
5000 IF A1 GT. 115 .OR. A1 LT. 15 THEN LET A1=115
5010 IF A1=15 THEN LET A1=1
5020 LET A2=15+((R(2,I4)/N(2))*10)
5030 IF A2 GT. 115 .OR. A2 LT. 15 THEN LET A2=116
5040 IF A2=15 THEN LET A2=2
5050 LET A3=15+((R(3,I4)/N(3))*10)
5060 IF A3 GT. 115 .OR. A3 LT. 15 THEN LET A3=117
5070 IF A3=15 THEN LET A3=3
5080 LET A4=15+((R(4,I4)/N(4))*10)
5090 IF A4 GT. 115 .OR. A4 LT. 15 THEN LET A4=118
5100 IF A4=15 THEN LET A4=4
5110 LET A5=15+((R(5,I4)/N(5))*10)
```

```
5120 IF A5 GT. 115 .OR. A5 LT. 15 THEN LET A5=119
5130 IF A5=15 THEN LET A5=5
5140 LET A6=15+((R(6,I4))/N(6)*10)
5150 IF A6 GT. 115 .OR. A6 LT. 15 THEN LET A6=120
5160 IF A6=15 THEN LET A6=6
5170 PRINT USING 5180,J(B),'+','H','C','M','2','E','W'
5180 FORM POS12,PIC(##),POS14,C,POSA1,C,POSA2,C,POSA3,C,
     POS A4,C,POSA5,C,POSA6,C,SKIP1
5190 IF I3=3 .AND. I2=1 THEN GOTO 5200 ELSE GOTO 5410
5200 IF I4=1 THEN PRINT USING 5230,J(B),'+' ELSE GOTO 5240
5210 IF A4=65 THEN LET A4=4
5220 GOTO 5640
5230 FORM POS12,PIC(##),POS14,C,SKIP
5240 LET A1=85+(R(10,I4)/4)
5250 IF A1 GT. 115 .OR. A1 LT. 15 THEN LET A1=115
5260 IF A1=85 THEN LET A1=1
5270 LET A2=85+(R(11,I4)/4)
5280 IF A2 GT. 115 .OR. A2 LT. 15 THEN LET A2=116
5290 IF A2=85 THEN LET A2=2
5300 LET A3=85+(R(12,I4)/4)
5310 IF A3 GT. 115 .OR. A3 LT. 15 THEN LET A3=117
5320 IF A3=85 THEN LET A3=3
5330 LET A4=85+(R(13,I4)/4)
5340 IF A4=85 THEN LET A4=4
5350 IF A4 GT. 115 .OR. A4 LT. 15 THEN LET A4=118
5360 LET A5=85+((R(1,I4)+R(2,I4)+R(3,I4)+R(4,I4))/4)
5370 IF A5 GT. 115 .OR. A5 LT. 15 THEN LET A5=119
5380 IF A5=85 THEN LET A5=5
5390 PRINT USING 5400,J(B)'+','L','D','S','I','T'
5400 FORM POS12,PIC(##),POS14,C,POSA1,C,POSA4,C,POSA3,
     C,POSA2,C,POSA5,C,SKIP1
5410 IF I3=3 .AND. I2=2 THEN GOTO 5420 ELSE GOTO 5640
5420 IF I4=1 THEN PRINT USING 5230,J(B),'+' ELSE GOTO 5440
5430 GOTO 5640
5440 LET A1=65+(R(1,I4)/1)
5450 IF A1 GT. 115 .OR. A1 LT. 15 THEN LET A1=115
5460 IF A1=65 THEN LET A1=1
5470 LET A2=65+(R(2,I4)/1)
5480 IF A2 GT. 115 .OR. A2 LT. 15 THEN LET A2=116
5490 IF A2=65 THEN LET A2=2
5500 LET A3=65+(R(3,I4)/1)
5510 IF A3 GT. 115 .OR. A3 LT. 15 THEN LET A3=117
5520 IF A3=65 THEN LET A3=3
5530 LET A4=65+(R(4,I4)/1)
5540 IF A4 GT. 115 .OR. A4 LT. 15 THEN LET A4=118
5550 IF A4=65 THEN LET A4=4
5560 LET A5=65+(R(5,I4)/1)
5570 IF A5=65 THEN LET A5=5
5580 IF A5 GT. 115 .OR. A5 LT. 15 THEN LET A5=119
5590 LET A6=65+(R(6,I4)/1)
5600 IF A6=65 THEN LET A6=6
5610 IF A6 GT. 115 .OR. A6 LT. 15 THEN LET A6=120
5620 PRINT USING 5630,J(B),'+','H','C','M','2','E','W'
5630 FORM POS12,PIC(##),POS14,C,POSA1,C,POSA2,C,
```

```
     POSA3,C,POSA4,C,POSA5,C,POSA6,C,SKIP1
5640 C = B+1
5650 G = J(Z)/30
5660 F1 = J(C)/G
5670 F2 = J(B)/G
5680 H = F1-F2
5690 IF H LT. 1 THEN GOTO 6080
5700 GOSUB 6770
5710 FOR I6=1TOH
5720 I7=I7+1
5730 FORM C
5740 FORM POS1,C
5750 IF I4=Z THEN GOTO 6090
5760 IF B LT. 3 GOTO 5840
5770 IF I7 LT. 10 THEN LET I8=0
5780 I8 = I8+1
5790 IF I8=2 .AND. H GT. =6 THEN PRINT USING 5740,'T'
5800 IF I8=3 .AND. H GT. =6 THEN PRINT USING 5740,'I'
5810 IF I8=4 .AND. H GT. =6 THEN PRINT USING 5740,'M'
5820 IF I8=5 .AND. H GT. =6 THEN PRINT USING 5740,'E'
5830 IF I8=7 .AND. H GT. =6 THEN PRINT USING 5740,'(MIN)'
5840 IF I3 LT. 3 THEN PRINT USING 6040,'▮'
5850 IF C=Z THEN GOTO 5860 ELSE GOTO 6020
5860 IF I3=3 .AND. I2=1 THEN GOTO 5960
5870 I9=I9+1
5880 IF I9=1 THEN PRINT USING 5940,'H=H2'
5890 IF I9=2 THEN PRINT USING 5940,'C=CO'
5900 IF I9=3 THEN PRINT USING 5940,'M=CH4'
5910 IF I9=4 THEN PRINT USING 5940,'2=CO2'
5920 IF I9=5 THEN PRINT USING 5940,'E=C2H6'
5930 IF I9=6 THEN PRINT USING 5940,'W=H20'
5940 FORM POS1,C
5950 GOTO 6020
5960 I1=I1+1
5970 IF I1=1 THEN PRINT USING 5940,'L=DIS & SRL'
5980 IF I1=2 THEN PRINT USING 5940,'D=DIS OILS'
5990 IF I1=3 THEN PRINT USING 5940,'T=TOTAL GAS'
6000 IF I1=4 THEN PRINT USING 5940,'S=SOL RES'
6010 IF I1=5 THEN PRINT USING 5940,'I=IOM'
6020 IF I3=3 .AND. I2=1 THEN PRINT USING 6050,'▮','▮'
6030 IF I3=3 .AND. I2=2 THEN PRINT USING 6060,'▮','▮'
6040 FORM POS14,C,SKIP1
6050 FORM POS14,C,POS85,C,SKIP1
6060 FORM POS14,C,POS65,C,SKIP1
6070 NEXT I6
6080 B = B+1
6090 NEXT I4
6100 N2=38-I7-Y
6110 PRINT USING 6120,' '
6120 FORM C,SKIPN2
6130 IF I3=3 THEN GOTO 6140 ELSE GOTO 6150
6140 NEXT I2
6150 RETURN
6160 REM THIS IS THE DATA INPUT SUMMARY TABLE SUBROUTINE
```

```
6170 GOSUB 6840
6180 PRINT USING 6190
6190 FORM SKIP N1
6200 PRINT USING 6210,'DATA FOR RUN #';P$;'COMPONENTS
     AT TERM COMPONENTS AT T=0'
6210 FORM POS1,C,C,POS78,C,SKIP
6220 FOR B=1 TO 6
6230 PRINT USING 6720,V(B,2)
6240 FOR C = 3 TO Z
6250 PRINT USING 6730, V(B,C)
6260 NEXT C
6270 IF R(B,1) = 0 GOTO 6300
6280 PRINT USING 6670, R(B,X),R(B,1)
6290 GOTO 6310
6300 PRINT USING 6680,R(B,X)
6310 NEXT B
6320 PRINT USING  6330,R(7,X),R(7,1)
6330 FORM POS 83,PIC(ZZZ.##),POS 104,PIC(ZZZ.##),SKIP
6340 PRINT USING 6350,'MOLES AT T=0',R(8,X),R(8,1)
6350 FORM POS1,C,POS83,PIC(ZZZ.##),POS 104,PIC(ZZZ.##),SKIP
6360 FOR Z8=1 TO 6
6370 PRINT USING 6650,W(Z8)
6380 B=Z8+8
6390 IF B LT. 14 GO TO 6420
6400 PRINT USING 6740,'TOTAL'
6410 GO TO 6440
6420 PRINT USING 6670,R(B,X),R(B,1)
6430 NEXT Z8
6440 PRINT USING 6450,'TOTAL MOLES AT T=';J(Z);Q2
6450 FORM POS1,C,PIC(ZZ),POS83,PIC(ZZZ.##),SKIP
6460 FOR Z8 = 2 TO Z
6470 PRINT USING 6660 ,Q(Z8)
6480 NEXT Z8
6490 PRINT USING 6750
6500 PRINT 'ASH,MDR,AND THF INSOL'
6510 FOR Z8 = 2 TO Z
6520 PRINT USING 6710,F(Z8),H(Z8),K(Z8)
6530 NEXT Z8
6540 PRINT USING 6750
6550 PRINT USING 6690,P$,Q$
6560 PRINT USING 6690,R$,S$
6570 PRINT USING 6690,T$,C$
6580 PRINT USING 6660,S8
6590 PRINT USING 6690,M$,J$
6600 PRINT USING 6660,H3
6610 PRINT USING 6660,Z1
6620 PRINT USING 6700,Y(1),Y(2),Y(3),Y(4),Y(5),Y(6)
6630 PRINT USING 6640
6640 FORM SKIP N2
6650 FORM POS10,PIC(ZZZ.##)
6660 FORM POS10,PIC(ZZZ.##),SKIP
6670 FORM POS 83,PIC(ZZZ.##),POS 104,PIC(ZZZ.##),SKIP
6680 FORM POS 83,PIC(ZZZ.##),SKIP
6690 FORM POS10,C,POS20,C,SKIP
```

```
6700 FORM POS10,PIC(ZZZ.###),POS18,PIC(ZZZ.###)
POS42,PIC(ZZZ.###),POS50,PIC(ZZZ.###),SKIP
6710 FORM POS10,PIC(ZZZ.##),POS20,PIC(ZZZ.##)
6720 FORM POS 1, PIC(ZZZ.###)
6730 FORM '  ',PIC(ZZZ.###)
6740 FORM SKIP1,POS83,C,SKIP1
6750 FORM SKIP1
6760 RETURN
6770 I=0
6780 H=H-1
6790 I=I+1
6800 IF H LT. 1 THEN GOTO 6820
6810 GOTO 6780
6820 H=I
6830 RETURN
6840 REM THIS ROUTINE CENTERS DATA VERTICALLY ON PAGE
6850 N =(50-(28+2*E1))/2
6860 N3=0
6870 N=N-1
6880 N3=N3+1
6890 IF N GT. 0 .AND. N LT. .99 THEN GOTO 6920
6900 IF N=0 THEN GOTO 6950
6910 GOTO 6870
6920 N1=N3
6930 N2=N3+1
6940 GOTO 6970
6950 N1=N3
6960 N2=N3
6970 RETURN
6980 FOR B=2 TO Z
6990 R(11,B)=100-R(11,B)
7020 R(16,B) = 100-R(11,B)
7030 NEXT B
7040 RETURN
7050 END
```

```
NAME: 4090 NORMAN7
CONTENT: BASIC
LENGTH: 2977 CHARS, 110 LINES
HIGHEST LINE NUMBER: 1250

  10 DIM P(15)
  20 DIM N(15)
  30 DIM G(15)
  40 DIM F(15)
  50 DIM N$(15)
  60 DIM M$(15)
  70 PRINT 'INPUT THE RUN NUMBER'
  80 INPUT V$
  90 PRINT 'INPUT THE GRAMS OF CO, H2, H2S, AND H2O THAT WAS
     CHARGED'
 100 INPUT C,H,H2,W
 110 LET C1=C/28.011
 120 LET H1=H/2.016
 130 LET W1=W/18.016
 135 LET H3=H2/34.032
 140 LET M=C1+H1+W1+H3
 141 IF V$  GT.  'N160' THEN GOTO 202
 150 PRINT 'INPUT THE BAROMETRIC PRESSURE, VOLUME, WET
     TEST METER TEMP,AND THE AVG RUN TEMP FOR THE PRODUCT
     GAS'
 160 INPUT P,V,F,F1
 170 LET M1=P*V*453.6/(21.85*(F+460))
 180 LET M2=2*3.7853/(301*.082057)
 190 LET M3=3.7853/((F1+273)*.082057)
 200 LET M4=M1+M2+M3
 201 GOTO 210
 202 PRINT 'INPUT THE VOLUME OF PRODUCT GAS FROM'
 203 PRINT 'MATBAL DATA SHEET'
 204 INPUT M4
 210 PRINT 'INPUT THE % CO2, % C2H6, % H2, % CH4, % CO,
     AND % H2S IN THE PRODUCT GAS'
 220 INPUT P(1),P(2),P(3),P(4),P(5),P(6)
 230 LET T=P(1)+P(2)+P(3)+P(4)+P(5)+P(6)
 240 FOR B=1 TO 6
 250 LET P(B)=P(B)*100/T
 260 NEXT B
 270 LET N(1)=44.011
 280 LET N(2)=30.07
 290 LET N(3)=2.016
 300 LET N(4)=16.043
 310 LET N(5)=28.011
 315 LET N(6)=34.032
 320 LET N(7)=18.016
 330 FOR B=1 TO 6
 340 LET G(B)=P(B)*M4*N(B)/100
 350 NEXT B
 360 LET T1=G(1)+G(2)+G(3)+G(4)+G(5)+G(6)
 370 PRINT 'INPUT THE GRAMS OF H20, ASH, IOM,
     SOL. RESID., AND DISTILL. OILS OUT'
```

```
 380 INPUT G(7),G(8),G(9),G(10),G(11)
 390 PRINT 'INPUT THE TOTAL GRAMS OF SLURRY AND GAS CHARGED'
 400 INPUT T2
 410 LET T3=T1+G(7)+G(8)+G(9)+G(10)+G(11)
 420 LET N=T2-T3
 430 LET T4=G(7)+G(8)+G(9)+G(10)+G(11)
 440 PRINT 'INPUT THE NORMALIZATION SCHEME
     USED IN MATBAL FOR THIS RUN'
 450 INPUT Z
 460 Z=Z/100
 480 FOR B=1 TO 6
 490 G(B) = G(B)+G(B)*(1-Z)*N/T1
 500 NEXT B
 510 FOR B=7 TO 14
 520 G(B) = G(B)+G(B)*Z*N/T4
 530 NEXT B
 750 LET T6=0
 755 FOR B=1 TO 7
 760 LET F(B)=G(B)/N(B)
 770 LET T6=T6+F(B)
 780 NEXT B
 790 PRINT ' '
 800 PRINT ' '
 810 PRINT ' '
 820 PRINT ' '
 830 PRINT ' '
 840 PRINT TAB(10);V$
 850 PRINT ' '
 860 PRINT USING 870, 'MOLES OF GAS IN
     (INCLUDING WATER)',M
 870 FORM POS10,C,POS60,PIC(ZZ.###), SKIP
 880 PRINT ' '
 890 PRINT USING 900, 'NORMALIZED MOLES OF GAS OUT
     (INCLUDING WATER)',T6
 900 FORM POS10,C,POS60,PIC(ZZ.###),SKIP
 920 LET N$(1)='GRAMS OF CO2'
 930 LET M$(1)='MOLES OF CO2'
 940 LET N$(2)='GRAMS OF C2H6'
 950 LET M$(2)='MOLES OF C2H6'
 960 LET N$(3)='GRAMS OF H2'
 970 LET M$(3)='MOLES OF H2'
 980 LET N$(4)='GRAMS OF CH4'
 990 LET M$(4)='MOLES OF CH4'
1000 LET N$(5)='GRAMS OF CO'
1010 LET M$(5)='MOLES OF CO'
1013 LET N$(6)='GRAMS OF H2S'
1017 LET M$(6)='MOLES OF H2S'
1020 LET N$(7)='GRAMS OF H2O'
1030 LET M$(7)='MOLES OF H2O'
1040 LET N$(8)='GRAMS OF ASH'
1050 LET N$(9)='GRAMS OF IOM'
1060 LET N$(10)='GRAMS OF SOL RESID'
1070 LET N$(11)='GRAMS OF DIST OILS'
1080 FOR B=1 TO 7
```

```
1090 PRINT ' '
1100 PRINT USING 1120, N$(B),G(B)
1120 FORM POS10,C,POS60,PIC(ZZZ.##), SKIP
1140 PRINT USING 1150, M$(B),F(B)
1150 FORM POS10,C,POS60,PIC(ZZZ.###), SKIP
1160 NEXT B
1170 FOR B=8 TO 11
1180 PRINT ' '
1190 PRINT USING 1200, N$(B),G(B)
1200 FORM POS10,C,POS60,PIC(ZZZ.##),SKIP
1210 NEXT B
1220 PRINT ' '
1230 PRINT ' '
1240 PRINT ' '
1250 END 'GRAMS AT TIME = T'
```

Appendix D

MATERIAL BALANCE SAMPLE CALCULATIONS

SAMPLE CALCULATIONS FOR RUN N174

Grams CO charged

$$K(1) = \frac{(\text{Moles CO in Feed})*(\text{Mole \% CO in Feed})}{\text{MW of CO}}$$

$$= \frac{(9.587) * (26.674)}{(100) * (28.011)} = 71.63$$

Grams H_2 charged

$$K(2) = \frac{(\text{moles } H_2 \text{ in feed gas})*(\text{mole \% } H_2 \text{ charged})}{\text{MW of } H_2}$$

$$= \frac{(9.587) * (73.326)}{(100) * (2.016)} = 14.172$$

Total grams feed gas charged

$$L1 = \text{grams CO} + \text{grams } H_2$$

$$= 71.63 + 14.172 = 85.80$$

Moles of product gas

$$L2 = \frac{(\text{BAROMETRIC PRESSURE})*(\text{FT}^3 \text{ PRODUCT GAS}) * 453.6}{(21.85) * (\text{temperature})}$$

$$= \frac{(29.205) * (7.576) * (453.6)}{(21.85) * (460 + 60)} = 8.833$$

Grams out, gases

General equation:

$$N(B) = \frac{\text{mole \% product gas (B)}}{(\text{total moles product gas})*(MW_B)}$$

$N(CO_2) = 95.87$

$N(C_2H_6) = 7.70$

$N(C_3H_8) = 0.0$

$N(H_2) = 10.52$

$N(CH_4) = 2.95$

$N(CO) = 27.44$

$N(C_2H_6) = .511$

$N(C_3H_8) = .045$

Grams water in alliquot

O1 = microdistillation sample, gms * (%water end product)

= (10.13) * (13.54) = 1.37%

Grams L.O. in alliquot

O2 = (gms L.O. in alliq)*(gms of condensed liquid from product gas stream)

- gms water in alliq

= (.076) * (1.653) - (.0137)

= .3574

Total grams oil (light + middle + heavy)

O3 = gms L.O. + gms M.O. + gms H.O.

= (.3474) + (5.615) + (.5436)

= 6.516

Total grams oil in alliquot (water, soluble residuum free)

O4 = (gms sample in microdistillation) -

(gms residue) - (gms water in alliq)

= (10.13) - (2.16) - (1.37)

= 6.60

Ratio of oil in end product to oil in alliquot

$$O5 = \frac{\text{(gms oil in alliq)*(gms liquid end product)}}{\text{(total gms oil)*(gms sample in microdistillation)}}$$

$$= \frac{(6.60) * (444.54)}{(6.52) * (10.13)}$$

$$= 44.44$$

Grams L.O. out

$$P(14) = \text{(gms L.O. in alliq)} * O5_{\text{above}}$$

$$+ \text{ gms L.O. in quench cold traps}$$

$$= (.36) * (44.44) + (.794)$$

$$= 16.68$$

Grams M.O. out

$$P(13) = \text{(gms M.O. from microdistillation)} * O5$$

$$= (5.61) * (44.44)$$

$$= 249.53$$

Grams H.O. out

$$P(12) = (\text{gms H.O. from microdistillation}) * 05$$

$$= (.54) * (44.44)$$

$$= 24.16$$

Grams water out

$$P(7) = \frac{(\text{gms liquid end product})*(\text{\% water in end product})}{(\text{gms water in quench cold traps})}$$

$$= \frac{(444.54) * (13.54)}{100 * (.206)}$$

$$= 60.40$$

Grams ash out

$$P(9) = (\text{\% ash in end product})*(\text{gms liquid end product})$$

$$= (0.0316) * (444.54)$$

$$= 14.05$$

Grams soluble residuum out

$$\begin{aligned} P(11) = {} & \text{(gms liquid end product)} \\ & * \text{(\% THF soluble end product)} \\ & - (\text{(gms water and oil in end product)} \\ & \quad - \text{(gms L.O. in quench traps)} \\ & \quad - \text{(gms water in quench cold traps)}) \end{aligned}$$

$$= (444.54)*(.91) - (7.0 - .79 - .21)$$

$$= 54.68$$

Grams IOM out

$$\begin{aligned} P(10) = {} & \text{(gms liquid end product)} \\ & * \text{(1 - \% THF solubles in end product)} \end{aligned}$$

$$= (444.54) * (1 - 0.91)$$

$$= 26.05$$

Grams L.O. in alliquot and cold trap

$$O8 = \frac{\text{(gms liquid left in system)* (gms solid residuum in microdistillation)}}{\text{(1 - \% THF solubles end product) * (gms in alliq)}}$$

$$\frac{(0) * (100) * (10.13)}{(1 - 0.91) * (10.03)}$$

$$= 0.0$$

Total grams non-distillables in liquid product

O9 = gms ash + gms IOM + gms solid residuum

= 14.05 + 26.05 + 54.68

= 94.78

Correction factor on IOM out

(adding fraction of IOM that goes over with L.O.)

$$P(10) = P(10) * \left(1 + \frac{\text{gms of L.O. in alliq and cold traps}}{\text{(gms in alliq)}}\right)$$

$$= 26.05 * ((0.0 * 26.05)/94.7811)$$

$$= 26.05$$

Correction factor on grams soluble residuum out

$$P(11) = P(11) * \left(1 + \frac{\text{gms L.O. in alliq and cold traps}}{\text{(gms non-distillables)}}\right)$$

$$= 54.68 + (0.00 * 54.68)/94.78)$$

$$= 54.68$$

Grams slurry total

R1 = gms coal + gms water + gms solvent

= 239.53 + 14.7 + 310.0

= 564.23

Grams of coal charged

$$R2 = \frac{(\text{gms coal in slurry})*(\text{gms slurry charged})}{(\text{total gms slurry})}$$

$$= \frac{(239.53) * (533.73)}{(564.23)}$$

$$= 226.58$$

Grams of ash charged

$$K(9) = (\text{gms coal charge}) * (\text{wt \% coal in charge})$$

$$= (226.58) * (0.081)$$

$$= 18.26$$

Grams of water charged

$$K(7) = (\text{gms coal charged}) * (\text{wt \% water in charge}) + \frac{(\text{gms water in slurry})*(\text{gms slurry charged})}{(\text{gms total slurry})}$$

$$= (226.58)*(0.263) + \frac{(14.7)*(533.73)}{(564.23)}$$

$$= 73.34$$

Grams MAF coal charged

$$K(10) = (\text{gms coal charged}) * (1 - \text{\% ash charged} - \text{\% water charged})$$

$$= (226.58) * (1 - 0.081 - 0.262)$$

$$= 148.89$$

Grams of solvent charged

$$K(15) = \frac{(\text{gms solvent in slurry})*(\text{gms slurry charged})}{(\text{total gms slurry})}$$

$$= \frac{(310) * (533.73)}{(564.23)}$$

$$= 293.24$$

Grams soluble residuum charged

$$K(11) = (\text{gms solvent charged}) *(\text{\% soluble residuum in solvent})$$

$$= (293.24) * (0.043)$$

$$= 12.61$$

Grams H.O. charged

$$K(12) = (\text{gms solvent charged})*(\text{\% H.O. in solvent})$$

$$= (293.24) * (0.076)$$

$$= 22.29$$

Grams L.O. charged

$$K(14) = (\text{gms solvent charged})*(\text{\% L.O. in slurry})$$

$$= (293.24) * (0.031)$$

$$= 9.09$$

Grams M.O. charged

K(13) = (gms solvent charged)

- (gms solid resid. + gms H.O. + gms L.O.)charged

= (293.24) - (12.61 + 22.29 + 9.09)

= 249.26

NORMALIZATION

Grams gas in process unaccounted for

R6 = (1 - (% wt loss assigned to liq))

* ((gms slurry charged + gms feed gas)

- (gms liq out + gms gas out))

= (1 - 1)* ...

= 0.0

Grams liquid in process unaccounted for

R8 = (% wt loss assigned to liquid)

* ((gms slurry charged + gms feed gas)

- (gms liq out + gms gas out))

= (1.00)*((533.73 + 85.80) - (445.45 + 145.03))

= 28.96

Normalized yield of gases per MAF coal charged

General equation:

$$S(B) = (\text{gms B out})*\left(1 + \frac{(\text{gms gas unaccouted for})}{(\text{gms gas out})}\right)$$

$$S(CO) = (27.44) + \frac{0 * (27.44)}{(145.03)}$$

$$= 27.44$$

$$S(H_2) = (10.52) + 0$$

$$= 10.52$$

$$S(CO_2) = 95.87 + 0$$

$$= 95.87$$

$$S(C1\text{-}C3) = 10.65 + 0$$

$$= 10.65$$

General equation:

$$T(B) = (\text{gms B out, corrected}) - (\text{gms B in})$$

$$T(CO) = 27.44 - 71.63 = -44.19$$

$$T(H_2) = 10.52 - 14.17 = -3.65$$

$$T(CO_2) = 95.87 - 0.0 = 95.87$$

$$T(C1\text{-}C3) = 10.65 - 0.0 = 10.65$$

Normalized yield of liquids per MAF coal charged

General equation:

$$S(B) = (\text{gms B out})*\left(1 + \frac{\text{gms liquid unaccounted for)}}{\text{(gms liquid out)}}\right.$$

$S(H_2O) = (60.40)*(1 + (28.96)/(445.54))$

$= 64.32$

$S(IOM) = (14.05)*(1 + (28.96)/(445.54))$

$= 14.97$

S(soluble residuum) = $(26.05)*(1 + (28.96)/(445.54))$

$= 27.74$

S(ash) = $(54.68)*(1 + (28.96)/(445.54))$

$= 58.24$

S(H.O.) = $(24.16)*(1 + (28.96)/(445.54))$

$= 25.73$

S(M.O.) = $(249.53)*(1 + (28.96)/445.54))$

$= 265.75$

S(L.O.) = $(16.68)*(1 + (28.96)/(445.54))$

$= 17.76$

General equation:

T(B) = (gms B out, corrected) - (gms B in)

$T(H_2O)$ = 60.40 - 73.33 = -9.02

T(soluble residuum) = 58.24 - 12.61 = 45.63

T(H.O.) = 25.73 - 22.29 = 3.44

T(M.O.) = 265.75 - 249.26 = 16.49

T(L.O.) = 17.76 - 9.09 = 8.67

T(IOM) = 27.74 + (14.96 - 18.26) = 24.44

NORMALIZATION OF YIELDS ON A 100 GRAM MAF COAL BASIS

Each corrected component (T-value) is divided by the total MAF coal charged to give normalized values on a 100 g MAF coal basis.

Material balance closure

$$V2 = \frac{\text{(gms liquid out) + (gms gas out)}}{\text{(gms liquid in) + (gms gas in)}}$$

= (590.57)/(619.53)

= 95.33%

<u>Grams distillate oils</u>

(based on 100 g MAF coal in)

V3 = gms L.O. + gms M.O. + gms H.O.

= 2.31 + 11.07 + 5.82

= 19.21

<u>Grams THF solubles</u>

(based on 100 g MAF coal in)

V4 = (gms soluble residuum) + (gms distillate oils)

= 30.65 + 19.21

= 49.86

<u>Grams gas product</u>

(based on 100 g MAF coal in)

V5 = gms CO + gms H_2 + gms CO_2 + gms C1-C3 + gms H_2S + NH_2

= -29.68 - 2.45 + 64.39 + 7.15 + .343 + 0.030

= 39.78

Percent conversion

$$V6 = 100 - \text{gms iom out start}$$

$$= 100 - 16.41$$

$$= 83.58\ \%$$

Solvent recovery

$$V7 = \frac{(\text{gms of oils out})}{(\text{gms of oils in})} * (100)$$

$$= (300.56)/(293.24) * (100)$$

$$= 102.50$$

Appendix E

ANALYTICAL PROCEDURES FOR GAS ANALYSIS

GPA PROJECT GC ANALYSIS SHEET

(HP F&M Scientific 700 Gas Chromatograph)

CALIBRATION

1. Porapak column valve in left-in-left postion.
2. Close all valves (manifold, tanks, etc.).
3. Open main valve on calibration tank, Calibration. Tank #1.
4. Turn on vacuum pump, then vacuum valve.
5. Turn on manifold evac. valve, allow vacuum for several minutes, flush line BRIEFLY with calibration gas.
6. Close manifold evac. valve, open black valve on calibration gas tank.
7. Close vacuum valve, shut off vacuum pump, open and close vacuum relief valve.
8. CAREFULLY open manifold evac. valve and charge calibration gas to equal legs on the manometer; close manometer evac. valve. (If gas is slightly overcharged, adjust back to equal legs on manometer by opening the vacuum valve.)
9. Close main valve on calibration tank.
10. Open manifold relief valve, close black valve on calibration tank and bleed gas out of the manifold; close all valves.
11. Repeat steps 1 thu 10 for Calibration Gas Tank #2.

GAS SAMPLE ANALYSIS

1. Start the Porapak column valve in left-in-left position.
2. Close all valves.
3. Turn on vacuum pump, open all valves.
4. Open sample valve, allow vacuum to stabilize.
5. Close sample valve, open sample bomb valve (be sure to observe manometer as a precaution).

6. Close vacuum valve, shut off vacuum pump, and open and close vacuum relief valve.
7. CAREFULLY open sample valve and charge gas sample to equal legs on the manometer. Adjust with vacuum valve if gas is overcharged. Close sample valve.
8. Close vacuum valve, turn off pump, and open and close relief valve.
9. Charge sample to GC with a right-out-right manuever on Porapak column.
10. Close sample bomb valve.
11. Check attenuations for previous day's analysis as a guide for current work.

Appendix F

ANALYTICAL PROCEDURE FOR LIQUID ANALYSIS

KARL FISCHER WATER DETERMINATION FOR SLURRY AND MISC.

HYDROCARBON SAMPLES

PLAM I-3-76T, II-14-76T, and V-A-3-77T

I. Startup procedure

a) Remove plugs from all desiccant tubes.

b) LIGHTLY grease all ground glass joints with vacuum grease.

c) Set magnetic stirrer motor to 1.5.

d) Add 45 ml of anhydrous methanol (AR) to titration cell.

If necessary, add enough KFR from buret to drive the conductance meter needle to the KFR end point zone. Note the meter reading in the KFR zone as this will be the reference number for the titration back to the end-point. Fill the 25 ml buret with KFR; if the sample is known to contain trace amounts of water, fill upper stage of buret also.

II. KFR Standardization[2]

a) Complete all steps outlined in step I.
Partially fill a disposable pipette with demineralized water and weigh to the nearest tenth of a milligram on the analytical balance.

b) With the cell stirrer and the cell plug removed, CAREFULLY add one drop of water to the cell, taking care not to touch the glass port of the cell; add sufficient water dropwise so that the conductance meter needle deflects into the water zone (yellow).

c) Quickly stopper the cell and reweigh the disposable pipet; record the sample weight in the sample book. (Use no more than approx. 25 mg of H_2O sample).

d) Titrate the KFR SLOWLY into the cell until the conductance meter needle remains stable in that zone at the meter reading determined in step I above. Normally, a KFR endpoint is considered to be valid if the conductance meter needle remains in one position for at

---

[2] KFR titer should be determined once every 24 hours.

least 60 seconds.

e) Calculate KFR titer (mg/ml) by dividing the weight of the water sample used by the volume (ml) of the KFR titrated.

f) Repeat steps a thru f and average the results; KFR standardization results should produce titers of approximately 5.0 to 5.5 mg/ml.

III. Sample Analysis

a) FOR SOLID SAMPLES, MIX WELL AND CHARGE TO DISPOSABLE pipet with the tip broken off. Weigh the pipet containing the sample to four places on the analytical balance. For liquid samples shake well and charge sample to disposable pipet with the tip intact; weigh the pipet containing the sample to four places on the analytical balance. Note: be sure to wipe exterior of disposable pipet before the initial weighing.

b) CAREFULLY ADD THE SAMPLE TO THE TITRATION CELL IN sufficient quantity to deflect the conductance meter needle and cause the KFR to turn yellow.

c) STOPPER THE TIRATION CELL, WEIGH BACK THE DISPOSABLE pipet, and slowly titrate with KFR to KFR endpoint.

d) CALCULATE WEIGHT % WATER IN THE SAMPLE AS FOLLOWS:

$$\text{Wt\% water} = \frac{\text{KFR titer x ml KFR titrated x 100}}{\text{Sample wt, mg}}$$

Determination of Coal Ash

PLAM I-4-76

I. Scope:

This method describes a procedure for the determination of ash in raw coal.

II. Outline:

A sample of pulverized coal is weighed into a filtered procelain crucible and placed in a muffle furnace; the sample is heated slowly from room temperature to 750°C and maintained at 750°C for four hours; the sample is then removed from the oven, allowed to cool to room temperature and weighed. The resulting residue in the crucible is calculated as wt.% ash in the sample.

III. Directions:

a) Apparatus

1. Muffle furnace, thermolyne, type 1500, temperature range from RT to 1200°C
2. Crucible, Coors, Size 34-S
3. Tongs, Stainless stell, zirconium tip.
4. Desiccator

b) Reagents

1. None

c) Sample

1. The coal sample must be pulverized either mechanically or by using mortar and pestal.

d) Procedure

1. Weigh a 2 g. sample into a fired porcelain crucible, using the analytical balance.
2. Place crucible containing the sample into the muffle furnace and shut muffle furnace door.
3. Turn muffle furnace rheostat setting to 30 and heat sample from room temperature to 400°C in a period of 2-1/4 hrs. (approx. 3°C/min).

4. Once the sample has attained a temperature of 400°C, turn furnace rheostat to high and heat sample to 750 °C ashing temperature.

5. At a sample temperature of approximately 740°C, dial rheostat to 35 (or recommended setting on the muffle furnace being used) and maintain a sample temperature of 750°C for four hours.

6. After 4 hours at 750°C, turn off furnace, remove crucible containing sample residue, and place on porcelain tile in front of furnace. Do not place crucible directly into desiccator glass. After a 1-2 minute cooling period on the porcelain tile, the crucible may be placed in the desiccator for cooling. Allow at least 25 minutes for cooling before attempting to reweigh crucible on analytical balance.

7. Weigh cooled crucible and sample on analytical balance.

e) Calculations

1. Calculate wt.% ash in the sample as follows:

$$\text{Wt.\% ash} = \frac{\text{Sample residue in crucible, x 100 g.}}{\text{Orignial sample weight, g.}}$$

example calc.:

Before ashing

```
   22.0000 (xble + Sx)
  -19.50000 (xble)
  ---------
  2.50000 g
```

After ashing

```
   19.6100 (xble + Sx residue)
  -19.50000 (xble)
   --------
   0.1100 g residue
```

$$\text{Wt.\% ash} = \frac{0.1100 \times 100}{2.5000}$$

$$= 4.40$$

IV. Discussion

a) If ashing temperature exceeds 750^{o}C., the test must be repeated.

b) Make sure the procelain crucible is clean and fired (at 800^{o}C) prior to use.

c) Allowable precision error: 2% difference.

d) Smoke fumes from pyrolyzed coal are toxic; be sure fume hood fan is on before heating sample.

This method is a modified version of ASTM D3174.

Test for THF Solubility

The solubility of the product sample is a factor used in determining the conversion of the lignite during the liquefaction process. We define the solubility as being able to pass through a 0.5 micron filter.

Procedure:

1. Use millipore fluoropore type FH filters with 0.5 micrpark pore size and type AP Mitrex prefilters.
2. Dry the refilters at least 1 hour at 105°C on a watch glass prior to use.
3. Remove the prefilters and watch glass from the oven and place in a desiccator to cool to room temperature. Add a 0,5 micron filter and weigh.
4. Assemble filters in the filter holder. The 0.5 micron filter goes on the support screen and the prefilter on top of that. Make sure the assembly is tightened snuggly.
5. Weigh out two grams of the sample material in a 100 ml beaker. Dissolve the sample in THF and pour into filter holder. A quatitative transfer of the solution from the beaker to the filter holder assembly is necessary.
6. Force the solution through the filters with nitrogen pressure. (Maximum flow rate for the filters is 450 ml/min.) Rinse the residue with THF until the washing are colorless.
7. When all THF has been forced through the filters, remove them from the holders and transfer the filters and insoluble residue quantitatively to the watch glass. Dry at 105°C for 1/2 hours, cool in desiccator and reweigh.

Calculations:

$$\%\text{THF insoluble} = \frac{\text{wt. res. + filter - wt. filter x 100}}{\text{wt. sample}}$$

Note:

- If filter holder leaks, the test must be repeated.
- Do not dry the 0.5 micron filters prior to use.
- Do test in duplicate.

STANDARD VACUUM DISTILLATION TEST (ASTM D-1160)

using standard apparatus (SA)

PLAM II-10-77T

1. SCOPE

This method covers the determination at reduce pressures of the boiling temperature ranges of petroleum products which decompose when distilled at atmospheric pressure.

2. OUTLINE

The sample is distilled at 5 mm. Hg absolute under conditions which provide approximately one theoetical plate fractionation. Data are obtained from which a distillation curve relating volume distilled and boiling point at the controlled pressure can be prepared. Weight % (sample weight basis) of fractions boiling over the range IBP to 121°C, 121°C to 260°C and 260°C + are determined using specific gravity/ volume relationships.

3. Directions

a) Apparatus

- i) ASTM D-1160 distillation equipment kit
- ii) Specific gravity hydrometers
- iii) Potentiometer
- iv) Kovar tip thermocouple
- v) Dewar flask
- vi) Manostat (optional)
- vii) Cartesian Diver Manometer
- viii) McLeod Gauge
- ix) Thermometer
- x) Variac

b) Reagents

ASTM Cetane Reference Fuel

c) Sample

The liquid sample must be homogenous and <u>free of visible aqueous phase</u>. Only liquid samples <u>from PDU SxPTs 12 or 13</u>, 14, and 34 or 39 are to be distilled using this procedure.

d) Calibration

i) Calibrate potentiometer; standardize against battery and reference junction (see 4 X 6 procedure card for potentiometer standardization).

ii) Assemble distillation apparatus empty, turn vacuum pump on, then off to check for leaks; turn on vacuum pump again, adjust pressure to 5 Tprr using the McLeod gauge. Set the Cartesian Diver manometer to 5 Torr, using the McLeod gauge as teh referenc.

iii) Record the atmospheric pressure and abient temperature, before and after the distillation test.

e) Procedure

i) Determine the specific gravity @ RT of the sample by hydrometer, using a 50 ml graduate.

ii) From the specific gravity of the sample at ambient temperature (RT), determine the weight, to the nearest 0.1 gram equivalent to 100 ml of the sample; weigh this quantity of sample into a 250 ml flat bottom distillation flask. Add a 1 1/2 inch stir bar to the flask.

iii) Mount distillation flask with the sample on the distillation apparatus, LIGHTLY GREASING the ground glass joints. Install 250 ml heating mantle on lower half of flask and an <u>unconnected</u> upper half heating mantle jacket on upper half of lask.

iv) Insert Kovar tip thermocouple in distillation column head, and turn on vacuum pump, adjust pressure to 5 Torr. If sample foams, allow foaming to continue until no further foaming is evident before applying heat.

v) Set variac at 65, activate timer, and abserve the initial boiling point (IBP) temperature - the IBP temperature is taken when the first drop of distillate is visible at the top of the drip chain in the receiver.

vi) Adjust the heating rate such that a distillate collection rate of 4 to 5 ml/min is achieved.

vii) Record distillate volume vs. temperature (potentiometer rdg., mv) at 5%, 10%, 20%, 30%, 40%, 50%, 60%, 70%, 80%, 90%, and 95% by volume of sample. IMPORTANT: Record volume of distillate received corresponding to IBP to 121°C, and 121°C to 260°C; these data will be used later on to compute the Wt% of each fraction in the sample.

viii) Continue the distillation until 260°C is reached or abrupt loss in temperature occurs.

ix) Place plastic shield in front of apparatus, turn off vacuum pump and heating mantle and allow to cool to room temperature. DO NOT ATTEMPT TO ADMIT AIR into the distillation apparatus until the system is at approximately 100°F of less.

x) Weigh back 250 ml distillation flask, and primary receiver.

xi) Enter all volume % vs. temperature data on worksheet and compute the Wt% recovery.

xii) Clean apparatus, using acetone and gentle heating at atmospheric pressure.

f) CALCULATIONS

Calculate the wt% of IBP to 121°C, 121°C to 260°c, and 260°C.+ residue fraction as follows:

$$\text{Wt\% IBP to } 121^{\circ}\text{C fraction (corr to 100\% rec) (light oil)} = \frac{(10{,}000)(\text{vol, ml})(1.003)}{(\%\ \text{rec})(\text{sample wt, gm})}$$

$$\text{wt\% } 121^{o}\text{c to } 260^{o}\text{C fraction (corr to 100\% rec)} = \frac{(10{,}000)\,(\text{vol, ml})\,(1.003)}{(\%\text{ rec})(\text{sample wt, gm})}$$

wt% residue frac
(corr to 100% rec) = 100 - Sum above Wt%'s

Notes:

1. Calibartion of distillation equipment with Astm Cetane reference fuel is being waived until the reference fuel source can be located.

2. All distillation data is to be logged on worksheet forms and stored in the appropriate ring binder. As with other testwork, the raw data is to be placed in the PDU data ring binders for use by the project engineering staff.

3. It is extremely important that the Cartesian Diver manaometer be calibrated against the McLeod gauge prior to <u>each</u> distillation test.

Appendix G

TIME SAMPLE CALCULATIONS

SAMPLE CALCULATIONS FOR RUN N175

Calculated Response Factors for Individual Gases

$$Y(1) = \frac{\text{(Total moles of gases at time = 60 minutes)}}{\text{(Area under G. C. curve of 60 minute sample)}}$$

$$= \frac{1.107}{1.795}$$

$$= 0.617$$

Corrected Areas for Individual Gases at Time = 1 Minute

General Equation:

C(G3,G4) = (Response Factor) * (Old Area under Curve)

$C(H_2) = 0.503$
$C(CO) = 2.990$
$C(CH_4) = 0.286$
$C(C_2H_6) = 0.000$
$C(H_2O) = 0.000$

Total Corrected Area at Time = 1 Minute

$$D(B) = 0.503 + 2.990 + 0.286 + 1.776 + 0.000 + 0.000 = 5.555$$

Mole Percent of Individual Gases at Time = 1 Minute

General Equation;

$$E(G7,G8) = \frac{\text{Area of Curve at Time = 1}}{\text{Total Area of Curve at Time = 1}} \times 100$$

$E(H_2) = 9.061$
$E(CO) = 53.823$
$E(CH_4) = 5.146$
$E(CO_2) = 31.970$

$E(C_2H_6)$ = 0.000
$E(H_2O)$ = 0.000

Grams of Gas at Time = 1 Minute

General Equation

$$R(C,B) = \frac{\text{Mole Percent of Gas}}{\text{Total Moles}} \times \text{Mole. Wgt.}$$

$R(H_2)$ = 2.765
$R(CO)$ = 228.211
$R(CH_4)$ = 12.496
$R(CO_2)$ = 212.980
$R(C_2H_6)$ = 0.000
$R(H_2O)$ = 0.000

Correction Factor for Gases so Grams at Time = 60 Minutes is equal to Grams at Termination

General Equation:

$$R(C,B) = \frac{\text{Grams At Time} = 1}{\dfrac{\text{Grams At Time} = 60}{\text{Grams At Time} = \text{Termination}}}$$

$R(H_2)$ = 2.250
$R(CO)$ = 285.750
$R(CH_4)$ = 10.140
$R(CO_2)$ = 173.370
$R(C_2H_6)$ = 0.000
$R(H_2O)$ = 0.000

Grams of Ash at Time = 1 Minute

$$R(9,2) = \text{Ash In} - \frac{(\text{Ash In} - \text{Ash Out}) * 1 \text{ Minute}}{60 \text{ Minutes}}$$

$$= 18.002$$

Grams of IOM at Time = 1 Minute

$$R(11,2) = \frac{\begin{matrix}\text{Grams} \\ \text{of Ash}\end{matrix} * \begin{matrix}\text{Microdistillation} \\ \text{Residue}\end{matrix} * \begin{matrix}\text{\% THF} \\ \text{Soluble}\end{matrix}}{(\text{Percent Ash})} - \begin{matrix}\text{Grams} \\ \text{of Ash}\end{matrix}$$

$$= \frac{(18.00) * (55.23) * (85)}{(7.79) * (100)} - 18.00$$

$$= 90.47$$

Grams of Soluble Residuum at Time = 1 Minute

$$R(12,2) = \frac{(\text{Grams of Ash})*(\text{Micro. Residue})*(1-\text{\%THF Soluble})}{(\text{Percent Ash})}$$

$$= \frac{(18.00) * (55.23) * (100-85)}{(7.79) * (100)}$$

$$= 19.14$$

Grams of Distillable Liquids at Time = 1 Minute

$$R(13,2) = \frac{(\text{Grams of Ash}) * (1 -\text{\% Residue})}{(\text{Percent Ash})}$$

$$= \frac{(18.00) * (100 - 85)}{(7.76) * (100)}$$

$$= 103.45$$

Correction Factor for Liquid Sample so Sample at Time = 60 Minutes equals Sample at Time = Termination

IOM

$$= \frac{(90.47) * (27.96)}{(37.07)}$$

$$= 68.23$$

Soluble Residuum

$$= \frac{(19.14) * (59.17)}{(54.68)}$$

$$= 20.71$$

Conversion at Time = 1 Minute

$$R(16,2) = 100 - \text{Insoluble Organic Matter}$$

$$= 100 - 46.82$$

$$= 53.18$$

Yields of Each Product at Time = 1 Minute

$$R(B,2) = \frac{\text{Grams In} - \text{Grams Out}}{(\text{MAF Charge})} \times 100$$

$R(H_2)$ = 1.54
$R(CO)$ = -85.76
$R(CH_4)$ = 6.96
$R(CO_2)$ = 118.95
$R(C_2H_6)$ = 0.00
$R(H_2O)$ = 0.00
R(Slurry) = -147.62
R(MAF) = 46.82
R(Soluble Residuum) = 5.61
R(Distillable Oils (Liq.)) = -126.59
R(Conversion) = 53.18

Appendix H

MATERIAL BALANCE AND TIME SAMPLE CALCULATION RESULTS

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RUN #                        N174          (100.0% LOSS ASSIGNED TO LIQUID)
COAL                         ZAP
SOLVENT                      A04-A04
ADDITIVE                     NONE
DATE                         08-24-83
CONDITIONS
RES TIME (MIN)               20-60
TIME SAMPLE ?                YES
AVG TEMP (C)                 370-439
MAX TEMP (C)                 371-440
MAX PRESS, PSIG              4000-2275
% H2 IN FEED GAS              73.33
                                                NET YIELDS   NET YIELDS
                                                  GMS OF      WT % MAF
COMPONENT              GRAMS IN    GRAMS OUT    MAF COAL IN   COAL IN
CO                       71.63       27.44      - 44.19      - 29.68
H2                       14.17       10.52      -  3.65      -  2.45
CO2                        .00       95.87        95.87        64.39
C1-C3                      .00       10.65        10.65         7.15
H2S                        .00         .51          .51          .34
NH3                        .00         .05          .05          .03

H20                      73.34       60.40      -  9.02      -  6.06
ADDITIVE                   .00         .00          .00          .00
ASH                      18.26       14.05          .00          .00
MAF IN-IOM OUT          148.89       26.05        24.44        16.42
SOLUBLE RESID            12.61       54.68        45.63        30.65
HO                       22.29       24.16         3.44         2.31
MO                      249.26      249.53        16.49        11.07
LO                        9.09       16.68         8.67         5.82

TOTAL                   619.53      590.57       148.89       100.00

MISC. NET YIELDS, WT % MAF COAL IN

    DISTILLABLE OILS                                           19.21
    SOLUBLE RESIDUUM                                           30.65
    TOTAL THF SOLUBLES                                         49.86
    UNCONVERTED COAL                                           16.42
    WATER                                                    -  6.06
    GAS                                                        39.78
    % CONVERSION                                               83.58
    SOLVENT RECOVERY, WT %                                    102.50
    MAT BAL CLOSURE, WT%                                       95.33
.........................................................................
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..............................................................................

RUN #                      N175          ( 40.0% LOSS ASSIGNED TO LIQUID)
COAL                       ZAP
SOLVENT                    A04-A04
ADDITIVE                   NONE
DATE                       08-31-83
CONDITIONS
RES TIME (MIN)             20-60
TIME SAMPLE ?              YES
AVG TEMP (C)               365-440
MAX TEMP (C)               367-441
MAX PRESS, PSIG            4000-2385
% H2 IN FEED GAS              .00
                                                 NET YIELDS  NET YIELDS
                                                   GMS OF     WT % MAF
COMPONENT               GRAMS IN    GRAMS OUT    MAF COAL IN  COAL IN
CO                       310.75      206.53      - 82.12     - 56.35
H2                          .00        2.19         2.42        1.66
CO2                         .00      144.12       159.54      109.46
C1-C3                       .00        6.10         6.75        4.63
H2S                         .00        1.56         1.73        1.18
NH3                         .00         .06          .07         .05

H2O                       72.89       42.20      - 28.07     - 19.26
ADDITIVE                    .00         .00          .00         .00
ASH                       18.05       13.31          .00         .00
MAF IN-IOM OUT           145.75       32.52        30.62       21.01
SOLUBLE RESID             12.53       47.97        38.41       26.36
HO                        22.15         .00      - 22.15     - 15.20
MO                       247.78      269.63        38.59       26.48
LO                         9.04        8.47      -   .04     -   .03

TOTAL                    838.95      774.66       145.75      100.00

MISC. NET YIELDS, WT % MAF COAL IN

    DISTILLABLE OILS                                          11.25
    SOLUBLE RESIDUUM                                          26.36
    TOTAL THF SOLUBLES                                        37.61
    UNCONVERTED COAL                                          21.01
    WATER                                                   - 19.26
    GAS                                                       60.64
    % CONVERSION                                              78.99
    SOLVENT RECOVERY, WT %                                   101.33
    MAT BAL CLOSURE, WT%                                      92.34
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RUN #                    N176         ( 40.0% LOSS ASSIGNED TO LIQUID)
COAL                     ZAP
SOLVENT                  A04-A04
ADDITIVE                 NONE
DATE                     09-07-83
CONDITIONS
RES TIME (MIN)           20-60
TIME SAMPLE ?            YES
AVG TEMP (C)             365-440
MAX TEMP (C)             369-441
MAX PRESS, PSIG          3910-2365
% H2 IN FEED GAS          57.09
                                                NET YIELDS  NET YIELDS
                                                 GMS OF     WT % MAF
COMPONENT              GRAMS IN    GRAMS OUT   MAF COAL IN  COAL IN
CO                      128.29       59.35     - 55.77     - 37.71
H2                       12.29       10.01     -   .05     -   .04
CO2                        .00       83.76      102.35       69.22
C1-C3                      .00        7.75        9.47        6.40
H2S                        .00        1.18        1.44         .98
NH3                        .00         .13         .16         .11

H20                      73.96       61.29     -  9.43     -  6.38
ADDITIVE                   .00         .00         .00         .00
ASH                      18.31       14.55         .00         .00
MAF IN-IOM OUT          147.88       31.15       29.80       20.15
SOLUBLE RESID            12.72       53.53       43.64       29.51
HO                       22.48         .00     - 22.48     - 15.20
MO                      250.51      275.21       39.23       26.53
LO                       10.06       18.60        9.52        6.44

TOTAL                   676.49      616.50      147.88      100.00

MISC. NET YIELDS, WT % MAF COAL IN

    DISTILLABLE OILS                                         17.77
    SOLUBLE RESIDUUM                                         29.51
    TOTAL THF SOLUBLES                                       47.28
    UNCONVERTED COAL                                         20.15
    WATER                                                  -  6.38
    GAS                                                      38.95
    % CONVERSION                                             79.85
    SOLVENT RECOVERY, WT %                                  101.36
    MAT BAL CLOSURE, WT%                                     91.13
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RUN #                         N177          (100.0% LOSS ASSIGNED TO LIQUID)
COAL                          ZAP
SOLVENT                       A04-A04
ADDITIVE                      NONE
DATE                          09-08-83
CONDITIONS
RES TIME (MIN)                20-60
TIME SAMPLE ?                 YES
AVG TEMP (C)                  366-440
MAX TEMP (C)                  368-444
MAX PRESS, PSIG               4090-2555
% H2 IN FEED GAS               18.29
                                                NET YIELDS  NET YIELDS
                                                 GMS OF      WT % MAF
COMPONENT               GRAMS IN    GRAMS OUT   MAF COAL IN  COAL IN
CO                       223.82      168.52     - 55.30     - 37.54
H2                         3.61        4.97       1.37         .93
CO2                         .00      118.33     118.33       80.33
C1-C3                       .00        7.34       7.34        4.99
H2S                         .00         .71        .71         .48
NH3                         .00         .10        .10         .07

H2O                       73.63       50.32     - 19.43     - 13.19
ADDITIVE                    .00         .00        .00         .00
ASH                       18.25       13.73        .00         .00
MAF IN-IOM OUT           147.30       28.44      27.16       18.44
SOLUBLE RESID             12.67       39.23      29.58       20.08
HO                        22.39        7.19     - 14.65     -  9.94
MO                       250.41      277.59      48.56       32.97
LO                         9.13       11.76       3.53        2.40

TOTAL                    761.21      728.23     147.30      100.00

MISC. NET YIELDS, WT % MAF COAL IN

    DISTILLABLE OILS                                          25.42
    SOLUBLE RESIDUUM                                          20.08
    TOTAL THF SOLUBLES                                        45.50
    UNCONVERTED COAL                                          18.44
    WATER                                                   - 13.19
    GAS                                                       49.25
    % CONVERSION                                              81.56
    SOLVENT RECOVERY, WT %                                   107.21
    MAT BAL CLOSURE, WT%                                      95.67
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RUN #                         N178            ( 80.0% LOSS ASSIGNED TO LIQUID)
COAL                          ZAP
SOLVENT                       A04-A04
ADDITIVE                      NONE
DATE                          09-12-83
CONDITIONS
RES TIME (MIN)                20-60
TIME SAMPLE ?                 YES
AVG TEMP (C)                  361-427
MAX TEMP (C)                  367-429
MAX PRESS, PSIG               3940-2490
% H2 IN FEED GAS               76.09
                                                NET YIELDS   NET YIELDS
                                                  GMS OF      WT % MAF
COMPONENT               GRAMS IN    GRAMS OUT   MAF COAL IN   COAL IN
CO                         71.04       27.30    - 42.41       - 28.58
H2                         16.27       15.12    -   .42       -   .28
CO2                          .00       88.95      93.27         62.87
C1-C3                        .00        3.87       4.05          2.73
H2S                          .00        1.02       1.07           .72
NH3                          .00         .09        .10           .06

H2O                        74.15       63.99    -  6.44       -  4.34
ADDITIVE                     .00         .00        .00           .00
ASH                        19.66       14.84        .00           .00
MAF IN-IOM OUT            148.37       24.72      22.21         14.97
SOLUBLE RESID              12.76       67.13      58.26         39.27
HO                         22.55       26.80       5.80          3.91
MO                        250.14      251.14      15.58         10.50
LO                         11.28        8.09    -  2.71       -  1.83

TOTAL                     626.21      593.06     148.37        100.00

MISC. NET YIELDS, WT % MAF COAL IN

    DISTILLABLE OILS                                            12.58
    SOLUBLE RESIDUUM                                            39.27
    TOTAL THF SOLUBLES                                          51.85
    UNCONVERTED COAL                                            14.97
    WATER                                                     -  4.34
    GAS                                                         37.52
    % CONVERSION                                                85.03
    SOLVENT RECOVERY, WT %                                     101.99
    MAT BAL CLOSURE, WT%                                        94.71
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RUN #                        N179          (100.0% LOSS ASSIGNED TO LIQUID)
COAL                         ZAP
SOLVENT                      A04-A04
ADDITIVE                     NONE
DATE                         09-14-83
CONDITIONS
RES TIME (MIN)               20-60
TIME SAMPLE ?                YES
AVG TEMP (C)                 358-441
MAX TEMP (C)                 360-444
MAX PRESS, PSIG              3970-2525
% H2 IN FEED GAS              38.05
                                                   NET YIELDS   NET YIELDS
                                                     GMS OF      WT % MAF
COMPONENT               GRAMS IN     GRAMS OUT    MAF COAL IN    COAL IN
CO                       186.88       128.49       - 58.39       - 38.33
H2                         8.26         6.61       -  1.66       -  1.09
CO2                         .00       126.55        126.55         83.09
C1-C3                       .00         7.66          7.66          5.03
H2S                         .00         1.30          1.30           .85
NH3                         .00          .00           .00           .00

H20                       76.07        46.14       - 23.67       - 15.54
ADDITIVE                    .00          .00           .00           .00
ASH                       18.89        12.96           .00           .00
MAF IN-IOM OUT           152.32        35.08         35.67         23.42
SOLUBLE RESID             13.10        43.91         36.77         24.14
HO                        23.15          .00       - 23.15       - 15.20
MO                       258.94       268.96         46.53         30.55
LO                         9.44        12.46          4.70          3.09

TOTAL                    747.06       690.12        152.32        100.00

MISC. NET YIELDS, WT % MAF COAL IN

    DISTILLABLE OILS                                                18.43
    SOLUBLE RESIDUUM                                                24.14
    TOTAL THF SOLUBLES                                              42.57
    UNCONVERTED COAL                                                23.42
    WATER                                                         - 15.54
    GAS                                                             49.55
    % CONVERSION                                                    76.58
    SOLVENT RECOVERY, WT %                                         103.37
    MAT BAL CLOSURE, WT%                                            92.38
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RUN #                      N180          (100.0% LOSS ASSIGNED TO LIQUID)
COAL                       ZAP
SOLVENT                    A04-A04
ADDITIVE                   NONE
DATE                       09-15-83
CONDITIONS
RES TIME (MIN)             20-60
TIME SAMPLE ?              YES
AVG TEMP (C)               359-440
MAX TEMP (C)               360-443
MAX PRESS, PSIG            4365-2750
% H2 IN FEED GAS           100.00
                                                  NET YIELDS  NET YIELDS
                                                    GMS OF     WT % MAF
COMPONENT              GRAMS IN   GRAMS OUT     MAF COAL IN   COAL IN
CO                          .00      12.00         12.00        8.21
H2                        21.29      16.45       -  4.84     -  3.31
CO2                         .00      59.81         59.81       40.89
C1-C3                       .00       9.80          9.80        6.70
H2S                         .00       1.60          1.60        1.09
NH3                         .00        .14           .14         .10

H20                       73.09      73.19          6.91        4.73
ADDITIVE                    .00        .00           .00         .00
ASH                       18.10      12.47           .00         .00
MAF IN-IOM OUT           146.25      45.93         45.74       31.28
SOLUBLE RESID             12.58      35.87         26.63       18.21
HO                        22.23        .00       - 22.23     - 15.20
MO                       248.67     231.01          3.86        2.64
LO                         9.07      14.54          6.83        4.67

TOTAL                    551.29     512.82        146.25      100.00

MISC. NET YIELDS, WT % MAF COAL IN

    DISTILLABLE OILS                                          -  7.89
    SOLUBLE RESIDUUM                                            18.21
    TOTAL THF SOLUBLES                                          10.31
    UNCONVERTED COAL                                            31.28
    WATER                                                        4.73
    GAS                                                         53.68
    % CONVERSION                                                68.72
    SOLVENT RECOVERY, WT %                                      89.42
    MAT BAL CLOSURE, WT%                                        93.02
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RUN #	N175
COAL	ZAP
SOLVENT	A04-A04
ADDITIVE	NONE
DATE	8-31-83
CONDITIONS	
RES TIME (MIN)	20-60
AVG TEMP (C)	365-440
MAX TEMP (C)	367-441
MAX PRESS, PSIG	4000-2385
% H2 IN FEED GAS	0-0

		NET YIELDS (WT% MAF)							
COMPONENTS	GRAMS @ T=00	T=01	T=03	T=06	T=10	T=20	T=40	T=60	GRAMS @ TERMINATION
H2	.00	1.54	.36	.44	.49	.48	.66	1.53	2.23
CO	310.75	- 85.76	-139.63	-137.57	-144.54	-152.42	-200.53	- 68.54	210.86
CH4	.00	6.96	1.65	1.81	2.29	2.35	.83	2.14	3.12
CO2	.00	118.95	243.93	238.34	246.54	258.25	332.55	100.95	147.14
C2H6	.00	.....	.....	.....	.....	.....	.....	.....	1.96
H2O	72.89	.....	.....	.....	.....	.....	.....	.....	48.10
SLURRY	446.25	-159.88	-159.69	-161.37	-163.72	-162.65	-155.43	-147.45	408.76
MAF	145.75	39.45	43.41	42.28	39.98	40.25	34.71	25.43	37.07
SOL RESID	12.53	3.38	- .47	1.68	3.51	3.58	12.30	28.92	54.68
DISTILLATES (LIQ)	287.97	-132.08	-133.74	-135.96	-136.82	-136.20	-129.79	-125.15	317.01
DISTILLATES (VAP)		.....	.....	.....	.....	.....	.....	.....	
DISTILLABLES		.....	.....	.....	.....	.....	.....	.....	
CONVERSION		60.55	56.59	57.72	60.02	59.75	65.29	74.57	

Z1= 1
Z9= 2

RUN #	N176
COAL	ZAP
SOLVENT	A04
ADDITIVE	NONE
DATE	9-7-83
CONDITIONS	
RES TIME (MIN)	2060
AVG TEMP (C)	365-440
MAX TEMP (C)	369-441
MAX PRESS, PSIG	3910-2365
% H2 IN FEED GAS	0-75

COMPONENTS	GRAMS @ T=00	NET YIELDS (WT% MAF) T=01	T=03	T=06	T=10	T=20	T=40	T=60	GRAMS @ TERMINATION
H2	12.29	.32	6.00	- 1.34	.88	- 1.06	1.82	- 2.49	8.61
CO	128.29	- 43.22	- 11.93	- 48.27	- 36.94	- 53.29	- 25.93	- 52.21	51.08
CH4	.00	.43	.76	3.38	1.45	1.31	.39	2.20	3.26
CO2	.00	19.30	.....	41.01	58.11	45.21	65.62	50.26	74.33
C2H6	.00	.....	.....	.....	.....	.....	.....	.....	2.09
H2O	73.96	14.48	.....	17.69	- 16.47	11.46	.....	- 1.11	72.32
SLURRY	443.65	-130.81	-165.52	-180.52	-158.04	-138.11	-135.22	-139.84	446.61
MAF	147.88	48.16	38.30	38.81	36.59	28.75	29.73	24.86	36.76
SOL RESID	12.72	5.07	4.09	1.24	9.14	25.65	31.87	34.11	63.16
DISTILLATES (LIQ)	283.05	- 98.13	-121.36	-134.39	-116.53	-102.85	-106.54	-107.91	346.69
DISTILLATES (VAP)		153.63	.....	181.90	163.80	144.87	.....	152.33	
DISTILLABLES		55.50	.....	47.51	47.27	42.02	.....	44.41	
CONVERSION		51.84	61.70	61.19	63.41	71.25	70.27	75.14	

Z1= 1
Z9= 2

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     RUN #                 N177
     COAL                  ZAP
     SOLVENT               A04
     ADDITIVE              NONE
     DATE                  9-8-83
     CONDITIONS
     RES TIME (MIN)        20-60
     AVG TEMP (C)         366-440
     MAX TEMP (C)          368-444
     MAX PRESS, PSIG       4090-2555
     % H2 IN FEED GAS      0-25

                                                          NET YIELDS (WT% MAF)
                                      .................................................................................
                        GRAMS @                                                                                                  GRAMS @
 COMPONENTS              T=00         T=01          T=03          T=06          T=10          T=20          T=40          T=60       TERMINATION

 H2                      3.61         1.05           .99           .89           .85           .92         .....           .49          4.33
 CO                    223.82      -  7.39       - 21.66       - 24.52       - 30.93       - 20.21         .....       - 52.32        146.75
 CH4                      .00          .28           .42          1.04          1.70           .83         .....          2.90          4.27
 CO2                      .00         6.39         29.41         33.70         41.95         24.05         .....         69.96        103.05
 C2H6                     .00        .....         .....         .....         .....         .....         .....         .....          1.06
 H2O                    73.63        .....         .....         .....         .....         .....         .....         .....         58.88
 SLURRY                441.90      -146.92       -154.56       -160.48       -163.91       -160.65       -161.97       -167.01        426.19
 MAF                   147.30        44.12         40.28         45.34         34.75         24.17         29.57         22.59         33.28
 SOL RESID              12.67        13.01         16.82          6.88         15.24         26.28         20.51         22.57         45.91
 DISTILLATES (LIQ)     281.93      -123.67       -129.28       -134.08       -130.25       -121.82       -125.68       -123.28        347.00
 DISTILLATES (VAP)                   .....         .....         .....         .....         .....         .....         .....
 DISTILLABLES                        .....         .....         .....         .....         .....         .....         .....

 CONVERSION                          55.88         59.72         54.66         65.25         75.83         70.43         77.41

     Z1= 1
     Z9= 2
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RUN #	N178
COAL	ZAP
SOLVENT	A04
ADDITIVE	NONE
DATE	9-12-83
CONDITIONS	
RES TIME (MIN)	20-60
AVG TEMP (C)	361-427
MAX TEMP (C)	367-429
MAX PRESS, PSIG	3940-2490
% H2 IN FEED GAS	0-100

		NET YIELDS (WT% MAF)							
COMPONENTS	GRAMS @ T=00	T=01	T=03	T=06	T=10	T=20	T=40	T=60	GRAMS @ TERMINATION
H2	16.27	- .37	- 1.27	- 4.87	.64	- 1.53	- 3.47	- 2.31	12.85
CO	71.04	- 29.53	- 29.74	- 35.80	- 22.49	- 33.90	- 35.16	- 32.24	23.20
CH4	.00	.08	.....	.10	.07	.17	.41	1.02	1.52
CO2	.00	27.33	41.88	24.15	58.36	40.96	37.39	50.93	75.57
C2H6	.00	.....	.....	.....	.....	.....	.....	.....	.77
H2O	74.15	9.98	11.66	53.80	- 19.26	11.04	23.58	- 1.68	71.66
SLURRY	445.01	-116.63	-134.49	-155.93	-169.59	-178.88	-153.69	-108.90	423.17
MAF	148.37	43.20	39.96	35.51	26.29	29.45	19.45	18.66	27.68
SOL RESID	12.67	7.29	14.51	6.68	7.70	.89	20.78	42.13	75.18
DISTILLATES (LIQ)	283.97	- 89.73	-108.56	-118.28	-120.36	-128.88	-104.92	- 74.73	320.31
DISTILLATES (VAP)		132.47	132.28	139.44	169.79	182.54	142.66	98.94	
DISTILLABLES		42.73	23.72	21.16	49.43	53.66	37.74	24.21	
CONVERSION		56.80	60.04	64.49	73.71	70.55	80.55	81.34	

Z1= 1
Z9= 2

RUN #	N179
COAL	ZAP
SOLVENT	A04
ADDITIVE	NONE
DATE	9-14-83
CONDITIONS	
RES TIME (MIN)	20-60
AVG TEMP (C)	358-441
MAX TEMP (C)	360-444
MAX PRESS, PSIG	3970-2525
% H2 IN FEED GAS	0-50

		NET YIELDS (WT% MAF)							
COMPONENTS	GRAMS @ T=00	T=01	T=03	T=06	T=10	T=20	T=40	T=60	GRAMS @ TERMINATION
H2	8.26	- 1.05	- 2.88	- 1.25	- 3.78	- 2.66	- 1.74	- 1.73	5.63
CO	186.88	- 20.36	- 60.56	- 20.89	- 83.05	- 57.97	- 40.01	- 50.84	109.44
CH4	.00	.....	.00	.....	.00	.00	.00	.....	3.30
CO2	.00	170.41	54.35	106.95	47.70	85.33	116.41	70.77	107.79
C2H6	.00	.....	.....	.....	.....	.....	.....	.....	2.05
H2O	76.07	.....	38.78	- 24.35	60.57	14.04	- 28.26	- 12.55	56.95
SLURRY	456.95	-121.32	-153.16	-153.72	-150.87	-152.59	-135.44	-136.85	444.82
MAF	152.32	54.88	40.67	35.64	36.54	36.39	35.11	28.43	43.30
SOL RESID	13.10	- 5.29	- 4.12	8.38	10.45	9.81	17.57	26.98	54.19
DISTILLATES (LIQ)	291.53	-100.31	-114.09	-118.14	-118.17	-119.18	-106.65	-106.80	347.33
DISTILLATES (VAP)		.....	148.45	114.24	150.34	134.83	108.16	146.34	
DISTILLABLES		.....	34.36	- 3.90	32.17	15.65	1.51	39.54	
CONVERSION		45.12	59.33	64.36	63.46	63.61	64.89	71.57	

Z1= 1
Z9= 2

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RUN #	N180
COAL	ZAP
SOLVENT	A04
ADDITIVE	NONE
DATE	9-15-83
CONDITIONS	
RES TIME (MIN)	20-60
AVG TEMP (C)	359-440
MAX TEMP (C)	360-443
MAX PRESS, PSIG	4365-2750
% H2 IN FEED GAS	100-10

		NET YIELDS (WT% MAF)							
COMPONENTS	GRAMS @ T=00	T=01	T=03	T=06	T=10	T=20	T=40	T=60	GRAMS @ TERMINATION
H2	21.29	- 2.96	- .34	- .60	- 2.65	1.13	- .75	- 4.81	14.25
CO	.00	.00	.....	.00	.00	.....	.....	.....	10.40
CH4	.00	12.20	1.61	1.61	3.31	4.98	7.39	2.96	4.33
CO2	.00	.00	.00	.00	.00	.00	.....	.....	51.80
C2H6	.00	.....	.....	.....	.....	.....	.....	.....	2.64
H2O	73.09	- 9.62	- 21.52	- 19.69	- 4.01	- 41.50	- 31.08	6.55	82.67
SLURRY	438.80	-144.20	-124.57	-133.34	-141.28	-121.43	-124.29	-132.58	369.75
MAF	146.25	35.13	40.67	40.90	36.94	41.77	42.54	35.47	51.88
SOL RESID	12.58	3.56	3.96	5.23	8.17	8.03	14.59	19.10	40.52
DISTILLATES (LIQ)	279.97	-111.83	-101.24	-111.42	-115.52	-103.17	-112.58	-113.42	277.35
DISTILLATES (VAP)		174.42	177.76	184.86	174.65	189.66	180.78	155.04	
DISTILLABLES		62.59	76.52	73.44	59.13	86.49	68.20	41.62	
CONVERSION		64.87	59.33	59.10	63.06	58.23	57.46	64.53	

Z1= 1
Z9= 2

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Appendix I

STATISTICAL ANALYSIS

Statistical Analysis of Operating Parameters

Operating Parameters

Run Number	Average Temperature 1st Stage		Average Temperature 2nd Stage	Maximum Temperature 1st Stage		Maximum Temperature 2nd Stage
N-174	365	-	439	371	-	440
N-175	365	-	440	371	-	441
N-176	365	-	440	369	-	441
N-177	366	-	400	368	-	444
N-178	361	-	427	367	-	429
N-179	358	-	441	360	-	441
N-180	359	-	440	360	-	443

$\bar{X}_{1st\text{-}A}{}^{a} = 363.43$ $\quad S_{1st\text{-}A} = 4.28$

$\bar{X}_{2nd\text{-}A}{}^{b} = 438.14$ $\quad S_{2nd\text{-}A} = 4.95$

$\bar{X}_{1st\text{-}M}{}^{c} = 366.00$ $\quad S_{1st\text{-}M} = 4.32$

$\bar{X}_{2nd\text{-}M}{}^{d} = 439.86$ $\quad S_{2nd\text{-}M} = 4.98$

[a] 1st Stage Average Temperature
[b] 2nd Stage Average Temperature
[c] 1st Stage Maximum Temperature
[d] 2nd Stage Maximum Temperature

At 97.5% probability t = 2.447

Sample Calculation:

$$t = \frac{\frac{(\bar{X} - U_o)}{S}}{(n-1)^5} = \frac{\frac{(363.43 - 370)}{4.276}}{(7-1)^5} = -3.764$$

$\bar{X}$ - Average Temperature for seven runs
S - Standard Deviation for seven runs
U_o - Temperature for Specific Run
n - Number of Runs

Test Results:

Run Number	Average Temperature 1st Stage	Average Temperature 2nd Stage	Maximum Temperature 1st Stage	Maximum Temperature 2nd Stage
N-174	-3.764*	-0.426	-2.835*	-0.069
N-175	-0.899	-0.921	-0.567	-0.561
N-176	-0.889	-0.921	-1.701	-0.561
N-177	-1.473	-0.921	-1.134	-2.036
N-178	1.391	5.516*	-0.567	5.341*
N-179	3.110*	-1.416	3.402*	-0.561
N-180	2.540*	-0.921	3.402*	-1.544

* t-test Rejects

t-test rejects:

N-174 1st Stage Average and Maximum Temperatures
N-178 2nd Stage Average and Maximum Temperatures
N-179 1st Stage Average and Maximum Temperatures
N-180 1st Stage Average and Maximum Temperatures

Statistical Analysis for Equivalent Hydrogen Consumption using Linear Regression

Equivalent Hydrogen Consumption during Second Stage Reaction Period[a]

Run	Hydrogen in	Time (Minutes)						
Number	Feed Gas (Mole %)	1	3	6	10	20	40	60
N-177	25	0.2	-	0.3	0.5	0.2	-	1.3
N-179	50	0.8	-	0.9	-	-	1.5	1.7
N-176	75	0.9	1.7	1.5	-	1.6	1.4	2.0
N-178	100	0.9	1.3	-	-	1.5	2.2	1.7

[a] - in Moles

x_i = Time of Sample

n = Number of Samples (7)

B_0 = y-intercept of Line

S_B= Estimated Variance of y-intercept of Line

y_i = Equivalent Hydrogen Consumption

B_1 = Slope of Line

S_B = Estimated Variance of Regression Coefficient B_1

Mathematical Definitions of Terms

$$(\bar{x}_i - x)*(\bar{y}_i - y) = \frac{\Sigma(x_i*y_i)-(\Sigma x_i * \Sigma y_i)}{n}$$

$$(x_i - \bar{x})^2 = \Sigma x_i^2 - \frac{\Sigma(x_i)^2}{n}$$

$$\hat{B}_1 = \frac{\Sigma(x_i - \bar{x})*(y_i - \bar{y})}{\Sigma(x_i - \bar{x})^2}$$

$$\hat{\sigma}^2 = \frac{\{\Sigma(\bar{Y}_i - Y)^2 - \hat{B}_1*(\bar{X}_i - X)*(\bar{Y}_i - Y)\}}{n-2}$$

$$s^2 = \hat{\sigma}^2$$

$$S_{B_1} = \left(\frac{s^2}{\Sigma (X_i - \bar{X})^2} \right)^{1/2}$$

$$t = \frac{\hat{B}_1 - B_1}{S_{\hat{B}_1}}$$

$$S_{\hat{B}_0} = \left(\left(\frac{1}{n} + \frac{\bar{X}^2}{(X_i - \bar{X})} \right) * \hat{\sigma}^2 \right)^{1/2}$$

$$t_{17,0\ 975} = 2.110$$

Lower Boundary for slope $= B_1 - S_{\hat{B}_1} * t_{n-2,1-\alpha/2}$
$= 0.0168 - 0.06688 * 2.11$
$= -0.124$

Upper Boundary for slope $= B_1 + S_{\hat{B}_1} * t_{n-2,1-\alpha/2}$
$= 0.0168 + 0.06688 * 2.11$
$= 0.158$

Lower Boundary for y-intercept $= B_0 - S_{\hat{B}_0} * t_{n-2,1-\alpha/2}$
$= 0.66 - 0.1539 * 2.11$
$= 0.335$

Upper Boundary for y-intercept $= B_0 + S_{\hat{B}_0} * t_{n-2,1-\alpha/2}$
$= 0.66 - 0.1539 * 2.11$
$= 0.985$

Determination of Common Line

The common line was found using linear regression for the data points from all four lines.

Slope of common line $B_1 = 0.0168$

Standard Deviation $S_{\hat{B}_1} = 0.06688$

Correlation $= 0.66$

Test for N-176

$B_1 = 0.0253$ $B_0 = 1.02$ Correlation = 0.84

$$\frac{0.0168 - 0.0253}{0.06688} = -0.127$$

Test for N-177

$B_1 = 0.018$ $B_0 = 0.15$ Correlation = 0.92

$$\frac{0.0168 - 0.018}{0.06688} = -0.0179$$

Test for N-178

$B_1 = 0.014$ $B_0 = 1.16$ Correlation = 0.75

$$\frac{0.0168 - 0.014}{0.06688} = -0.0419$$

Test for N-179

$B_1 = -.016$ $B_0 = 0.81$ Correlation = 0.99

$$\frac{0.0168 - 0.016}{0.06688} = 0.012$$

Hypothesis: Slope is the same as common line.

N-176 $-0.124 < -0.127 < 0.158$ Reject: Slope is not the same.

N-177 $-0.124 < -0.018 < 0.158$ Accept: Slope is the same.

N-178 $-0.124 < -0.419 < 0.158$ Accept: Slope is the same.

N-179 $-0.124 < 0.012 < 0.158$ Accept: Slope is the same.

Hypothesis: y-intercept is the same as common line

N-176 $0.335 < 1.02 > 0.985$ Reject: y-intercept is not the same.

N-177 $0.335 > 0.15 < 0.985$ Reject: y-intercept is not the same.

N-178 $0.335 < 1.16 > 0.985$ Reject: y-intercept is not the same.

N-179 $0.335 < 0.81 < 0.985$ Accept: y-intercept is the same.

Conclusion:

The lines are not the same as the common line and therefore the lines are not the same.

Appendix J

WATER-GAS SHIFT REACTION RESULTS

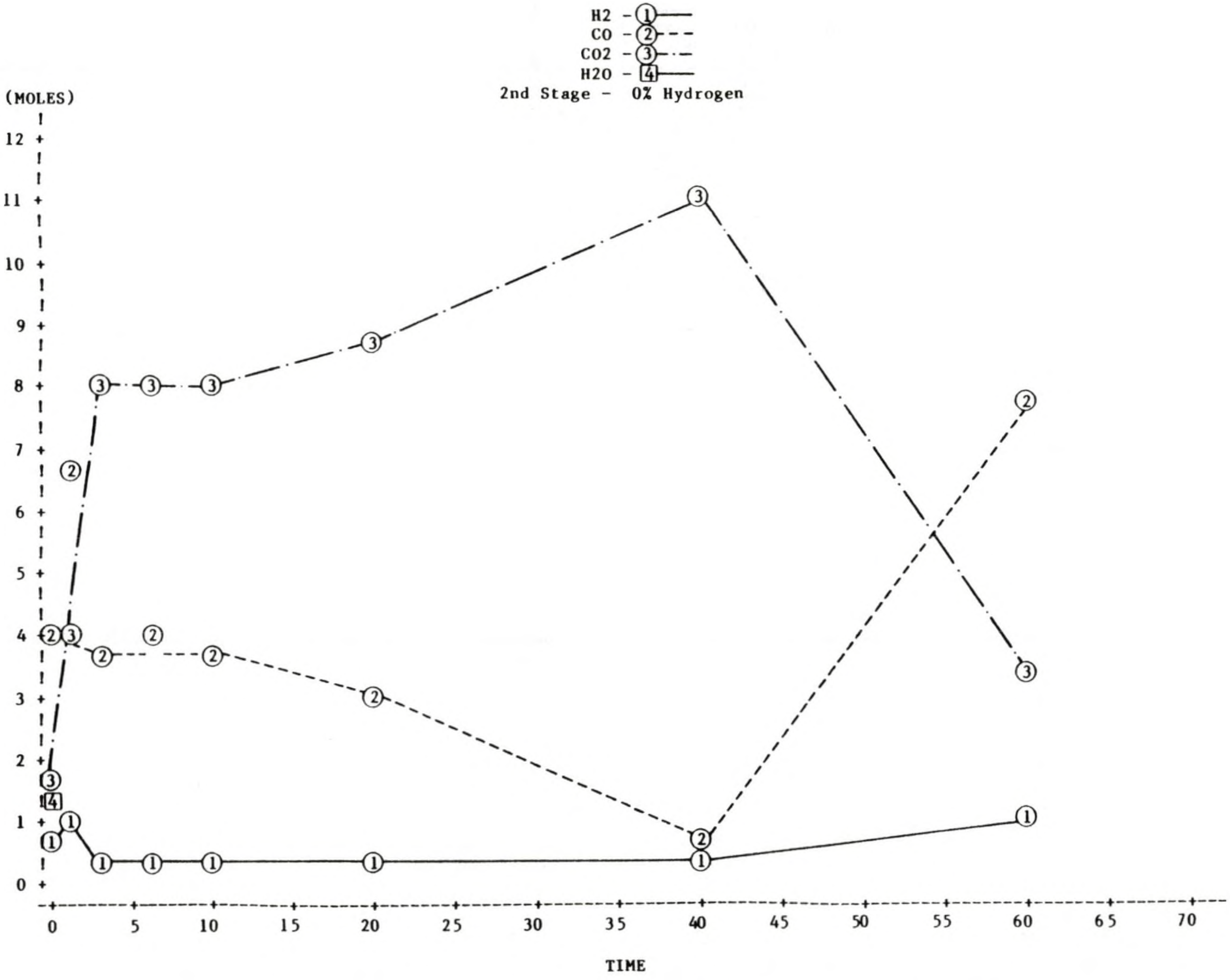

Figure 12. Gas Composition for Run N-175

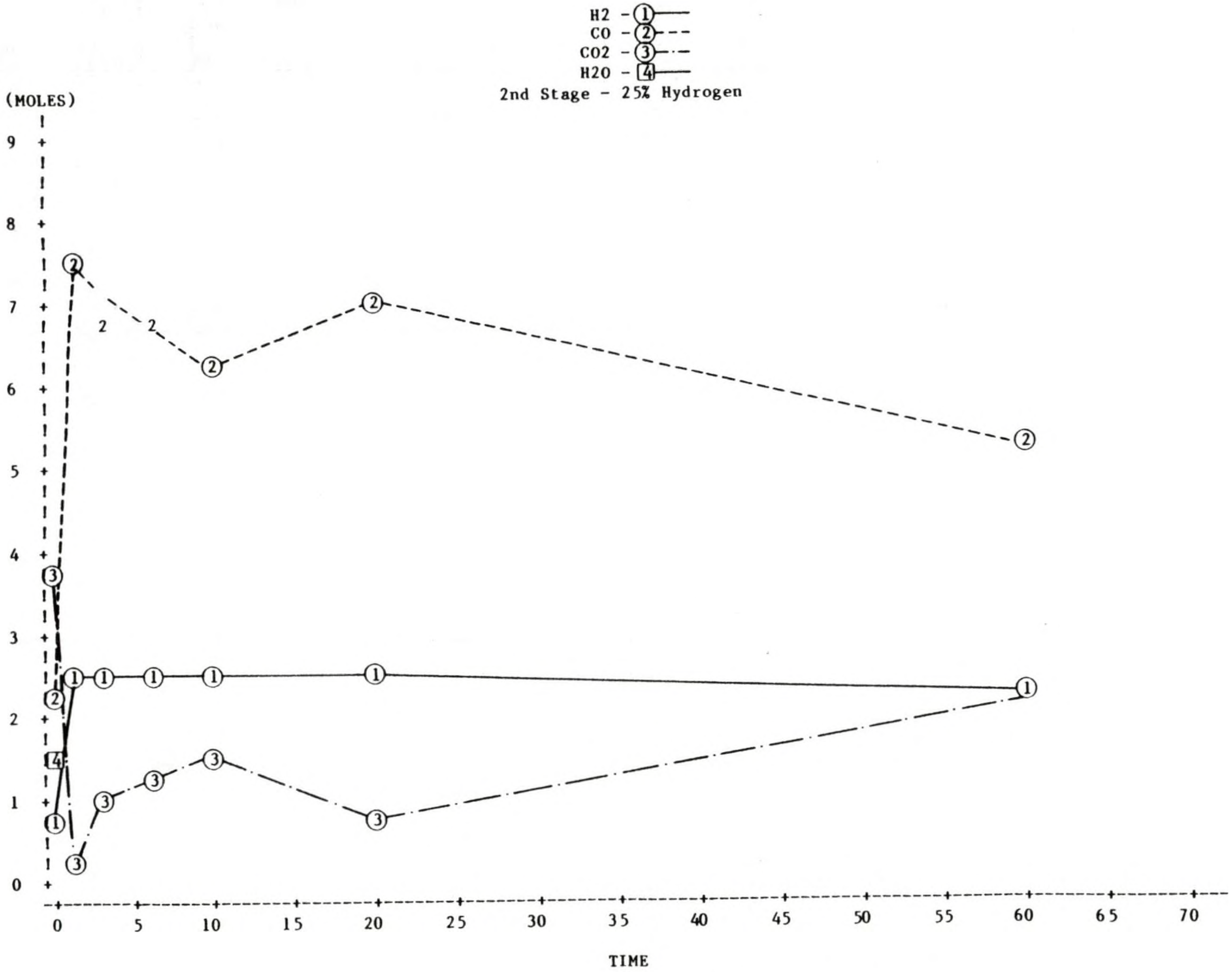

Figure 13. Gas Composition for Run N-177

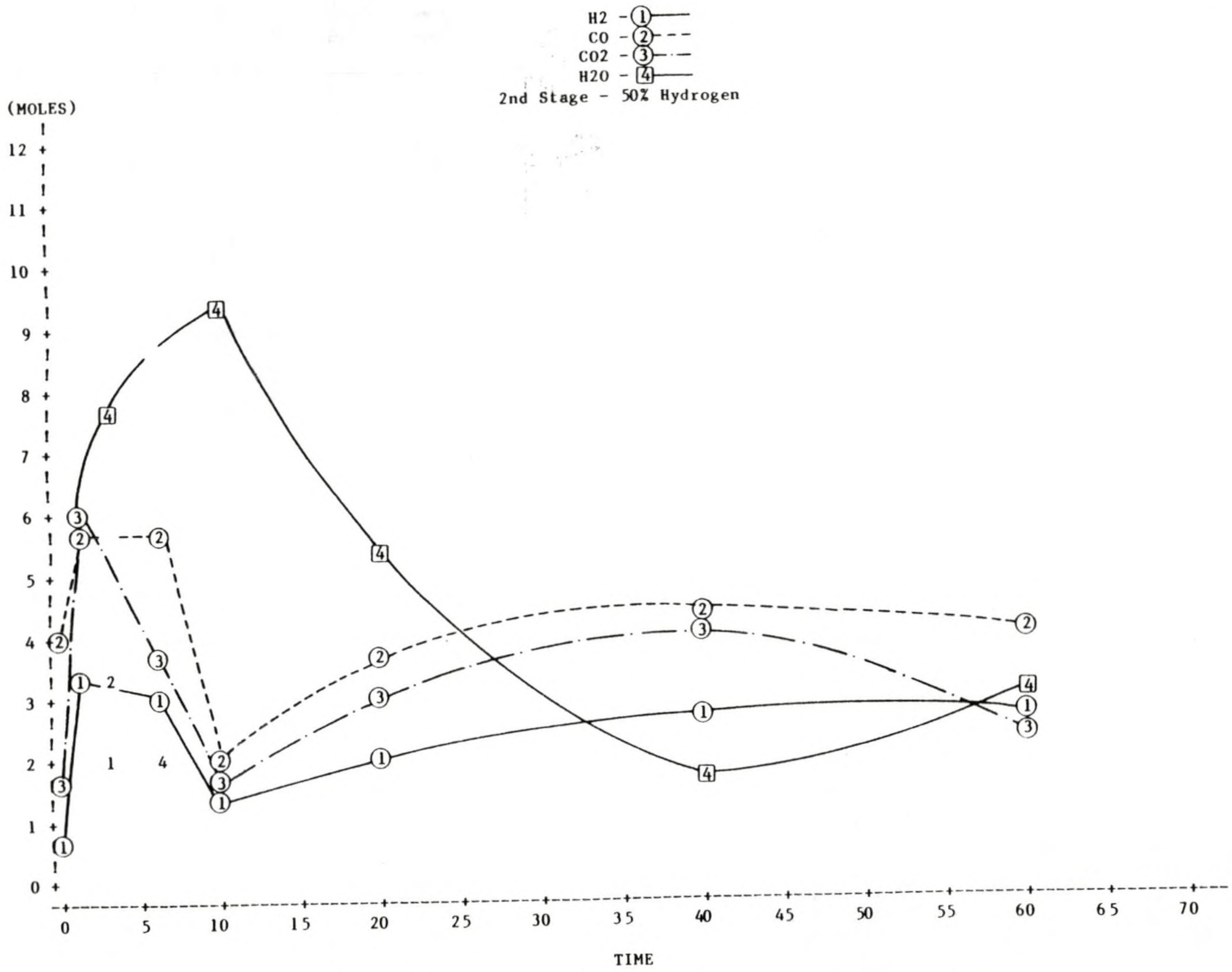

Figure 14. Gas Composition for Run N-179

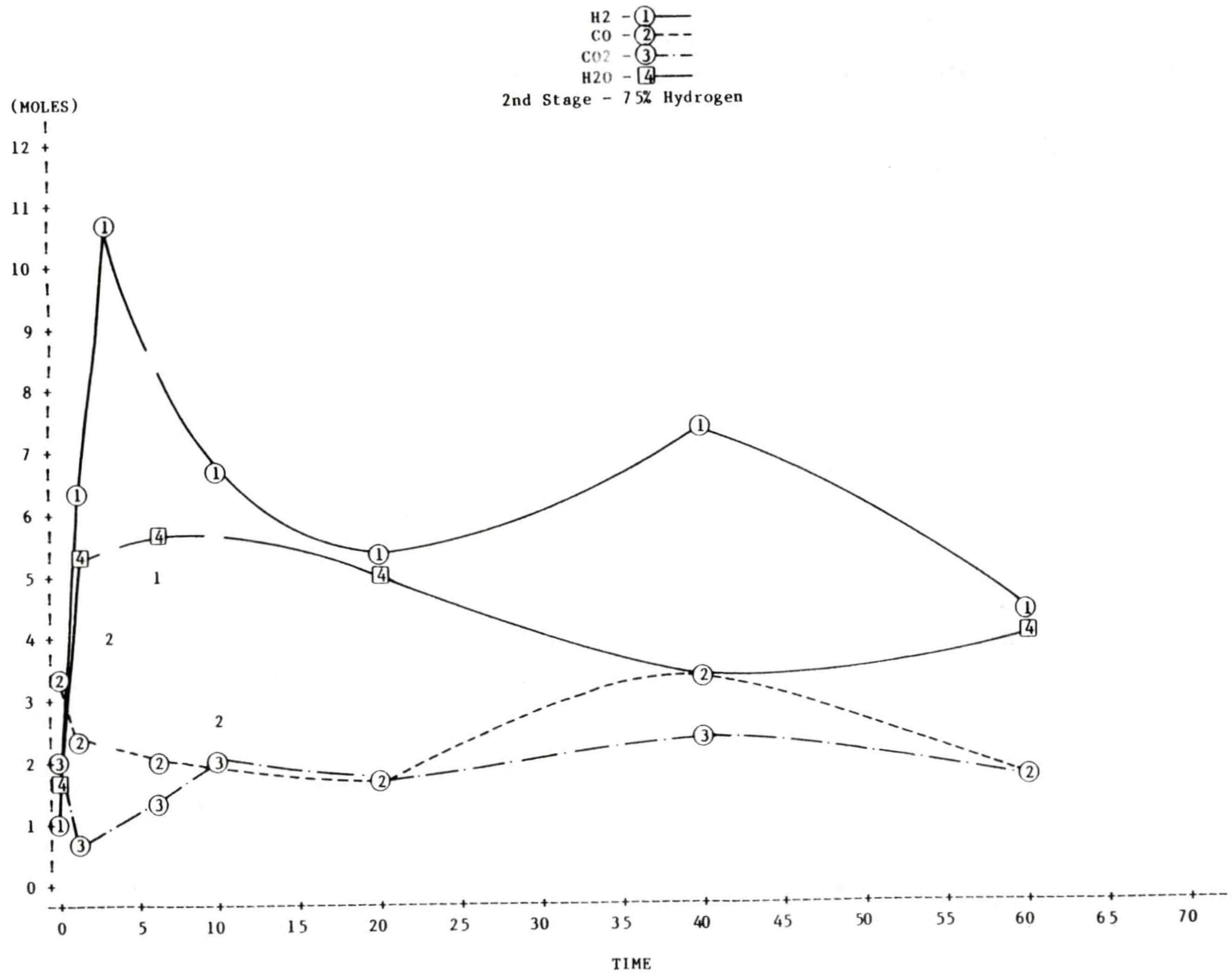

Figure 15. Gas Composition for Run N-176

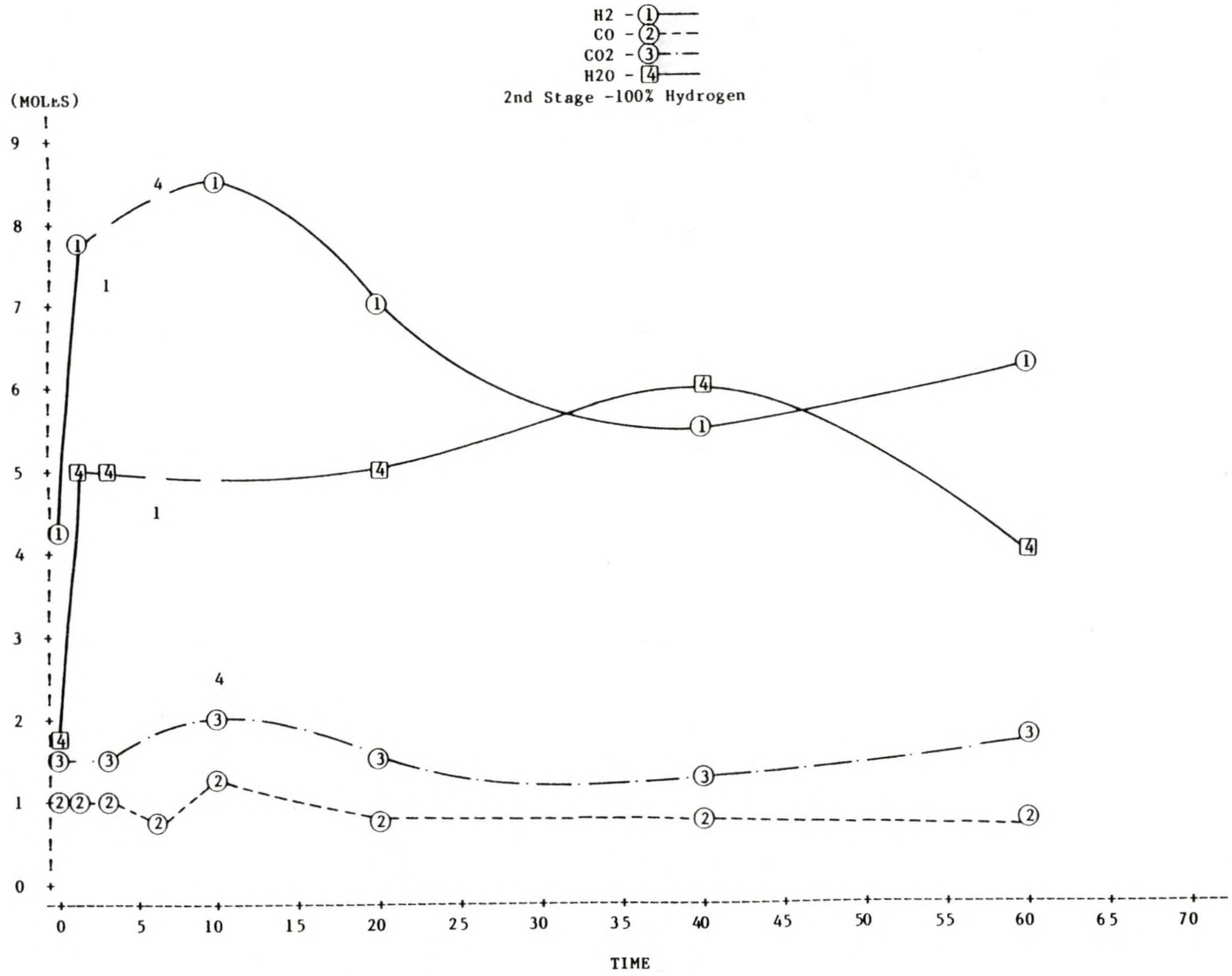

Figure 16. Gas Composition for Run N-178

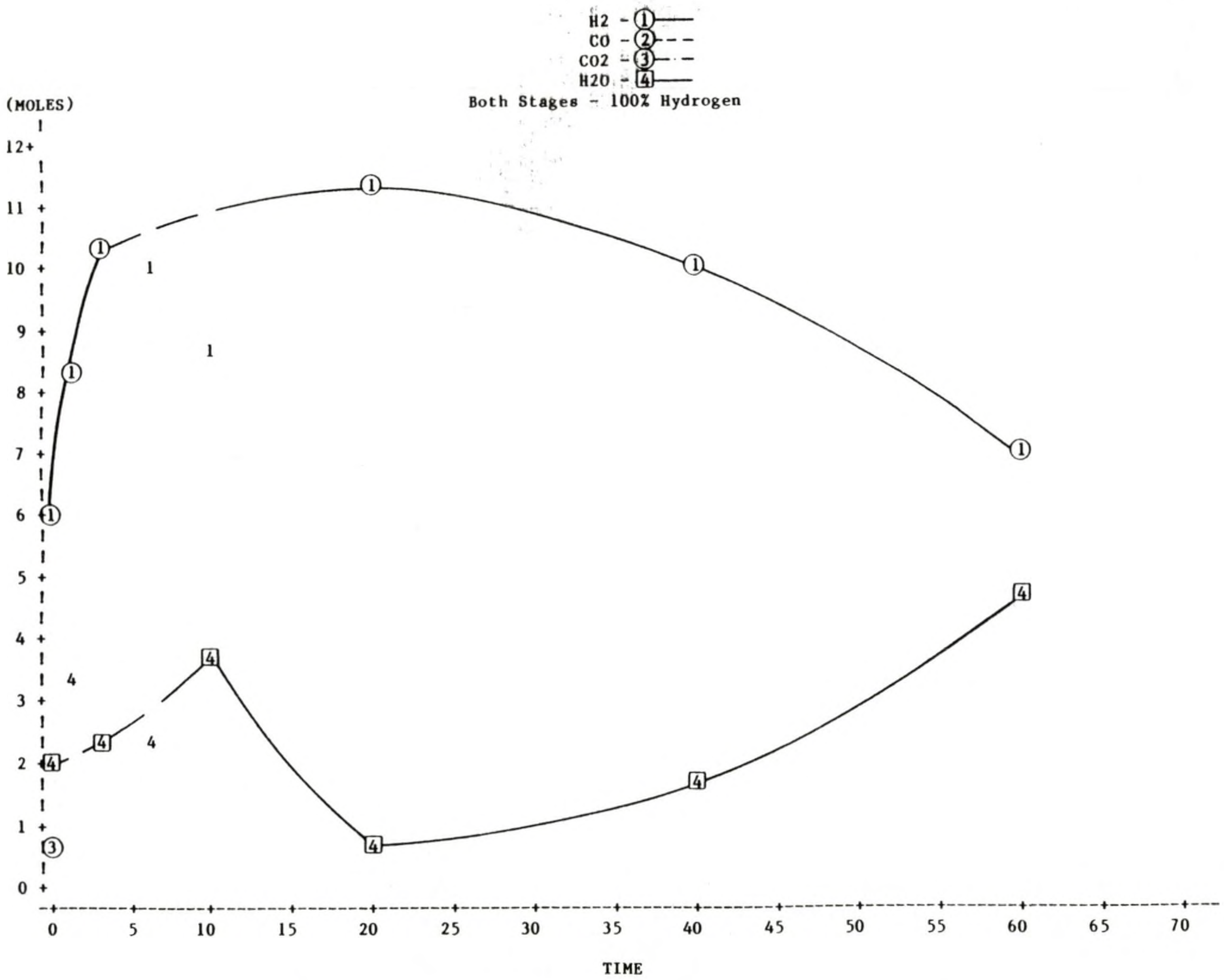

Figure 17. Gas Composition for Run N-180

Appendix K

ABBREVIATIONS

ABBREVIATIONS

aliq	Aliquot
AO4	Anthracene oil from run number 4
atm	Atmosphere
C	Celsius
CO	Carbon monoxide
CO_2	Carbon dioxide
C1-C3	Methane, ethane, propane
CPU	Continuous Process Unit
et al.	And others
GFETC	Grand Forks Energy Technology Center
hr	Hour
H_2O	Water
H_2	Hydrogen
H.O.	Heavy oil
i.e.	Id est, that is
IOM	Insoluble organic matter
gms	Grams
lb	Pounds
L.O.	Light oil
MAF	Moisture- and ash-free
mg	Milligram
ml	Milliliter
M.O.	Middle oil
MW	Molecular weight

PETC	Pittsburgh Energy Technology Center
PDU	Process development unit
psi	Pounds per square inch
psia	Pounds per square inch absolute
psig	Pounds per square inch gage
SRL	Solvent refined lignite
THF	Tetrahydrofuran
UND	University of North Dakota
USDI	United States Department of Interior
vol	Volume
wt	Weight

REFERENCES

1. Berkowitz, N. An Introduction to Coal Technology. Academic Press, Inc., New York, 1979, pp xi

2. Berkowitz, N. An Introduction to Coal Technology. Academic Press, Inc., New York, 1979, pp 303-305

3. Berkowitz, N. An Introduction to Coal Technology. Academic Press, Inc., New York, 1979, pp 305-312

4. Probstein, Ronald F., and R. Edwin Hicks. Synthetic Fuels. McGraw-Hill, Inc., New York, 1982, pp 131-133

5. Probstein, Ronald F., and R. Edwin Hicks. Synthetic Fuels. McGraw-Hill, Inc., New York, 1982, pp 293-309

6. Appel, H. R., I. Weder, and R. D. Miller. Liquefaction of Lignite with Carbon Monoxide and Water. Symp. on Tech. and Use of Lignite, Bismarck, N. D., May 12-13, 1971, Bur. Mines Infor. Circ. 8543, Supt. of Documents, U. S. Gov. Printing Office, Wash., D. C., 1972, pp 32-39

7. Appell, H. R., I. Wender, and R. D. Miller. Dissimilar Behavior of Carbon Monoxide plus Water and of Hydrogen in Hydrogenation. Div. Fuel Chem. preprints, Am. Chem. Soc., 13, No. 4, 39-44 (1969)

8. Sondreal, Evert A., Warrack G. Wilson, and Virgil I. Stenburg. Mechanisms Leading to Process Improvements in Lignite Liquefaction using CO and H_2. Fuel, Vol 61, pp 925-937

9. Appell, H. R., E. C. Moroni, and R. D. Miller. Hydrogenation of Lignite with Synthesis Gas. Energy Sources, 3, No. 2, 163-175 (1977)

10. Del Bel E., S. Friedman, P. M. Yavorsky, and I. Wender. Oil by Luquefaction of Lignite. Coal Processing Tech., Vol.2, AICHE, 1975, pp 104-108

11. Severson, D. E., A. M. Souby, and W. R. Kube. Laboratory Studies of Solution-Hydrogenation of Lignite. Symp. on Tech. and Use of Lignite, Grand Forks, N. D., May 9-10, 1973, Bur. Mines Infor. Circ. 8650. Supt. of Documents, U. S. Gov. Printing Office, Wash., D. C., 1974, pp 236-246

12. Souby, A. M., D. E. Severson, and W. R. Kube. Project Lignite: Convenience Fuels from Northern Great Plains Province Lignite. Annual Proc. from the N. D. Academy of Science 28, Part II, 50-59 (1976)

13. Severson, D. E., A. M. Souby, and G. C. Baker. Continuous Liquefaction of Lignite in a Process Development Unit. Div Fuel Chem., Preprint, Am. Chem. Soc. 22, No. 6, pp 161-182 (1977)

14. Sondreal, E. A., C. L. Knudson, J. E. Schiller, and T. H. May. Development of the CO-STEAM Process for Liquefaction of Lignite and Western Sub-bituminous Coals. Symp on Tech. and Use of Lignite, Grand Forks, N. D., May 18-19, 1977, GFERC/IC-77/1, NTIS, Springfield, Virg., 1978, pp 129-158

15. Willson, W. G., C. L. Knudson, G. C. Baker, T. C. Owens, and D. E. Severson. Application of Liquefaction Processes to Low Rank Coals. Symp. on Tech. and Use of Lignite, Grand Forks, N. D., May 30-31, 1979, GFERC/IC-79/1, NTIS, Springfield, Virg. pp 170-206

16. Knudson, G. L. W. G. Willson, and G. C. Baker. Hydrogen-Carbon Monoxide Reactions in Low-Rank Coal Liquefaction Div. Fuel Chem. Preprint, Am. Chem. Soc., 26, No. 1 132-141 (1981)

17. Rindt J. R., D. E. Severson, and A. M. Souby. UND Hot-Charge Time-Sample Batch Autoclave Facility. For Presentation at the American Institute of Chemical Engineers, June 8-12, 1980, pp 1-61

18. Rindt, J. R. Private Communications, 1983, Available upon request from J. R. Rindt, University of North Dakota Energy Research Center, Grand Forks, ND

19. Rindt, J. R. Private Communications, 1983, Available upon request from J. R. Rindt, University of North Dakota Energy Research Center, Grand Forks, ND

20. American Society for Testing and Materials. Standard Test Method for Hydrogen Sulfide and Mercaptan Sufur in Natural Gas (Cadium Sulfate-Iodometric Titration Method) D 2385-66 in 1977 Annual Book of ASTM Standards: Part 24. Petroleum Products and Lubricants, Philadelphia, Pa., 1977, pp 333-336

21. Crockfor, H. D. J. W. Nowell, H. W. Baird, and F. W. Getzeru. Laboratory Manual of Physical Chemistry. John Willey and Sons Inc., New York, 2nd Ed., 1975, pp 44-49

22. Wiltsee, A. W., Jr. Low-Rank Coal Research Under the UND/DOE Cooperative Agreement. Quarterly Technical Progress Report for the Period April-June, 1983, Prepared for the U. S. Department of Energy, Energy Research Center, University of North Dakota, pp 5.1-5.7

23. Wiltsee, A. W., Jr. Low-Rank Coal Research Under the UND/DOE Cooperative Agreement. Quarterly Technical Progress Report for the Period Oct.-Dec., 1983, Prepared for the U. S. Department of Energy, Energy Research Center, University of North Dakota, pp 5.14-5.23

24. Gagliardi, John James. Lignite Liquefaction Using Carbon Monoxide, Hydrogen And Water In A Hot Charged, Two-Staged, Batch Autoclave System. Masters Thesis, University of North Dakota, January 1984

25. Lee, Chia-Shium. The Effect of Two Catalysts (Ammonia and Quinoline) on the Liquefaction of North Dakota Lignite. Masters Thesis, University of North Dakota, June 1984, pp 27-33

26. Sondreal Evert A., Warrack G. Wilson, and Virgil I. Stenberg. Mechanisms leading to process improvements in Lignite Liquefaction using CO and H_2S. Fuel, Vol 61, Oct. 1982, pp 925-937

27. Wiltsee, A. W., Jr. Low-Rank Coal Research Under the UND/DOE Cooperative Agreement. Quarterly Technical Progress Report for the Period April-June, 1983, Prepared for the U. S. Department of Energy, Energy Research Center, University of North Dakota, pp 5.1-5.7

28. Wiltsee, A. W., Jr. Low-Rank Coal Research Under the UND/DOE Cooperative Agreement. Quarterly Technical Progress Report for the Period Oct.-Dec., 1983, Prepared for the U. S. Department of Energy, Energy Research Center, University of North Dakota, pp 5.14-5.23

29. Berkowitz, N. An Introduction to Coal Technology. Academic Press, Inc., New York, 1979, pp 523-524

30. Rindt, J. R. Private Communications, 1983, Available upon request from J. R. Rindt, University of North Dakota Energy Research Center, Grand Forks, ND

31. Cassidy, Peter J. Where's the Key to Coal Liquefaction?, Chemtech, Sept. 1983, pp 562-567