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## Autoclave Studies of Lignite Liquefaction

Bruce G. Miller

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AUTOCLAVE STUDIES OF LIGNITE LIQUEFACTION

by  
Bruce G. Miller, M.S.

Bachelor of Science in Chemical Engineering  
University of North Dakota, 1981

A Thesis

Submitted to the Graduate Faculty

of the

University of North Dakota

in partial fulfillment of the requirements

for the degree of

Master of Science

Grand Forks, North Dakota

August  
1982

## Autoclave Studies of Lignite Liquefaction

Bruce G. Miller, M.S.

The University of North Dakota, 1982

Faculty Advisor: Dr. Thomas C. Owens

The effect of time at reaction temperature on the liquefaction of lignite using hydrogen or synthesis gas was studied. A slurry containing 200 grams of moisture-ash-free (MAF) lignite, 100 grams of water, and 400 grams of hydrogenated anthracene oil solvent was fed into the University of North Dakota Time-Sampling Hot-Charge Batch Autoclave System with the gas at an initial pressure of 400 psig.

Oil yields and conversions ranged from 0.9 percent to 35.7 percent and 66.7 percent to 90.6 percent, respectively, increasing as the time at or near the reaction temperature (380-420°C) increased. Higher conversions and greater oil and gas yields were obtained when using synthesis gas as compared to pure hydrogen. The increase in gas yield for the synthesis gas runs can be attributed to increased carbon dioxide production.

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T1982  
M612

This Thesis submitted by Bruce G. Miller 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  
(Chairman)

R. Hara

Wm J. Halverson

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.

\_\_\_\_\_  
Dean of the Graduate School

Permission

Title Autoclave Studies of Lignite Liquefaction

Department Chemical Engineering

Degree Master of Science

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Signature Bruce G. Miller

Date July 27, 1987

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## ABSTRACT

The effect of time at reaction temperature on the liquefaction of lignite using hydrogen or synthesis gas was studied. A slurry containing 200 grams of moisture-ash-free (MAF) lignite, 100 grams of water, and 400 grams of hydrogenated anthracene oil solvent was fed into the University of North Dakota Time-Sampling Hot-Charge Batch Autoclave System with the gas at an initial pressure of 400 psig.

Oil yields and conversions ranged from 0.9 percent to 35.7 percent and 66.7 percent to 90.6 percent, respectively, increasing as the time at or near the reaction temperature (380-420°C) increased. Higher conversions and greater oil and gas yields were obtained when using synthesis gas as compared to pure hydrogen. The increase in gas yield for the synthesis gas runs can be attributed to increased carbon dioxide production.

## CHAPTER I

### INTRODUCTION

Interest in converting coal to liquid products has been rather cyclic and affected by the cost and availability of petroleum. In the beginning of the industrial revolution, coal was the major source of energy in the United States and continued to dominate the United States' energy supply for the next hundred years, as shown in Figure 1 (1).<sup>1</sup> Petroleum quickly became the preferred energy source after its discovery in Pennsylvania in 1859 and its rapid commercial production in the early 1900's. By the early 1920's, worries that oil supplies were being depleted along with an expanding automobile industry caused coal liquefaction research to flourish. But this was short-lived; when oil was discovered in Texas in the mid-1920's, further work on coal liquefaction ceased. After World War II the United States experienced petroleum shortages, and coal liquefaction was again considered as an alternative. A sizeable research effort resulted. However, discovery of massive petroleum reserves in the Middle East in the mid-1940's once again made coal liquefaction uneconomical. In 1973 the United States' petroleum production began to decline and unrest developed in the Middle East. The limited availability of domestic supplies of natural gas and crude oil and the desire to reduce the country's dependence on foreign sources of energy have promoted considerable interest in this country in

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<sup>1</sup>Numbers in parenthesis refer to items on the List of References at the end of this paper.

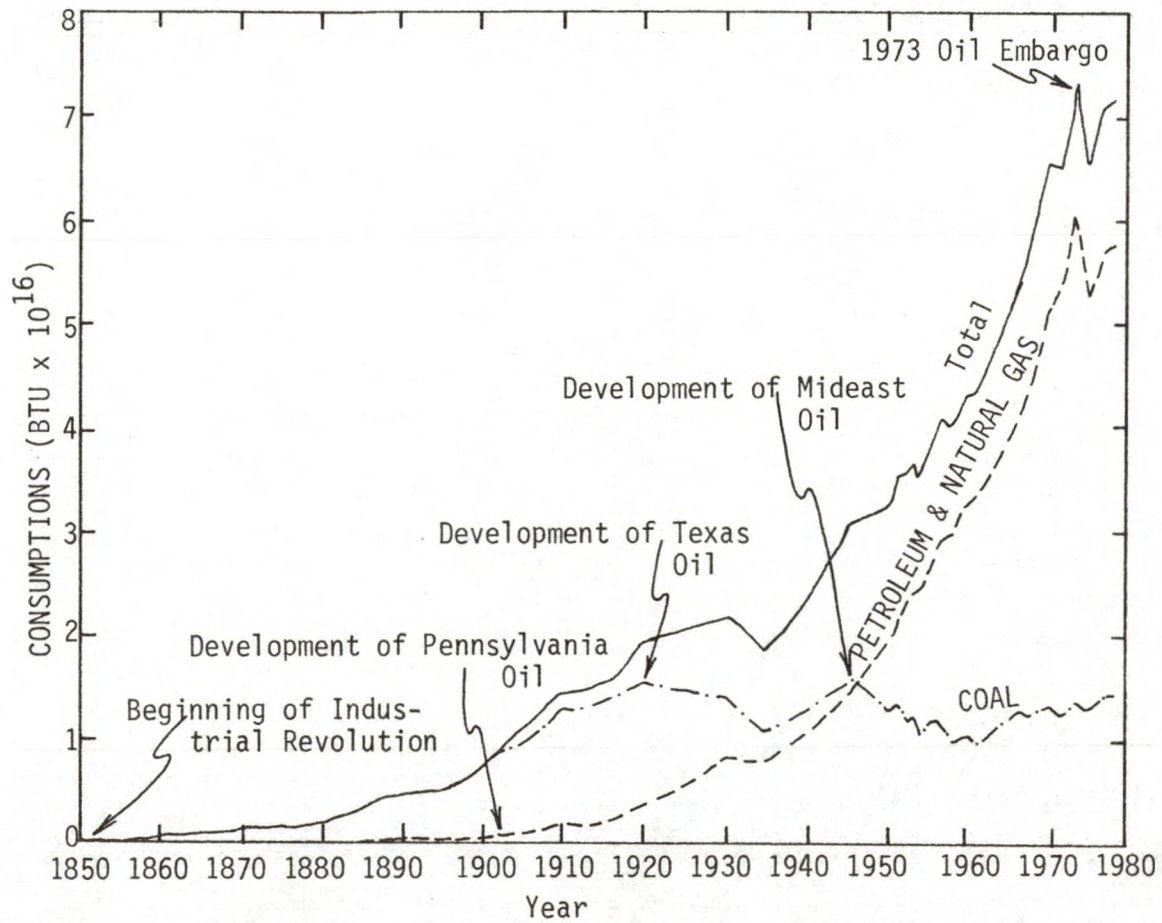


Figure 1. Consumption of Fossil Fuels in the United States.

developing alternative domestic sources of fuel. Because of the abundance of mineable coal reserves in the United States, coal liquefaction is once again being considered as a major source of liquid fuels.

The major differences between coal and petroleum are the ratio of hydrogen to carbon and the ash content (2). Coal has an atomic hydrogen to carbon ratio of approximately 0.8, while the ratio for oil is about 1.8. Coal has an ash content that can be as high as 15 percent, whereas oil seldom has over a few tenths of a percent. Thus, the problem in liquefaction is to increase the hydrogen content of the material and to eliminate the ash.

In this work, lignite liquefaction using hydrogen or synthesis gas was studied. The effects of temperature and feed gas composition on lignite liquefaction were examined.

## CHAPTER II

### PREVIOUS EXPERIMENTAL WORK

Coal was hydrogenated in the laboratory by Berthelot as early as 1869. The reaction was carried out with hydriodic acid at 270°C for 24 hours, and a 67 percent yield of oil containing aromatics and naphthenes was obtained (3).

In 1911 Bergius obtained oil by hydrogenating coal without a catalyst under hydrogen pressure at 300 to 350°C. In 1913 he applied for the first patent on coal hydrogenation, and in 1931 he was awarded the Nobel Prize in Chemistry (3,4). Bergius also observed that coal paste could be injected readily into a vessel under pressure. The role of catalysts in the hydrogenation of coal was not realized until later.

At the end of 1925, I. G. Farben hydrogenated coal using a molybdenum oxide catalyst. The presence of the catalyst allowed the hydrogenation of coal in the presence of excess hydrogen at low partial pressure and at temperatures of 400 to 450°C.

In the following year, Farben conducted the liquefaction process in two steps because high-molecular-weight materials in the intermediate hydrogenation product fouled the catalyst. Coal was mixed with catalyst and hydrogenated in the liquid phase to middle oil, which was further hydrogenated to gasoline in vapor-phase over a fixed bed of catalyst (3).

Pott and Broche showed that it was possible to liquefy coals to considerable extent, in some cases to 80 percent and higher, into

suitable oils or mixtures of oils by the method of pressure extraction (5). Also, it was noted that in order to effect considerable liquefaction of the coal, the pressure extraction must be carried out at increasing temperatures up to 400°C. The temperature increase must be regulated so it follows the gradually increasing temperature of decomposition of the material undergoing extraction. The coal fractions going into solution were then easily converted into oils by pressure hydrogenation at temperatures of 430 to 450°C.

The Bureau of Mines started work on the hydrogenation of coal at Pittsburgh, Pennsylvania in 1934 and used batch autoclaves. This was followed by continuous liquid-phase hydrogenation in 1936.

The feasibility of using total extraction as a means for producing clean fuels from coals was investigated in the early 1960's by Spencer Chemical Company under a contract with the then U.S. Office of Coal Research and a Spencer affiliate, the Pittsburgh and Midway Coal Mining Company (4). This work led to the Solvent-Refined Coal (SRC) process and was based on the older Pott-Broche technology. The purpose of the SRC process is to produce an environmentally acceptable solid fuel from coal. Hydrogenation of the coal in the SRC process takes place at elevated pressure and temperature in the presence of hydrogen and without the addition of any catalyst.

In 1921, Fischer and Schrader showed that brown coal could be readily liquefied using carbon monoxide and water (6). This work was not pursued further until the 1960's when Appel, Wender, and other workers at the Pittsburgh Energy Research Center (PERC) demonstrated in autoclave experiments that high yields of a benzene soluble oil could be obtained by treating low rank coals by this method in the presence

of 1,1-a-naphthol-phenanthrene as a solvent (7,8). Most experiments were conducted at fairly high total pressures (270-340 atm) and moderate temperatures (380°C) and obtained as much as 89 percent conversion of lignite to benzene solubles in ten minute residence time.

From 1965 to 1970, batch autoclave runs were carried out at the University of North Dakota (UND) with support from the Great Northern Railway, to investigate the production of liquids from lignite. The process involved contacting lignite slurried with recycle solvent and synthesis gas (50-60% CO + 30-50% H<sub>2</sub>) in a noncatalytic reactor. Conversions as high as 96 percent were obtained and optimum operating conditions of 750°F and 1500 psig initial hydrogen pressure were determined (9). Anthracene oil appeared to be the most promising commercial material for use as a starting solvent (9,10).

In 1972, UND signed a research contract with the U.S. Office of Coal Research to develop the necessary data to design a lignite refinery which would produce high-quality solid, liquid, and gaseous fuels from Northern Plains Province lignite (11,12). Primary effort was directed toward operating a 0.6 ton/day process development unit (PDU) designed for continuous solvent extraction of lignite at pressures up to 2500 psig and at temperatures up to 950°F in an atmosphere of reducing gases, usually synthesis gas, and with a hydrogen donor solvent. The Project Lignite PDU produced approximately 15 pounds per hour of solvent refined lignite (SRL) with a melting point of 300-400°F, as well as additional quantities of lighter liquids and gases (13,14). Continuous operation of the PDU for twenty-eight day periods was demonstrated, but a problem with solids buildup in the reactor was never completely solved (15).



A technique that was under active investigation at the PERC was the COSTEAM process which is intended to produce low-sulfur fuel oils from lignite and subbituminous coals and for this purpose reacts coal-oil slurries with synthesis gas (16). Conversion is assisted by the natural moisture content of the coal which increases the hydrogen partial pressure in the reactor because of the water-gas shift reaction, and by mild catalytic activity of iron-bearing compounds in the mineral matter of the coal.

The COSTEAM process has also been under investigation at the Grand Forks Energy Technology Center (GFETC) since 1975, with the design and construction of a 5 pound per hour continuous process unit (CPU) and a hot-charge time-sampled autoclave system (17,18). The present investigation was performed using the latter system.

## CHAPTER III

### COAL LIQUEFACTION

The term liquefaction as used here includes conversion of coal in which the major product is liquid. The liquid has been rather broadly defined on the basis of solubility in various solvents, and in many cases a major liquid product is solid at room temperature. Gases, water, and distillate oils are produced in addition to the heavy liquid product. Therefore, before an introduction to coal liquefaction is presented, terms used in this study will be defined.

#### Definitions of Terms

The percent conversion is defined as 100 minus the ash-free tetrahydrofuran (THF)-insoluble material as weight percent of MAF lignite charged:

$$\text{Percent Conversion} = \frac{\text{MAF lignite in} - \text{MAF THF-insoluble}}{\text{MAF lignite in}} (100)$$

Oil yield is the MAF ratio of cyclohexane solubles to lignite charged. Cyclohexane was chosen as a solvent based on trial extractions of previously analyzed samples from UND's hot-charged time-sampled batch autoclave studies. The amounts of cyclohexane soluble materials in these samples were closely related to the oil yields determined by microdistillation at 250°C and one torr as shown in Table 1.

SRL is the weight percent of the material soluble in THF but insoluble in cyclohexane expressed as percent of the MAF lignite charged.

TABLE 1  
COMPARISON OF OIL YIELDS DETERMINED BY EXTRACTION AND DISTILLATION

Sample	N-44	N-54	N-68
Distillable Oils	62.9%	66.2%	79.2%
Cyclohexane Solubles	69.5%	70.6%	79.9%

The insoluble organic matter (IOM) is the ash-free portion of the THF insoluble expressed as weight percent of the MAF lignite charged. Previous work at UND showed little difference in the change in mass of ash during liquefaction of Zap coal; therefore, the mass of ash in the THF insoluble material was assumed equal to the mass of ash in the lignite charged (19).

Gas yield is the increase in mass of gas expressed as percent of MAF lignite charged.

### Direct Coal Liquefaction

Most direct coal liquefaction processes react coal, a solvent, and hydrogen gas in the presence of a catalyst at high temperatures and pressures. Catalysts have been omitted from the following discussion because they were not used in this study.

The primary liquefaction processes involve the consumption of hydrogen. The solvent is thought to play the essential role of transferring hydrogen from the gas phase to the coal. Most liquefaction processes are usually conducted between 375 and 465°C, that is, within the range in which pyrolysis or thermal decomposition of the coal becomes important (6). A number of competing chemical processes are taking place simultaneously; the more important ones are coal pyrolysis, coking, hydrogen transfer from solvent to coal, solvent rehydrogenation, and direct interaction of molecular hydrogen with the coal.

### Effect of Solvent

The properties of the solvent can affect the primary liquefaction process in various ways. The donor properties of the solvent are of prime importance in donor solvent extraction. The yield structure will

depend on the nature and concentration of the donors in the solvent present in the system at any time. Once the donor concentration has been increased beyond a minimum value, the yield structure becomes relatively independent of further increases in donor concentration (6).

The operating procedure, conditions, and nature of the solvent can affect the yield structure. For example, previous work has shown that high heating rates such as those obtained when using microautoclaves and continuous units usually result in a lower conversion at the same hydrogen input than when using batch autoclaves (20). Yield structure may also be affected by operating temperature. Relatively high temperatures (350-425°C) cause the coal matrix to swell and also aid in overcoming the van der Waals forces holding the soluble molecules in place (6). It has been noted that lower yields are obtained for the same amount of hydrogen transfer at reduced temperatures (21). The upper limit for the yield of liquid products has been in the range of 40-50 percent of the moisture -and ash- free coal and has been achieved at temperatures in the range 450-460°C. Recent research has shown that it is possible to achieve almost complete conversion of coal to liquid products under relatively mild process conditions when basic nitrogen compounds are present in the solvent (22).

#### Effect of Feed Gas Composition

When carbon monoxide and water were used to liquefy low-rank coals in autoclave experiments, high yields of a benzene soluble oil were obtained (7,8). Low-rank coals were shown to react more readily with carbon monoxide and water than with hydrogen under comparable conditions (23). The relative advantages of carbon monoxide and water versus

hydrogen were also shown to decrease with increasing temperature. Despite this fact, higher conversion is obtained using carbon monoxide over the range of 375 to 425°C.

Experiments done by Appell using pure hydrogen gas have shown that some carbon dioxide is split from the lignite during processing (24).

The use of both pure carbon monoxide and synthesis gas has been investigated for liquefaction; improvements in operating conditions and yield structure were noted as compared with the use of pure hydrogen (25).

Synthesis gas has been used as a replacement for carbon monoxide because it is less expensive and hydrocracking occurs more readily in the presence of hydrogen than in the presence of carbon monoxide (24). Extensive hydrocracking is not desirable but some is needed to reduce the average molecular weight and viscosity of the product. The somewhat higher reactivity of carbon monoxide is offset by the lower cost of the synthesis gas and the need to increase the extent of hydrocracking sufficiently to obtain a liquid product.

The high activity of carbon monoxide in reducing carbonyl groups is believed to be the reason that low-rank coals are liquefied more readily in the presence of carbon monoxide than hydrogen (7). Low-rank coals not only contain more carbonyl groups than higher rank coals but also contain the alkaline materials that are converted to formates, the probable active reducing agents (16). In work done by Appell (25), the high reactivity of aldehydes, and to a lesser extent ketones, with carbon monoxide in the presence of alkali metal carbonates suggested that a crossed Cannizaro-type reaction was occurring between the carbonyl compound and the formate anion with its aldehydic hydrogen.

The effectiveness of carbon monoxide is, therefore, due to its ability to remove a cross-linking rather than any ability to cleave bonds in the lignite. The extent of the reducing action on carbonyl groups is not large because of the limited number of these groups, but the effect is believed to be a significant factor in the liquefaction process.

## CHAPTER IV

### EXPERIMENTAL PROCEDURE AND EQUIPMENT

#### Materials

The lignite used was strip-mined at the Indian Head Mine of the North American Coal Company located near Zap, North Dakota. It was obtained from the Grand Forks Energy Technology Center (GFETC) in powdered form. Size distribution, proximate, and ultimate analyses are shown in Table 2.

The lignite sample was dried at 75-85°F for 23 hours and stirred every half hour to reduce the moisture content from approximately 32 to 30 percent. After the coal was dried, it was stored in plastic garbage bags, double wrapped, with as much air forced out of the bags as possible.

A catalytically hydrogenated anthracene oil (HAO-61) was the solvent used for each run. It was obtained from the GFETC where it was made from an anthracene oil (AO-4). Table 3 shows the analyses of HAO-61 and AO-4.

The water used for each run was distilled water.

Tetrahydrofuran (THF), cyclohexane, and methanol were purchased from Fisher Scientific.

Hydrogen and synthesis gas (44.36 and 55.64 mole percent hydrogen and carbon monoxide, respectively) were purchased from the Linde Division of Union Carbide.



TABLE 2  
ANALYSES OF ZAP LIGNITE

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Size Distribution

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U.S. Screen Size	Percent Passing
60 mesh	100.0
100 mesh	97.5
150 mesh	87.3
170 mesh	75.7
200 mesh	62.8

---

Proximate Analysis<sup>a</sup>

---

Constituent	Percent
Volatile Matter	33.6
Moisture	30.6
Fixed Carbon	28.1
Ash	7.7
Total	<u>100.0</u>

---

<sup>a</sup>as received basis

Ultimate Analysis<sup>b</sup>

---

Constituent	Percent
Carbon	62.07
Hydrogen	3.81
Nitrogen	1.02
Sulfur	1.03
Oxygen (by diff.)	20.97
Ash	11.10

---

<sup>b</sup>moisture free basis

---

TABLE 3  
ANALYSES OF SOLVENTS

Solvent	A04(a)	HA061(b)
ASTM D-1160 Distillation @ 5 torr		
IBP, °C	94	42
Vol. % off at, °C	135	93
10	146	107
20	163	122
30	175	134
40	185	144
50	195	152
60	205	161
70	217	175
80	231	182
90	251	207
95	276	252
Max. Temp., °C	288	273
Vol. % off at Max. Temp.	96.5	97
Calculated from ASTM D-1160		
IBP - 120°C Fraction, Wt. %	3.1	19.2
120 - 260°C Fraction, Wt. %	85.0	77.5
260°C - Max. Temp. Fraction, Wt. %	7.6	1.3
Vacuum Bottoms, Wt. %	4.3	2.0
Density, Gms/ml @ RT	1.107	1.050
Elemental Analysis		
Carbon, Wt. %	90.17	90.29
Hydrogen, Wt. %	5.94	6.99
Nitrogen, Wt. %	0.83	0.37
Sulfur, Wt. %	0.68	0.15
Oxygen, Wt. % (by difference)	2.38	2.20
H/C Ratio	0.79	0.93
(a) As-received anthracene oil from Crowley Tar & Chemical		
(b) Anthracene oil catalytically hydrogenated in Continuous Process Unit Run 61 at Grand Forks Energy Technology Center		

## Equipment

The UND hot-charge time-sample batch autoclave facility was used for this study. The autoclaves and primary support equipment such as the slurry charge and gas compression equipment are discussed briefly in this section. For a detailed description of the autoclaves, primary support equipment, instrumentation and control equipment, and building modifications, see Appendix A. Appendix A is a copy of a report on the facility prepared by Rindt, Severson, and Souby for presentation at the 88th National AICHE meeting on June 8-12, 1980 at Philadelphia, Pennsylvania.

Figure A-1 is an overall flow diagram of the components of the autoclaves and primary support equipment. Figure A-1 is broken down into seven areas as indicated by the dotted lines.

Figure A-2 is a detailed diagram of Area III, the hot-charge autoclave. This autoclave was used for both hot- and cold-charge runs. The autoclave (AU-101) is a one-gallon stainless steel pressure vessel rated at 5,100 psi at 510°C. It is also equipped with an explosion proof, variable speed, packless, magnetically coupled stirrer. The autoclave (QV-146) used to quench the products instantaneously to room temperature is also in Area III.

Figure A-3 is a detailed diagram of Area II, the slurry charge system for the autoclave. The principal component of this system is the piston accumulator used as the slurry charge vessel (PA-102). The slurry charge vessel is a one-gallon stainless steel accumulator equipped with a movable 4-inch piston with a 10,000 psi rating at room temperature. The seals between the piston and the cylinder walls are made of Viton. The upper portion of the accumulator, above the piston,

contains hydraulic oil. The slurry is placed in the lower portion. When charging the slurry into the autoclave, the hydraulic oil may be pumped to pressures as high as 7,500 psi. This system is capable of charging one gallon of slurry into the autoclave at high temperature and pressure in two minutes.

Figure A-4 is a detailed diagram of Area IV, the gas compression system. The major components of this system are two 2-½ gallon piston accumulators (PA-201, PA-202) rated at 10,000 psi at room temperature. In this system gas is on the upper side of the piston and hydraulic oil on the lower side. Gas is supplied from cylinders shown in Area VII-B of Figure A-1. This system is capable of compressing as much as 100 SCF of gas at tank pressure (up to 2,200 psi) to 7,500 psi.

#### Cold-Charge Runs

The slurry and feed gas were charged into the autoclave and slowly heated to 410°C for the cold-charge runs. At this point the heaters were turned off and the product slurry temperature peaked at 420°C and began to cool down slowly. The product gas was removed at 204°C.

#### Hot-Charge Runs

The hot-charge runs consisted of two types: hot-charge at 320°C, and hot-charge at 360°C. The first type involved charging the feed gas into the autoclave and heating it to 340°C. The slurry was then charged into the autoclave and the temperature dropped below 320°C. The constituents were then heated to 420°C and upon reaching 420°C the products were quenched to room temperature. The residence time (time the constituents are in the reactor) was recorded; it was 29 minutes.

The second type of hot-charge run involved charging the feed gas into the autoclave and heating it to 380°C. The slurry was then charged into the autoclave and the temperature dropped below 360°C. The constituents were heated to 420°C and held at 420°C until the residence time of 29 minutes was attained. The products were then quenched to room temperature.

#### Slurry Preparation

Moisture and ash of the feed coal were determined by American Society for Testing and Materials (ASTM) procedures #D3173 and D3174, respectively. After the moisture content of the coal was determined, the slurry was prepared according to the ratio; Water:MAF lignite: Solvent = 100 grams:200 grams:400 grams. To compensate for losses of slurry during charging, 202 grams and 205 grams of MAF lignite were used for the cold-charge and hot-charge runs, respectively.

#### Addition of Slurry to the Reactor

The slurry was charged directly into the reactor for the cold-charge runs by drawing the slurry through the head with a vacuum. For the hot-charge runs the slurry was charged into the reactor from the charger. Weighed disposable wipes were used to recover any slurry that was not charged into the reactor.

#### Product Slurry Removal

Upon completion of a run, the reactor or quench vessel was opened and the products were transferred into a previously weighed quart can. Weighed disposable wipes were used to recover any remaining material. Small portions of the product slurry were analyzed to determine

solubility in cyclohexane, solubility in THF, moisture content, and ash content. Figure 2 is a flowsheet of a typical run and the subsequent analyses of the products.

#### Product Gas Analysis

The product gas was released from either the reactor or quench vessel through three cold traps located in series. The function of the cold traps was to remove water from the gas. Next, the gas passed through a meter calibrated for 60°F and atmospheric pressure before it was collected in a 15 cubic foot gas sampling bag. Hydrogen sulfide was determined by ASTM method #D2385, and ammonia was determined by the Nessler Method as found in APHA Standard Methods (26). The gas was then analyzed using a Hewlett Packard F and M Scientific 700 Laboratory Chromatograph using Porapak Q and 5A Mole Sieve columns. Carbon dioxide, ethane, and propane were analyzed from the Porapak Q column, and hydrogen, oxygen, nitrogen, methane, and carbon monoxide were analyzed from the Mole Sieve column. The specific gravity of the gas was determined by the Regnault method using a gas density bulb (27).

#### Solubility in Cyclohexane and THF

Approximately 1.0 gram of product slurry was extracted with cyclohexane and filtered through a preweighed 0.5 micron filter (Millipore, type FH). Dry nitrogen gas was used for pressure filtration. The filter cake was washed with cyclohexane until the filtrate ran clear. Approximately 200 ml of cyclohexane was used for the extraction and washing.

The residue from the cyclohexane extraction was extracted with THF by the same procedure. Conversion and oil yields were calculated

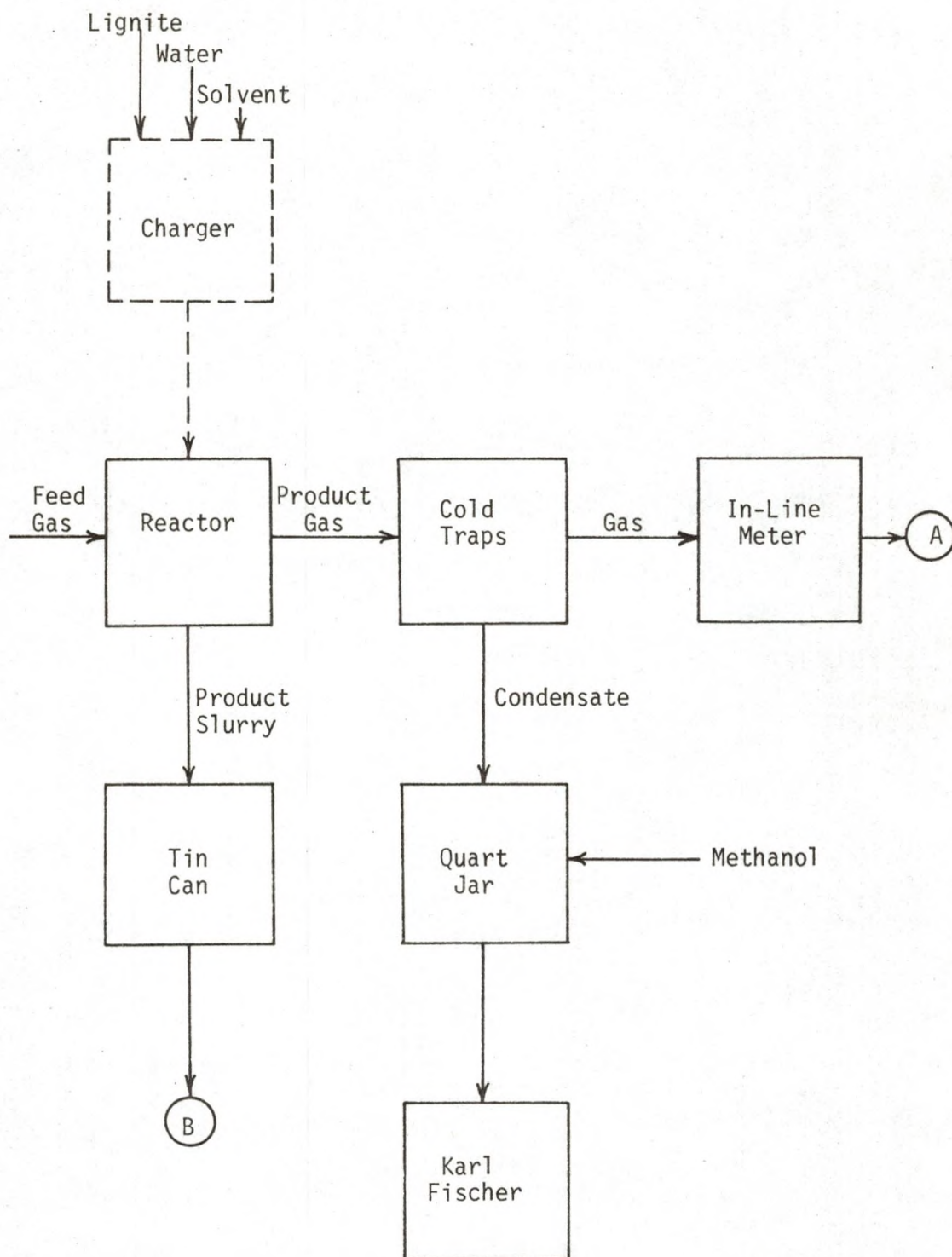
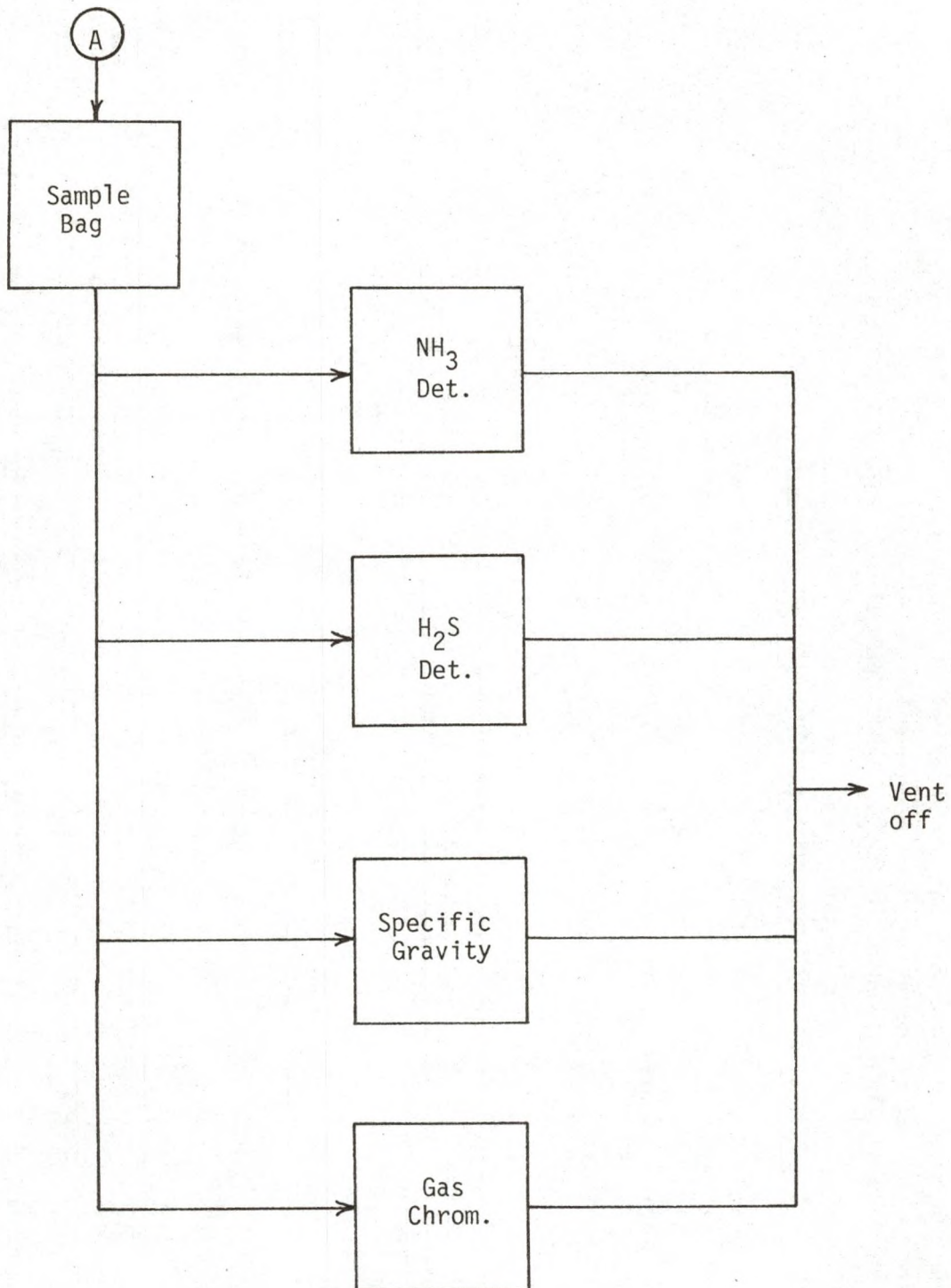
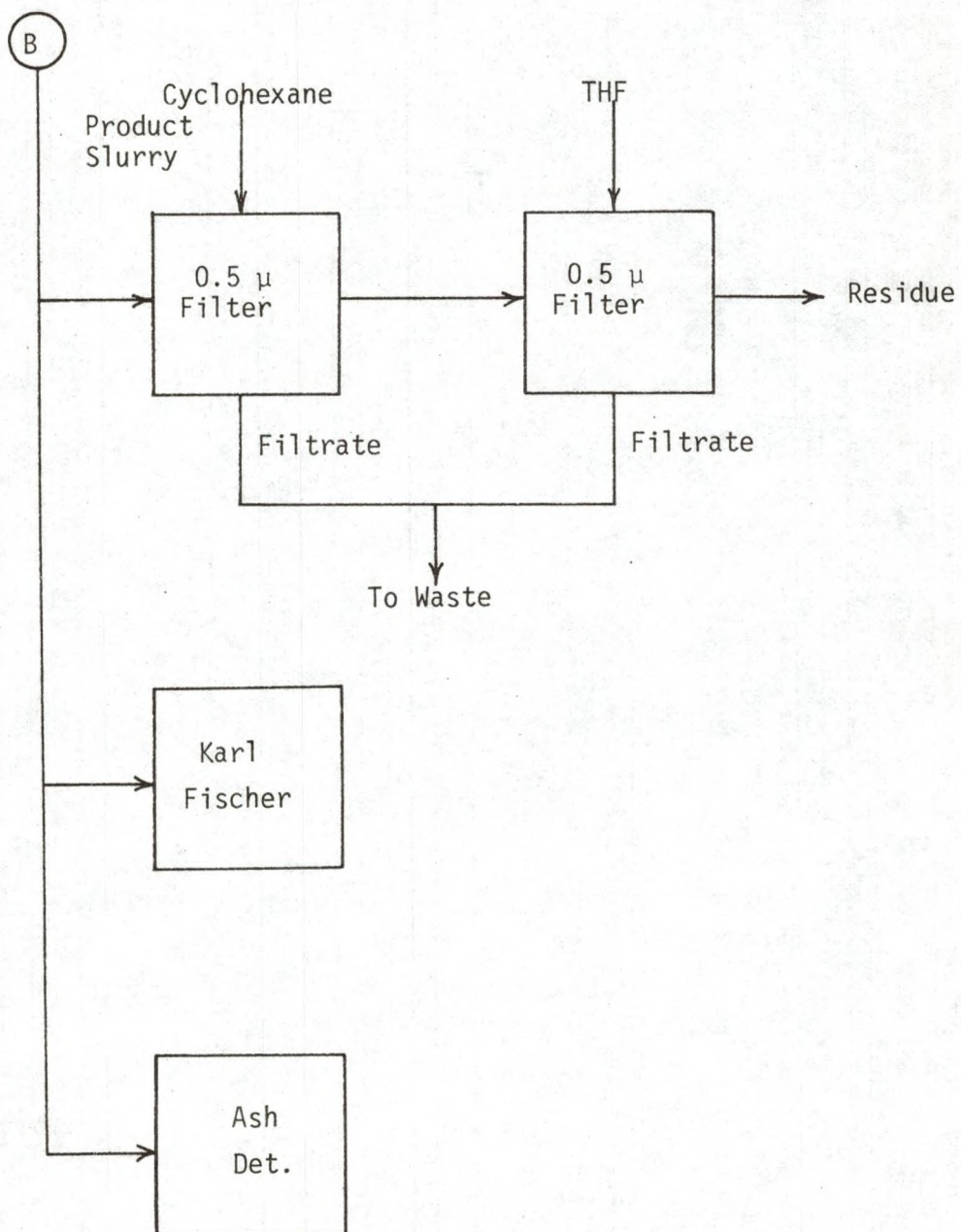


Figure 2. Flowsheet of A Typical Run and Analyses.

Figure 2. Continued.



Figure 2. Continued.

from the raw data as shown in Appendix B.

#### Moisture Determination

The moisture content of the product slurry from the cold-charge runs was expected to be low because the gas was removed from the reactor at 204°C with the moisture leaving the reactor as water vapor. Small portions of the product slurry were removed using a micropipette, weighed, and analyzed for moisture by the Karl Fischer method using a Photovolt Aquatest IV automatic titrator.

The moisture content of the product slurries from the remaining runs was expected to be higher because the products (slurry and gas) were quenched to room temperature before the gas was removed. Because of the expected high moisture content, small portions of the slurry were analyzed by the Karl Fischer method by hand titrating. The automatic titrator was not used because concentrations over 10 percent take a considerable amount of time to titrate.

Methanol was added to the cold trap condensate to dilute it to a 15:1 ratio. Upon complete mixing, small portions of the homogeneous methanol phase containing the moisture were removed and analyzed by the Karl Fischer method using the Photovolt Aquatest IV automatic titrator. The product moisture calculations are shown in Appendix B.

#### Ash Determination

Ash content of the product slurry was determined by ASTM procedure #D3174.

## CHAPTER V

### DISCUSSION OF RESULTS

A summary of the conditions of all runs appears in Appendix C. Computer printouts of the run summaries are included in Appendix D. The material balance closure for run B-4 was only 90.1 percent; therefore, the results were not used in this study. Run B-6 is a repeat of run B-4 and had material balance closure near 100 percent, so run B-6 was used instead of B-4. The computer program used to do all calculations is shown in Appendix E.

Oil yields and conversions ranged from 0.9 percent to 35.7 percent and 66.7 percent to 90.6 percent, respectively, and are consistent with earlier liquefaction work that used a non-basic nitrogen heterocyclic solvent (22). Specifically, these results are consistent with similar work done by Hanson at UND using HAO-61 and synthesis gas in a micro-reactor (28).

#### Hydrogen as Feed Gas

Runs B-1, B-2, and B-3 were conducted using pure hydrogen as feed gas and are summarized in Table 4. Run B-1, a cold-charge run, has higher conversion than either runs B-2 or B-3, hot charge runs at 320 and 360°C, respectively. This is consistent with results obtained by Gorin and co-workers (20) in which it was found that the longer overall reaction time, resulting from the slow heat-up and cool-down periods, has a favorable effect on the extent of conversion. This is further

TABLE 4

## SUMMARY OF RUNS USING HYDROGEN AS FEED GAS

Run Number	Temperature	History	Normalized Net Yields as Weight % of MAF Lignite						
			Water	Oil	SRL	IOM	Gas	Conversion	% Closure
B-1		Cold Charge Heat to 420°C Slow Cooling	10.7	14.2	41.0	11.6	25.3	88.4	98.2
B-2		Hot Charge at 320°C Heat to 420°C Quench Residence time = 29 min.	10.9	0.9	38.8	33.3	16.7	66.7	101.1
B-3		Hot Charge at 360°C Heat to 420°C Hold at 420°C until total time = 29 min. Quench	7.5	19.6	45.4	15.9	11.4	84.1	96.6

demonstrated in run B-2 where the products were quenched to room temperature immediately upon reaching 420°C. In this run the conversion was approximately 75 percent of the conversion attained in run B-1.

Hydrogen becomes more effective in hydrogenating coal as the time at reaction temperature is increased; this is confirmed by the results shown in Table 5. Table 5 shows the product gas compositions for runs B-1, B-2, and B-3; the gas consisted primarily of hydrogen and carbon dioxide. More hydrogen was recovered in the product gas for runs that had shorter residence times at or near 420°C. Concurrently, carbon dioxide production decreased. Experiments using pure hydrogen have shown that some carbon dioxide is split from the lignite during processing (24). This could explain the increase in carbon dioxide production as the residence time at or near 420°C increased.

The SRL yields for all three runs are similar, while the oil yields for runs B-1 and B-3 are approximately equal and considerably larger than the oil yield for run B-2. Another similarity between runs B-1 and B-3 is seen in the IOM yields, which are again nearly equal but less than that observed for run B-2. This suggests that the IOM may be converted to SRL, which in turn is converted to oil during longer times at or near 420°C. Another possibility, and probably more likely because the SRL yields are similar, is that the IOM is converted directly to oils. Two statistically similar kinetic models were developed by Culpon (26) to describe the liquefaction of North Dakota lignite in a continuous stirred tank reactor. In his first model, Model B, coal is converted to asphaltenes (SRL) or directly to oils. Asphaltenes can be further converted to oils. In his second model, Model C, coal is either converted to asphaltenes or to oils. Culpon reports that more

TABLE 5  
PRODUCT GAS COMPOSITION USING HYDROGEN AS FEED GAS

Run Number	CO <sub>2</sub>	C <sub>2</sub> H <sub>6</sub>	C <sub>3</sub> H <sub>8</sub>	H <sub>2</sub>	CH <sub>4</sub>	CO	H <sub>2</sub> S	NH <sub>3</sub>
B-1	8.93	0.40	0.19	86.44	1.06	1.88	0.16	0.02
B-2	7.04	0.06	0.01	89.75	0.39	0.22	0.15	0.01
B-3	4.50	0.52	0.00	92.02	0.86	0.15	0.19	0.04

kinetic data is needed on the rate of conversion of the asphaltenes to oils. Therefore, there may be shortcomings to both models and no definite conclusion can be drawn for the above observation.

#### Synthesis Gas as Feed Gas

Runs B-5 and B-6 were conducted using synthesis gas as feed gas. A summary of these runs is shown in Table 6. It can be seen that SRL and IOM values for both runs are approximately the same. Run B-6, a cold-charge run, has slightly better conversion and higher gas and oil yields. Higher conversion and greater oil yield for the cold-charge run were expected because earlier batch studies using synthesis gas have shown similar trends (30).

The product gases consisted primarily of carbon monoxide, hydrogen, and a significant amount of carbon dioxide, as shown in Table 7. The greater yield of carbon dioxide agrees with prior results obtained when hydrotreating coal using synthesis gas, where it was determined that large amounts of the oxygen in the coal were removed as carbon dioxide (31).

#### Synthesis Gas Versus Hydrogen as Feed Gas

The cold-charge run using synthesis gas, run B-6, had higher conversion and oil production than run B-1 where hydrogen was used as the feed gas. This trend was also observed between runs B-5 and B-3, hot-charge runs at 360°C, using synthesis gas and hydrogen as feed gases, respectively. A comparison of hydrogen and carbon monoxide for liquefaction has shown that carbon monoxide is selective to reduction of the carbonyl group, whereas hydrogen causes more cracking. As mentioned earlier, the high activity of carbon monoxide for reducing carbonyl

TABLE 6

## SUMMARY OF RUNS USING SYNTHESIS GAS AS FEED GAS

Run Number	Temperature History	Normalized Net Yields as Weight % of MAF Lignite						
		Water	Oil	SRL	IOM	Gas	Conversion	% Closure
B-5	Hot Charge at 360°C Heat to 420°C Hold at 420°C until total time = 29 min. Quench	-18.9	27.2	45.6	10.8	34.7	89.2	101.5
B-6	Cold Charge Heat to 420°C Slow Cooling	-18.9	35.7	49.6	9.4	27.6	90.6	98.7



TABLE 7  
PRODUCT GAS COMPOSITION USING SYNTHESIS GAS AS FEED GAS

Run Number	CO <sub>2</sub>	C <sub>2</sub> H <sub>6</sub>	C <sub>3</sub> H <sub>8</sub>	H <sub>2</sub>	CH <sub>4</sub>	CO	H <sub>2</sub> S	NH <sub>3</sub>
B-5	29.71	0.53	0.00	41.81	0.70	26.45	0.18	0.02
B-6	22.98	0.27	0.10	46.13	0.73	28.87	0.20	0.02

groups is believed to be the reason that low-rank coals are liquefied more readily in the presence of carbon monoxide than hydrogen.

Gas yield was similar for both run B-6 and run B-1; however, run B-5 had three times as much gas produced as did run B-3. It was the large amount of carbon dioxide produced in run B-5 that caused this significant difference.

Water yields as shown in Tables 4 and 6 are positive for runs using hydrogen and negative for runs using synthesis gas. This implies that water is produced in the former runs and depleted in the latter. Water was expected to react with carbon monoxide from the synthesis gas and form carbon dioxide and hydrogen via the water-gas shift reaction.

## CHAPTER VI

### CONCLUSIONS AND RECOMMENDATIONS

#### Conclusions

1. Higher conversions and greater oil yields were obtained when using synthesis gas as compared to pure hydrogen. This is consistent with previous work and occurred because of the presence of carbon monoxide in the feed gas.
2. Longer times at or near the reaction temperature (380°-420°C) resulted in higher conversions and greater oil yields under the conditions used.
3. Synthesis gas produced more carbon dioxide than similar runs using hydrogen, resulting in a higher gas yield.
4. Oil yields and conversions ranged from 0.9 percent to 35.7 percent and 66.7 percent to 90.6 percent, respectively.
5. SRL yields were similar for all runs.

#### Recommendations

1. A hot-charge run should be performed to determine the identity of reactor contents at residence time equal to zero. As mentioned earlier, for a hot-charge run at 320°C the feed gas was heated to approximately 340°C before the slurry was charged. Upon slurry charging the temperature dropped below 320°C and it took several minutes before the temperature was back up to 320°C and timing of the residence period began. A run conducted where the contents of the autoclave are quenched as soon as they reach 320°C and

subsequently analyzed would determine if there are products formed before timing begins. If products are formed, these could be subtracted from the final results to give a better overall picture of what is occurring during the residence time only.

2. A hot charge run could be conducted at 420°C using synthesis gas and the results compared with those obtained for the hot-charge run at 360°C.

APPENDICES

APPENDIX A

UNIVERSITY OF NORTH DAKOTA HOT-CHARGE TIME-SAMPLE  
BATCH AUTOCLAVE FACILITY

UND HOT-CHARGE TIME-SAMPLE BATCH AUTOCLAVE FACILITY

By

Rindt, J. R., Severson, D. E., and Souby, A. M.

University of North Dakota

For Presentation at the 88th National AICHE

June 8-12, 1980

Philadelphia, Pennsylvania

## UND HOT-CHARGE TIME-SAMPLE BATCH AUTOCLAVE FACILITY

The University of North Dakota (UND) Chemical Engineering Department is performing contract research with the Grand Forks Energy Technology Center (GFETC) on the liquefaction of low rank coals. One task under this contract is the design and construction of a versatile two autoclave system, with one autoclave run in a mode suitable for obtaining accurate material balances (charged cold) and the other for accurate kinetics data (charged hot). The autoclave facility is designed to meet current federal health and safety guidelines.

When operating to obtain accurate material balances (with the cold charge autoclave), the reactant materials, gas, solvent, and coal are charged to the autoclave at room temperature. The autoclave is then heated to the desired reaction temperature, held there for the desired reaction time, allowed to cool, and all products collected. This procedure allows 95 to 99 percent product recovery, and thus is quite useful in obtaining material balance data.<sup>(1)</sup>

The hot charge autoclave, operated to obtain kinetic data, is equipped so that the reactant materials may be charged rapidly into the preheated autoclave. This allows the reactants to reach operating temperatures in a few minutes. Both vapor and liquid phase reactants are sampled at intervals during the reaction to obtain data for kinetic studies. A hot charge, 1-liter autoclave facility operated at GFETC has resulted in product recoveries of 90 to 92 percent. This recovery is less than the 95 to 99 percent recovery reported for a cold charge



autoclave facility operated at UND. The lower recovery for the hot charge system is due to unavoidable losses during sampling.

The new UND autoclave system was designed to minimize sampling losses and enhance charging reliability with improvements based on past autoclave experience. The UND autoclave facility will be discussed in terms of three major elements, as follows:

1. The autoclaves and primary support equipment such as the slurry charge and gas compression equipment,
2. The instrumentation and control equipment which includes temperature, pressure, gas and liquid phase flow measurement systems, and,
3. The building modification primarily related to meeting requirements suggested by current federal health and safety guidelines, as listed in the Fossil Energy Program, Environment and Safety Program.

The remainder of this paper will describe the features and innovations of the UND Batch Autoclave Facility.

Figure 1 is an overall flow diagram of the components of the autoclaves and primary support equipment. Figure 1 is broken down into seven areas as indicated by the dotted lines.

Figure 2 is a detailed diagram of Area III, the hot charge autoclave. The autoclave (AU-101) is a one-gallon stainless steel pressure vessel rated at 5,100 psi (35,000 kPa) at 510°C. It is also equipped with an explosion proof, variable speed, packless, magnetically coupled stirrer. The autoclaves are equipped with flush valves at the bottom for liquid phase time sampling. When closed, the valves leave no pockets or dead spots on the inside of the autoclave into which reactant materials

may accumulate or settle. This feature should provide reliable liquid sampling data with a minimum of sampling losses since fewer purge samples will be required.

In the one-liter time-sampled autoclave at GFETC, a minimum of 400-gram slurry charge is used to limit change in reactor contents during the run and allow reasonable material balances. This leaves so little remaining space in the reactor that the reaction is depleted of gas. Use of the one-gallon reactor will permit much higher gas-slurry ratio with adequate material balances, thus providing a substantially lengthened time during which the vapor phase reactant is not the limiting reactant. Also, the higher gas volume of the one gallon autoclave reduces the effect of vapor phase time sampling on pressure. The size of each of the time samples is approximately the same as that for the one-liter autoclave, while the reactive vapor volume increases significantly. The time sampling in the one-gallon autoclave produces a smaller pressure loss, resulting in less change in pressure during the reaction time.

Figure 3 is a detailed diagram of Area II, the slurry charge system for the hot charge autoclave. Principal components of this system are the low pressure slurry pump (PD-351) and the piston accumulator used as the slurry charge vessel (PA-102). The low pressure slurry pump is a double diaphragm, positive displacement, variable flow, metering pump, which charges the slurry into the slurry charge vessel. The slurry charge vessel is a one-gallon stainless steel accumulator equipped with a movable 4-inch piston with a 10,000 psi rating (69,000 kPa) at room temperature. The seals between the piston and the cylinder walls are made of Viton. The low-pressure slurry pump charges slurry to the accumulator below the piston. The upper portion of the accumulator

contains hydraulic oil. When charging the slurry into the autoclave, the hydraulic oil may be pumped up to pressures as high as 7,500 psi (52,000 kPa). Two valves between the charge vessel and the autoclave are used to control the charge amount and rate. This highly flexible positive displacement feed system is capable of charging one gallon of slurry into the autoclave at high temperature and pressure in two minutes. The system is also capable of injecting small increments of slurry or other liquid reactants during the reaction. The entire slurry system can be flushed and recharged to allow different materials to be added during a run, e.g., a catalyst may be added after a run is started. This system has several advantages over the use of a slurry pump for direct charging to an autoclave. These include low initial investment as compared to a slurry pump capable of metering slurry at pressure of 7,500 psi (52,000 kPa), improved reproducibility of quantity and composition of slurry charged, and positive displacement of the slurry which avoids the losses due to adherence to charger walls.

Figure 4 is a detailed diagram of Area IV, the gas compression system. The major components of this system are two 2- $\frac{1}{2}$  gallon piston accumulators (PA-201, PA-202) rated at 10,000 psi (69,000 kPa) at room temperature. In this system gas is on the upper side of the piston and hydraulic oil on the lower side. Gas is supplied from cylinders shown in Area VII-B of Figure 1. This system is capable of compressing as much as (100 SCF) of gas at tank pressure (up to 2,200 psi) (15,000 kPa) to 7,500 psi (52,000 kPa). This system enjoys the same versatility as the slurry charge system with the additional advantage of continuous feed capability when the two accumulators are used alternately.

Figure 5 is a detailed diagram of Area I, the high pressure hydraulic oil supply system. The major component of the system is the high-pressure, positive-displacement, packed-plunger, metering hydraulic pump (PD-301) which is capable of pumping hydraulic oil at 30 gallons/hour and 7,500 psi (52,000 kPa). It supplies oil to both the slurry charge system and the gas compressor system at a rate which allows both slurry and gas to be charged simultaneously. In addition, the hydraulic pump and slurry pump are driven by the same system to save space and expense. As both pumps have metering capabilities, they may be used simultaneously or independently in the operation of the gas compressor and slurry charge systems. The combination of these systems has greatly reduced initial investment, parts inventory, and maintenance expense, while affording a high degree of flexibility and system independence.

The cold charge autoclave, shown in Figure 1 as Area VII-A, is supplied by the same gas supply and compression system as the hot charge facility.

The second major element of the UND autoclave facility is the instrumentation and controls system. Figure 6 is a schematic diagram of the instrumentation for both the hot and cold charge autoclave. Because of the safety requirements, which will be discussed later, all pressure and temperature measurement are remote. Type J thermocouples are used for temperature signal generation and pressure transducers with 0-20 mA output for pressure signals. Four recorders and four digital displays show temperature and pressures. Autoclave temperature and pressure are recorded with continuous pen recorders, while other temperature and pressure data are recorded on multipoint dot recorders.

All recorders have one second full scale response times. The multipoint recorders have a skip function enabling any point or points to be eliminated during any given run. Digital display of points not requiring recording is on four 5-place multi-display pressure and temperature indicators.

The quality of slurry or gas charged is determined by measuring the hydraulic fluid displaced during the charge procedure. The hydraulic oil, under constant pressure, is measured by two independent methods, one being by observation of a sight glass on the seven-gallon oil reservoir and the other by means of a turbine flow meter equipped with a flow rate indicator and totalizer. The gas charged may also be measured with a gas phase turbine flow meter with a temperature and pressure compensated flow rate indicator and totalizer.

The temperature programmed gas chromatograph (GC) used for analyzing vapor samples is equipped with two columns with column packing capable of separating  $H_2O$ ,  $CO$ ,  $CO_2$ , and light hydrocarbons. The analysis of  $H_2O$  by the gas chromatograph, not usually incorporated into autoclave gas sampling systems, required that the sample collection system be held at a temperature above the boiling point of  $H_2O$  at sample pressure. A gas sample storage system is provided to improve data collection versatility. Figure 7 is a drawing of the GC sample loops and hot box. The sample loops are also shown diagrammatically in Figure 1, Area IV. The storage system provides for storage of up to 10 samples, which may be collected at sampling intervals as short as 20 seconds, for a time long enough for all 10 samples to be analyzed.

The GC sample loop storage system, as well as the vapor and liquid phase sampling systems, have several simultaneous timed operations, all

of which can be handled by a programmable controller. The programmable controller provides reliable, reproducible timing for sequenced operations. It can be programmed to operate on a time table in increments as small as 0.01 seconds, and thus essentially eliminates variability for the timed sample events. The controller is also capable of controlling the GC operation, the slurry charge system, and the gas charge system, thus further standardizing operations.

A significant effort in the facility preparation program is the building modification for compliance with federal health and safety guidelines. Figure 8 is a floor plan of the portion of the UND building housing the project. The areas of major building modifications are cells 1 through 4, the lunchroom, and locker rooms 1 through 4.

One guideline employed at GFETC is that direct personnel exposure to high pressure equipment be limited to a vessel at 100°C or less and 3,000 psi (21,000 kPa) or less.<sup>(2)</sup> As the UND autoclave system is to be operated in excess of these limitations, special enclosures are required. The enclosures are cells 1 through 4 of Figure 8. Figure 9 is a detailed diagram of the barricade structure. The autoclave barricade system is set up to allow the cleaning and maintenance of one autoclave during the operation of the other. Cell 1 contains the hydraulic and slurry pumps; cell 2, the hot charge autoclave; cell 3, the gas compression equipment and GC sample loops; and cell 4, the cold charge autoclave. Each cell has a blast window which opens during an explosion, protecting operators behind the opposing barricade from the consequences of dangerous pressure buildups should an explosion occur. Beyond the blast windows are blast mats woven of  $\frac{1}{2}$ -inch steel cables. The barricade itself is constructed of  $\frac{1}{2}$ " Cor-ten steel plate. The

barricade and blast mats have been calculated to provide protection against shrapnel.

Federal health and safety guidelines<sup>(3)</sup> also specify conditions for extended work in an environment containing coal liquefaction products. Personnel in potential contact with coal liquefaction products are to be supplied with clean work clothing at the start of each work day and must properly dispose of them at the close of each work day. Further requirements include separate change facilities to isolate the area for changing work clothing from that for street clothing. These guidelines require the presence of two locker rooms if only one sex is employed and four if both male and female employees are present. Locker rooms 1 through 4 of Figure 8 are the change facilities planned for the UND autoclave installation.

There are also guidelines for break and lunch times during the work day. A break room isolate from the bulk of the work area, equipped with wash facilities, is required to provide a safe area in which food may be consumed. The lunchroom, shown in Figure 8, meets these requirements.

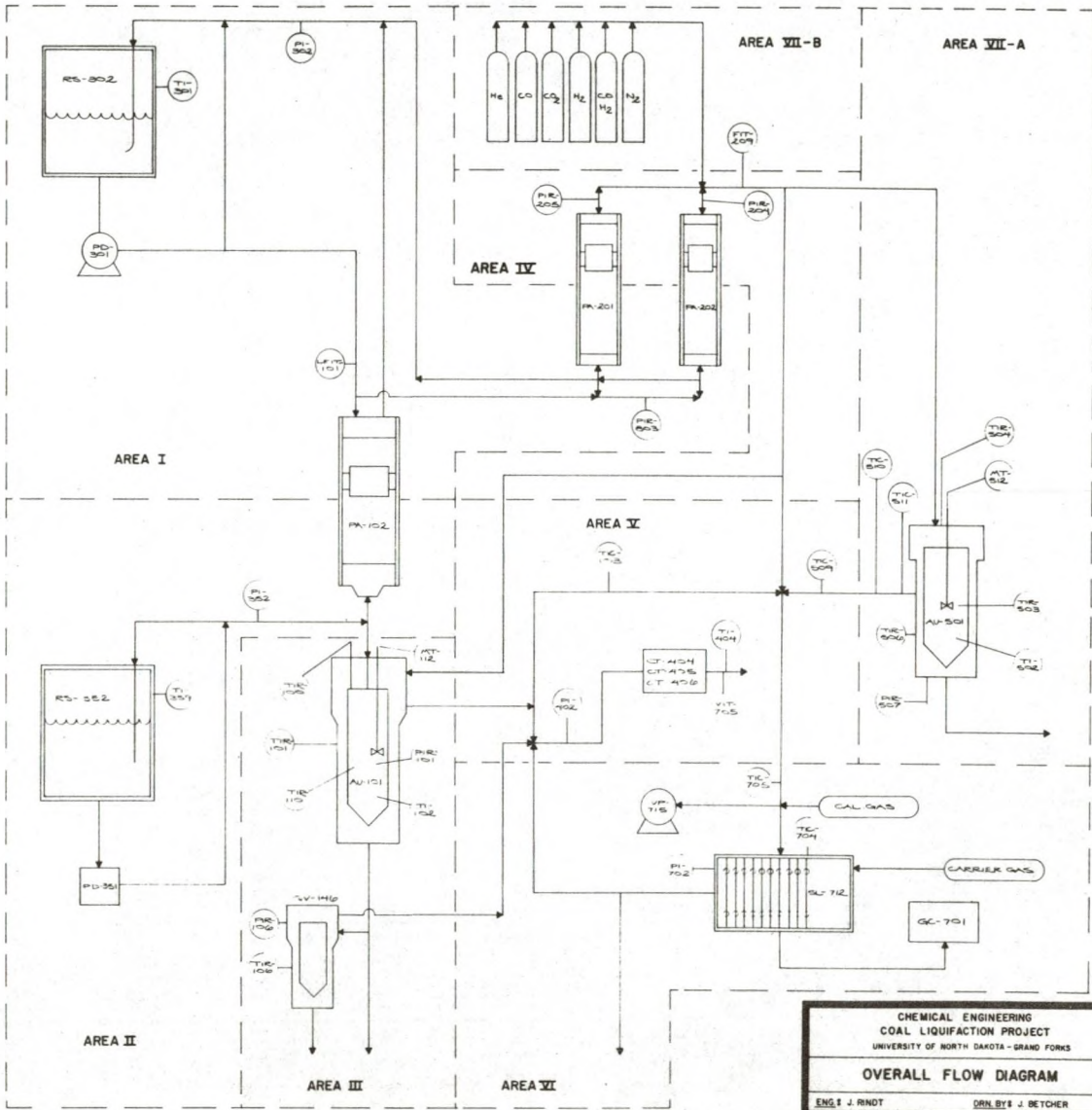
Ventilation requirements are also quite rigorous. Twenty changes of air per hour are suggested in the barricaded area with 10 changes per hour in the work and locker room areas.

The UND autoclave facility has enough flexibility to be useful for many different research programs and is expected to supply extensive data from studies on the liquefaction of low-rank western coals.

AB - ACRYLATE	FIR - PRESSURE INDICATOR RECORDER
AC - CHECKER GAS CLEANER APPARATUS	FR - PRESSURE RELIEF VALVE
BA - BEATER ACCUMULATOR	RV - ROUGHED VALVE
CP - COLLECTOR PUMP	OV - OVEN VESSEL
CS - COLD TRAP	RS - RESSURFER
CV - CHECK VALVE	RSI - REMOVE SAMPLE INLET
DV - DOUBLE VALVE	RV - PRESSURE REGULATOR VALVE
FI - FLOW INDICATOR	RSV - REMOVE VACUUM SOURCE
FIV - FLOW INDICATOR VORALIZER	SB - SOURCE CHECK BYPASS VALVE
FL - FLETER	SC - SOURCE CHECK VALVE
FM - FINE METERING VALVE	SL - SAMPLE LOOP
FN - FLOW VALVE	SP - SOURCE FLOW ACCUMULATOR
GC - GAS CHROMATOGRAPH	ST - SAMPLE TRAY
GV - GAS SAMPLING VALVE	SV - SHUTOFF VALVE
HF - MAGNETIC FLOWMETER PICKUP	TI - TEMPERATURE INDICATOR
PA - PISTON ACCUMULATOR	TIC - TEMPERATURE INDICATOR CONTROLLER
PD - POSITIVE DISPLACEMENT PUMP	TIA - TEMPERATURE INDICATOR RECORDER
PG - BACK PRESSURE REG. VALVE	
PI - PRESSURE INDICATOR	

PIPE SCHEDULE		
①	3/16" O.D.	20,000 psi (S.S.)
②	1/8" O.D.	20,000 psi (S.S.)
③	1/4" O.D.	Plastic Instrument Tubing
④	9/16" O.D.	60,000 psi (S.S.)
⑤	1/4" O.D.	60,000 psi (S.S.)
⑥	3/8" O.D.	60,000 psi (S.S.)
⑦	1/8" O.D.	11,000 psi (S.S.)
⑧	1/4" O.D.	11,000 psi (S.S.)

FITTING SCHEDULE		
1	30922564	Bullhead (9/16" - 20K) A.E.
2	3092648	Adaptor (9/16" 20K + 3/8" 20K) A.E.
3	309286	Adaptor (1/4" NPTM + 9/16" 20K) A.E.
4	309286	Adaptor (1/4" NPTM + 9/16" 20K) A.E.
5	309286	Adaptor (1/4" NPTM + 9/16" 20K) A.E.
6	3092128	Adaptor (3/4" NPTT + 9/16" 20K) A.E.
7	309286	Adaptor (1/4" NPTM + 3/8" 20K) A.E.
8	309286	Adaptor (9/16" 20K + 3/8" 20K) A.E.
9	309286	Adaptor (1" NPTM + 9/16" 20K) A.E.
10	3092566	Bullhead (3/8" 20K) A.E.
11	309286	Adaptor (3/4" NPTM + 3/8" 20K) A.E.
12	309286	Adaptor (9/16" 60K + 3/8" 20K) A.E.
13	309286	Adaptor (9/16" 60K + 3/4" 60K) A.E.
14	309286	Adaptor (1" NPTT + 3/8" 20K) A.E.
15	309286	Adaptor (1/4" NPTM + 3/8" 20K) A.E.
16	3092433	Bullhead (1/2" 60K) A.E.
17	309286	Adaptor (1" NPTM + 3/8" 60K) A.E.
18	309286	Adaptor (1/4" 60K + 3/8" 60K) A.E.
19	309286	Adaptor (1/4" NPTM + 1/4" 60K) A.E.
20	309286	Adaptor (1/4" 60K + 3/8" 60K) A.E.
21	309286	Adaptor (1/4" NPTM + 1/4" 60K) A.E.
22	309286	Adaptor (1/4" NPTM + 1/4" 60K) A.E.
23	309286	Adaptor (1/4" NPTM + 1/4" 60K) A.E.
24	309286	Adaptor (1/4" NPTM + 1/4" 10K) A.E.
25	309286	Adaptor (1/4" NPTM + 1/4" 10K) A.E.
26	309286	Adaptor (1/4" NPTM + 1/4" 11K) A.E.
27	309286	Adaptor (1/4" NPTM + 1/4" 11K) A.E.
28	309286	Adaptor (1/4" NPTM + 1/4" 11K) A.E.
29	309286	Adaptor (1/4" NPTM + 1/4" 11K) A.E.
30	309286	Adaptor (1/4" NPTM + 1/4" 11K) A.E.
31	3092211	Bullhead (1/8" 11K) A.E.
32	309286	Adaptor (1/4" NPTM + 1/4" 10K) A.E.
33	309286	Adaptor (1/4" NPTM + 1/4" 11K) A.E.
34	309286	Adaptor (1/4" NPTM + 1/4" 11K) A.E.
35	309286	Adaptor (1/4" NPTM + 1/4" 11K) A.E.
36	309286	Adaptor (1/4" NPTM + 1/4" 11K) A.E.



CHEMICAL ENGINEERING  
COAL LIQUIFICATION PROJECT  
UNIVERSITY OF NORTH DAKOTA - GRAND FORKS

**OVERALL FLOW DIAGRAM**

ENG. J. RINDT	DRN. BY J. BETCHER
DATE: 11-7-79	DRG. NO. 1 F79-TD-4-10

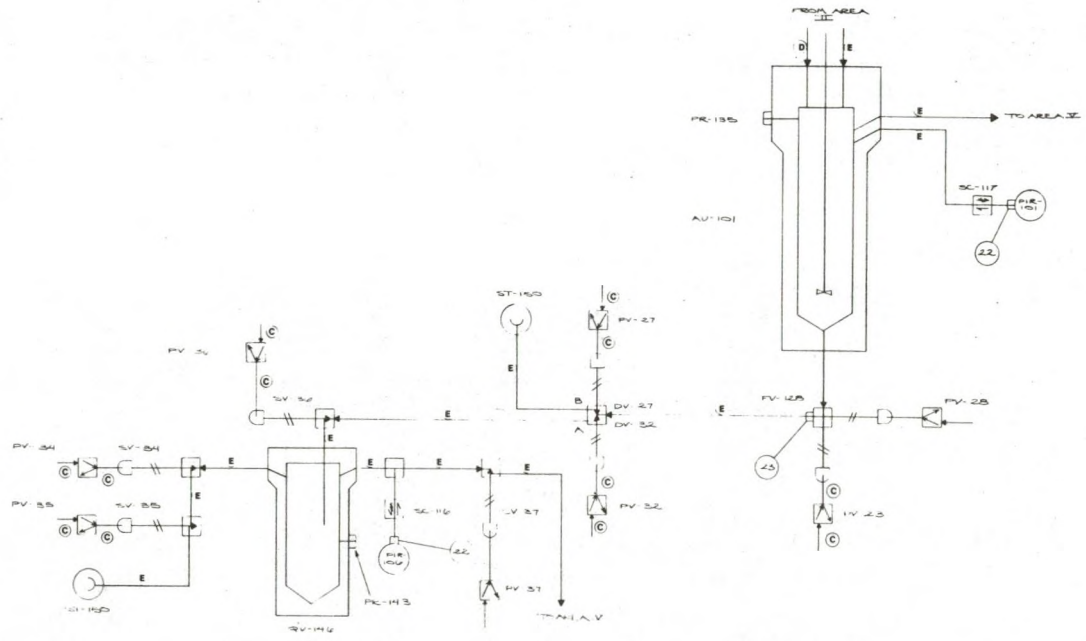
Figure A-1. Overall Flow Diagram.



AD - ADVOLVANT	FIR - PRESSURE INDICATOR RECORDER
AG - GASEOUS GAS CLEANUP APPARATUS	FR - PRESSURE RELIEF VALVE
BA - BLASTER ACCUMULATOR	FV - SOLENOID VALVE
CB - COLLECTOR POST	CV - CHECK VALVE
CF - COLD TRAP	CS - CONDENSER
CV - CHECK VALVE	DS - DRAIN
DV - DRAIN VALVE	FI - FLOW INDICATOR
FI - FLOW INDICATOR	FIW - FLOW INDICATOR W/WEIGHT
FL - FILTER	FM - FLOW MEASUREMENT VALVE
FR - FLOW MEASUREMENT VALVE	FV - FLOW VALVE
GC - GAS CHROMATOGRAPH	GS - GAS SAMPLING VALVE
GD - GAS SAMPLING VALVE	HT - HEAT EXCHANGER
HP - MAGNETIC FLOWMETER PICKUP	HT - HEAT EXCHANGER
IA - TYPING INDICATOR	HTC - TEMPERATURE INDICATOR CONTROLLER
IP - POSITIVE DISPLACEMENT PUMP	IR - TEMPERATURE INDICATOR RECORDER
IS - BACK PRESSURE REG. VALVE	
IT - PRESSURE INDICATOR	

PIPE SCHEDULE		
(A)	1/2" O.D.	20,000 psi (U.S.)
(B)	3/8" O.D.	20,000 psi (U.S.)
(C)	1/4" O.D.	Plastic Instrument Tubing
(D)	3/16" O.D.	60,000 psi (U.S.)
(E)	1/4" O.D.	60,000 psi (U.S.)
(F)	3/8" O.D.	60,000 psi (U.S.)
(G)	1/2" O.D.	11,000 psi (U.S.)
(H)	1" O.D.	11,000 psi (U.S.)

FITTING SCHEDULE		
1	20927964	Bulkhead 19/16" - 2061 A.E.
2	20929564	Adapter 19/16" 206 + 1/8" 2061 A.E.
3	206396	Adapter 1 1/4" NPTM - 9/16" 2061 A.E.
4	206396	Adapter 1 1/4" NPTM - 9/16" 2061 A.E.
5	2061296	Adapter 1 1/4" NPTM - 9/16" 2061 A.E.
6	2061296	Adapter 1 1/4" NPTM - 9/16" 2061 A.E.
7	206396	Adapter 1 1/4" NPTM - 9/16" 2061 A.E.
8	206396	Adapter 1 1/4" NPTM - 9/16" 2061 A.E.
9	2061296	Adapter 1 1/4" NPTM - 9/16" 2061 A.E.
10	20927964	Bulkhead 1 3/8" 2061 A.E.
11	2061296	Adapter 1 3/8" NPTM - 3/8" 2061 A.E.
12	206396	Adapter 1 1/2" 60K - 3/8" 2061 A.E.
13	20929564	Adapter 1 1/2" 60K - 3/8" 2061 A.E.
14	2061296	Adapter 1 1/2" NPTM - 3/8" 2061 A.E.
15	2061296	Adapter 1 1/2" NPTM - 3/8" 2061 A.E.
16	2092443	Bulkhead 1 1/4" 4061 A.E.
17	2061296	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
18	2064683	Adapter 1 1/4" 40K - 3/8" 4061 A.E.
19	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
20	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
21	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
22	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
23	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
24	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
25	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
26	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
27	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
28	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
29	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
30	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
31	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
32	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
33	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
34	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
35	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.
36	2064683	Adapter 1 1/4" NPTM - 1/4" 4061 A.E.



CHEMICAL ENGINEERING  
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**AREA III**

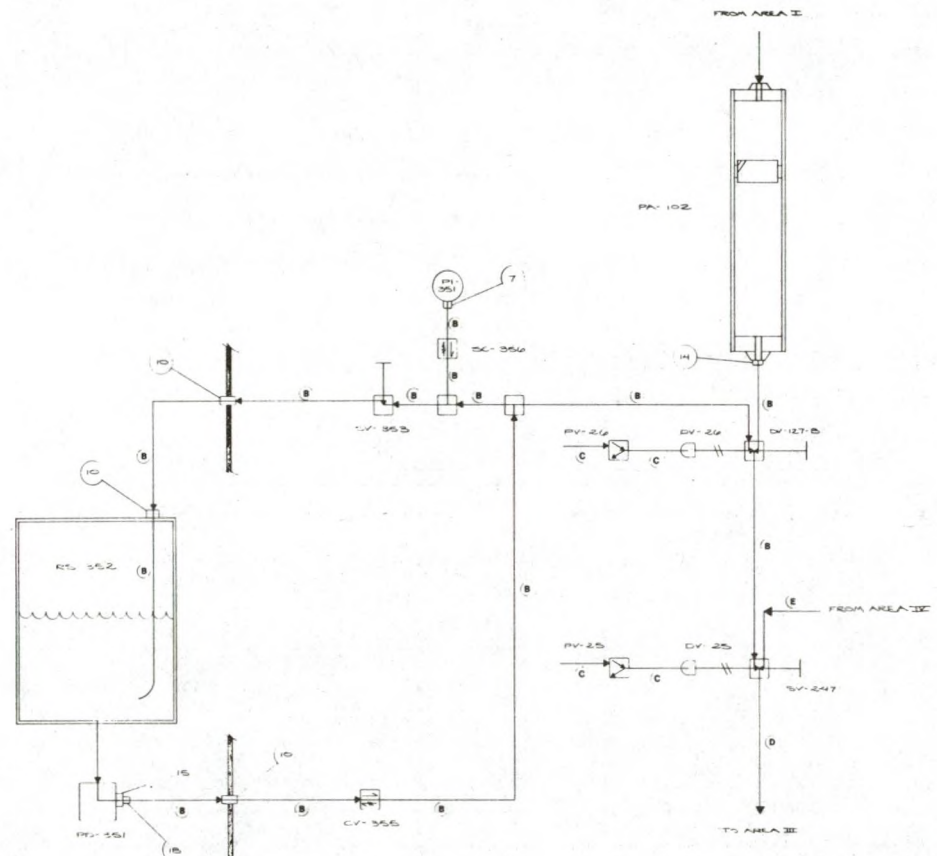
ENG. J. RINDT                      DRN. BY J. BETCHER  
DATE: 11-7-79                      ORG. NO. F79-TO-4-13

Figure A-2. Detail diagram of the UND hot charge autoclave components.

AG - AUTOCLAVE	FTS - PRESSURE INDICATOR RECORDER
AC - CARRIER GAS CLEANUP APPARATUS	FR - PRESSURE RELIEF VALVE
AD - HEATER ACCUMULATOR	FS - SOLIDUS VALVE
AE - COLLECTOR PUMP	FV - FLOW VALVE
AF - CHECK VALVE	FZ - FLOW INDICATOR
AG - CHECK VALVE	GA - FLOW INDICATOR TOTALIZER
AI - DOUBLE VALVE	FL - FILTER
AL - FLOW INDICATOR	FM - FINE METERING VALVE
AM - FLOW INDICATOR TOTALIZER	FN - FLOW VALVE
AN - FINE METERING VALVE	FO - GAS CHROMATOGRAPH
AO - FLOW VALVE	FP - GAS SAMPLING VALVE
AP - GAS CHROMATOGRAPH	FR - ISOMETRIC THERMISTOR PICKUP
AR - GAS SAMPLING VALVE	FS - FLOW ACCUMULATOR
AS - ISOMETRIC THERMISTOR PICKUP	FT - FLOW ACCUMULATOR
AT - FLOW ACCUMULATOR	FA - FLOW ACCUMULATOR
AV - POSITIVE DISPLACEMENT PUMP	FB - POSITIVE DISPLACEMENT PUMP
AW - BACK PRESSURE REG. VALVE	FC - PRESSURE INDICATOR
AX - PRESSURE INDICATOR	

PIPE SCHEDULE		
A	9/16" O.D.	20,000 psi (S.S.)
B	3/8" O.D.	20,000 psi (S.S.)
C	1/4" O.D.	Plastic Instrument Tubing
D	9/16" O.D.	60,000 psi (S.S.)
E	1/4" O.D.	60,000 psi (S.S.)
F	3/8" O.D.	60,000 psi (S.S.)
G	1/8" O.D.	11,000 psi (S.S.)
H	1/4" O.D.	11,000 psi (S.S.)

FITTING SCHEDULE		
1	SOB7254	Bulkhead 19/16" - 20K1 A.E.
2	SOB564	Adaptor 19/16" 20K + 3/8" 20K1 A.E.
3	SOB565	Adaptor 1/4" NPTM - 9/16" 20K1 A.E.
4	SOB566	Adaptor 1/4" NPTM - 9/16" 20K1 A.E.
5	SOB567	Adaptor 1/4" NPTM - 9/16" 20K1 A.E.
6	SOB568	Adaptor 1/4" NPTM - 9/16" 20K1 A.E.
7	SOB569	Adaptor 1/4" NPTM - 3/8" 20K1 A.E.
8	SOB570	Adaptor 19/16" 20K + 3/8" 20K1 A.E.
9	SOB571	Adaptor 1" NPTM - 9/16" 20K1 A.E.
10	SOB7564	Bulkhead 3/8" 20K1 A.E.
11	SOB7565	Adaptor 3/4" NPTM - 3/8" 20K1 A.E.
12	SOB568	Adaptor 19/16" 20K + 3/8" 20K1 A.E.
13	SOB569	Adaptor 19/16" 20K + 3/8" 20K1 A.E.
14	SOB570	Adaptor 1" NPTM - 3/8" 20K1 A.E.
15	SOB571	Adaptor 3/4" NPTM - 3/8" 20K1 A.E.
16	SOB7443	Bulkhead 1/4" 20K1 A.E.
17	SOB569	Adaptor 1" NPTM - 3/8" 20K1 A.E.
18	SOB570	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
19	SOB571	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
20	SOB572	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
21	SOB573	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
22	SOB574	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
23	SOB575	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
24	SOB576	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
25	SOB577	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
26	SOB578	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
27	SOB579	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
28	SOB580	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
29	SOB581	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
30	SOB582	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
31	SOB583	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
32	SOB584	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
33	SOB585	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
34	SOB586	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
35	SOB587	Adaptor 1/4" 20K + 3/8" 20K1 A.E.
36	SOB588	Adaptor 1/4" 20K + 3/8" 20K1 A.E.



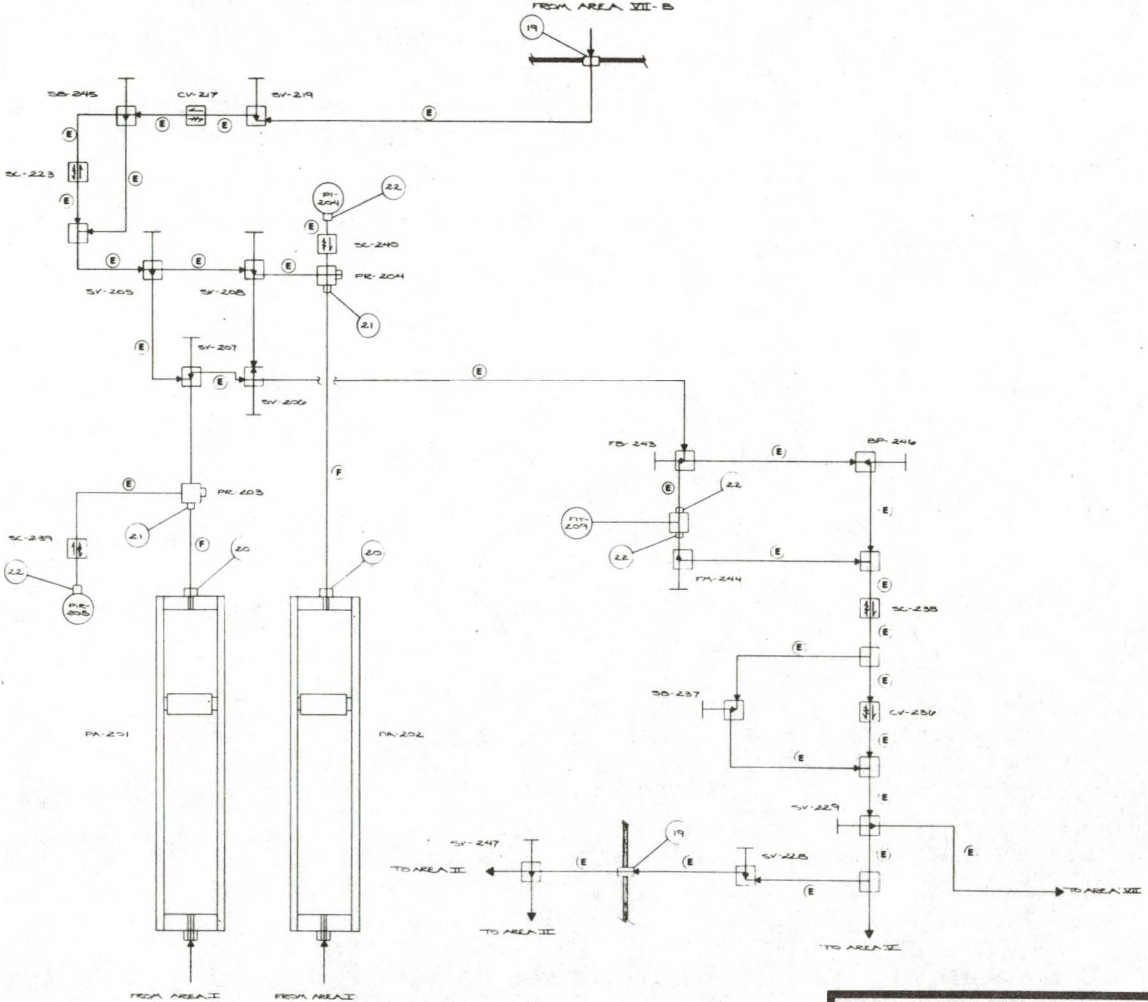
CHEMICAL ENGINEERING COAL LIQUIFICATION PROJECT UNIVERSITY OF NORTH DAKOTA - GRAND FORKS	
<b>AREA II</b>	
ENG. J. RINDT	DES. BY J. BETCHER
DATE: 11-7-79	DWG. NO. 1 FY79-TO-4-12

Figure A-3. Detail diagram of the UND autoclave facilities slurry charge components.

AP - APPCLAVE	PIR - PRESSURE INDICATOR RECORDER
AC - CHARGING GAS CLEANUP APPARATUS	PI - PRESSURE INDICATOR
BA - BLASTER ACCUMULATOR	PV - SOLENOID VALVE
CB - COLLECTION POINT	QV - QUENCH VALVE
CC - COOL TANK	RS - RESERVOIR
CV - CHECK VALVE	RS1 - REMOTE SAMPLE TRIP
DC - DROOP VALVE	RI - PRESSURE INDICATOR VALUE
FT - FLOW INDICATOR	RVS - REMOTE VACUUM SOURCE
FTT - FLOW INDICATOR TOTALIZER	RS - SURGE CHECK BYPASS VALVE
FL - FLEES	SC - SURGE CHECK VALVE
FM - FINE FILTERING VALVE	SL - SAMPLE LOOP
FT - FLOW VALVE	SP - SURGE FLOW ACCUMULATOR
GC - GAS CHROMATOGRAPH	ST - SAMPLE TRAY
GS - GAS SAMPLING VALVE	SV - SHUTOFF VALVE
MC - MAGNETIC TACHOMETER PICKUP	TI - TEMPERATURE INDICATOR
PA - FLOW ACCUMULATOR	TIC - TEMPERATURE INDICATOR CONTROLLER
PD - POSITIVE DISPLACEMENT PUMP	TIR - TEMPERATURE INDICATOR RECORDER
PC - CHECK PRESSURE RED. VALVE	
PI - PRESSURE INDICATOR	

PIPE SCHEDULE			
(A)	3/16" O.D.	20,000 psi (S.S.)	
(B)	3/8" O.D.	20,000 psi (S.S.)	
(C)	1/4" O.D.	Plastic Instrument Tubing	
(D)	3/16" O.D.	40,000 psi (S.S.)	
(E)	1/4" O.D.	40,000 psi (S.S.)	
(F)	3/8" O.D.	60,000 psi (S.S.)	
(G)	1/8" O.D.	11,000 psi (S.S.)	
(H)	1/4" O.D.	11,000 psi (S.S.)	

FITTING SCHEDULE			
1	30W2296	Bullhead	(3/16" - 20K) A.E.
2	30W564	Adaptor	(3/16" 20K x 3/8" 20K) A.E.
3	30W786	Adaptor	(1/4" NPTM - 3/16" 20K) A.E.
4	30W786	Adaptor	(1/4" NPTM - 3/16" 20K) A.E.
5	30W1786	Adaptor	(3/4" NPTM - 3/16" 20K) A.E.
6	30W1786	Adaptor	(3/4" NPTM - 3/16" 20K) A.E.
7	30W966	Adaptor	(1/4" NPTM - 3/8" 20K) A.E.
8	30W966	Adaptor	(1/4" NPTM - 3/8" 20K) A.E.
9	30W966	Adaptor	(3/16" 20K - 3/8" 20K) A.E.
10	30W966	Adaptor	(1" NPTM - 3/16" 20K) A.E.
11	30W2466	Bullhead	(3/8" 20K) A.E.
12	30W1786	Adaptor	(3/4" NPTM - 3/8" 20K) A.E.
13	30W966	Adaptor	(3/16" 60K - 3/8" 20K) A.E.
14	30W966	Adaptor	(3/16" 60K - 3/4" 60K) A.E.
15	30W1466	Adaptor	(1" NPTM - 3/8" 20K) A.E.
16	30W1466	Adaptor	(3/4" NPTM - 3/8" 20K) A.E.
17	30W4433	Bullhead	(1/4" 60K) A.E.
18	30W1466	Adaptor	(1" NPTM - 3/8" 60K) A.E.
19	30W4433	Bullhead	(1/4" 60K) A.E.
20	30W1466	Adaptor	(1" NPTM - 3/8" 60K) A.E.
21	30W4433	Bullhead	(1/4" 60K - 3/8" 60K) A.E.
22	30W966	Adaptor	(3/16" 60K - 1/4" 60K) A.E.
23	30W966	Adaptor	(1/2" NPTM - 1/4" 60K) A.E.
24	30W966	Adaptor	(1/4" 60K - 3/4" 10K) A.E.
25	DCB-8-500,000-2-500	Dutch Connect	(1/8" Swagelok)
26	30PC-6	Adaptor	(3/8" 11K - 1/4" 11K) Swagelok
27	30PC-6-1/2-3/8-11K	Adaptor	(3/8" - 1/4" - 3/8" - 1/8") Swagelok
28	30W4433	Bullhead	(1/4" 10K - 1/8" 10K) A.E.
29	30W4433	Bullhead	(1/4" 10K - 1/8" 10K) A.E.
30	30W4433	Bullhead	(1/4" 10K - 1/8" 10K) A.E.
31	30W2231	Bullhead	(1/8" 11K) A.E.
32	30W232	Adaptor	(1/2" NPT - 1/8" 10K) A.E.
33	30W232	Adaptor	(1/2" NPT - 1/8" 10K) A.E.
34	30-2-30K-1	Weld Tee	(1/8" 11K) CA316
35	30W4433	Bullhead	(1/4" 10K - 1/2" 11K) A.E.
36	30W4433	Bullhead	(1/4" 10K - 1/2" 11K) A.E.



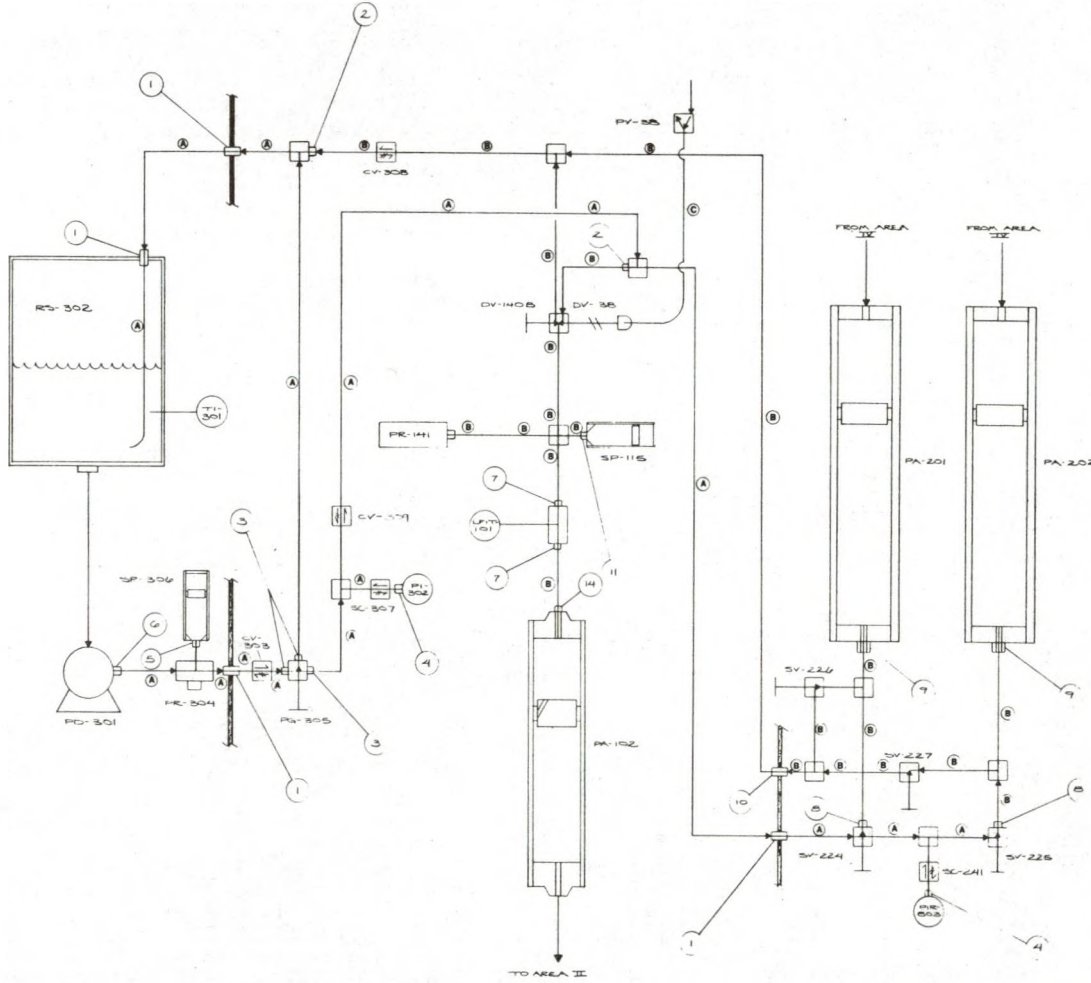
CHEMICAL ENGINEERING COAL LIQUIFICATION PROJECT UNIVERSITY OF NORTH DAKOTA - GRAND FORKS	
<b>AREA IV</b>	
ENGR. J. RINDT	DRN. BY J. BETCHER
DATE: 11-7-79	DRG. NO. 1FY79-4-14

Figure A-4. The Gas Compression System.

AG - AUTOCLAVE	PIR - PRESSURE INDICATOR RECORDER
AC - CARRIER GAS CLEANUP APPARATUS	PR - PRESSURE RELIEF VALVE
BA - BLOWER ACCUMULATOR	PV - SOLENOID VALVE
CP - COLLECTION PUMP	CV - CHECK VALVE
CT - COLD TRAP	RS - RESERVOIR
CV - CHECK VALVE	RSI - REMOVE SAMPLE INLET
DV - DOUBLE VALVE	RV - PRESSURE REGULATOR VALVE
FI - FLOW INDICATOR	RVS - REMOVE VALUUM SOURCE
FTI - FLOW INDICATOR TRANSDUCER	SB - SURGE CHECK BYPASS VALVE
FS - FILTER	SC - SURGE CHECK VALVE
FW - FLOW RETURNING VALVE	SL - SAMPLE LOOP
FV - FLOW VALVE	SP - SURGE PISTON ACCUMULATOR
GC - GAS CHROMATOGRAPH	ST - SAMPLE TRAP
GV - GAS SAMPLING VALVE	SV - SHUTOFF VALVE
HV - MAGNETIC TACHOMETER PICKUP	TI - TEMPERATURE INDICATOR
PA - PISTON ACCUMULATOR	TIC - TEMPERATURE INDICATOR CONTROLLER
PD - POSITIVE DISPLACEMENT PUMP	TIR - TEMPERATURE INDICATOR RECORDER
PG - BACK PRESSURE REG. VALVE	
PI - PRESSURE INDICATOR	

PIPE SCHEDULE			
A	9/16" O.D.	20,000 psi (S.S.)	
B	3/8" O.D.	20,000 psi (S.S.)	
C	1/4" O.D.	Plastic Instrument Tubing	
D	9/16" O.D.	60,000 psi (S.S.)	
E	1/4" O.D.	60,000 psi (S.S.)	
F	3/8" O.D.	60,000 psi (S.S.)	
G	1/8" O.D.	11,000 psi (S.S.)	
H	1/4" O.D.	11,000 psi (S.S.)	

FITTING SCHEDULE		
1	ADW4206	Bulkhead 19/16" - 20K A.F.
2	ADW4206	Adaptor 19/16" 20K x 3/8" 20K1 A.F.
3	ADW4206	Adaptor 1 1/4" NPTF - 9/16" 20K1 A.F.
4	ADW4206	Adaptor 1 1/4" NPTF - 9/16" 20K1 A.F.
5	ADW4206	Adaptor 1 3/4" NPTF - 9/16" 20K1 A.F.
6	ADW4206	Adaptor 1 3/4" NPTF - 9/16" 20K1 A.F.
7	ADW4206	Adaptor 1 3/4" NPTF - 3/8" 20K1 A.F.
8	ADW4206	Adaptor 19/16" 20K - 3/8" 20K1 A.F.
9	ADW4206	Adaptor 1 1/2" NPTF - 9/16" 20K1 A.F.
10	ADW4206	Bulkhead 1 3/8" 20K1 A.F.
11	ADW4206	Adaptor 1 3/4" NPTF - 3/8" 20K1 A.F.
12	ADW4206	Adaptor 19/16" 60K - 3/8" 20K1 A.F.
13	ADW4206	Adaptor 19/16" 60K - 1/4" 60K1 A.F.
14	ADW4206	Adaptor 1 1/2" NPTF - 3/8" 20K1 A.F.
15	ADW4206	Adaptor 1 3/4" NPTF - 3/8" 20K1 A.F.
16	ADW4206	Bulkhead 1 1/4" 60K A.F.
17	ADW4206	Adaptor 1 1/2" NPTF - 3/8" 60K1 A.F.
18	ADW4206	Adaptor 1 1/4" 60K - 3/8" 60K1 A.F.
19	ADW4206	Adaptors 1 1/2" NPTF - 1/4" 60K1 A.F.
20	ADW4206	Adaptors 1 1/2" NPTF - 1/4" 60K1 A.F.
21	ADW4206	Adaptor 1 1/4" 60K - 1/4" 10K1 A.F.
22	ADW4206	Adaptor 1 1/4" 60K - 1/4" 10K1 A.F.
23	ADW4206	Adaptor 1 1/4" 60K - 1/4" 10K1 A.F.
24	ADW4206	Adaptor 1 1/4" 60K - 1/4" 10K1 A.F.
25	ADW4206	DN10 Connect 1/2" NPTF
26	ADW4206	Adaptor 1 1/2" 11K - 1/4" 11K1 A.F.
27	ADW4206	Adaptor 1 1/2" 11K - 1/4" 11K1 A.F.
28	ADW4206	Adaptor 1 1/4" 10K - 1/2" 10K1 A.F.
29	ADW4206	TEE 1 1/4" 60K1 A.F.
30	ADW4206	Adaptor 1 1/4" 60K - 1/2" 11K1 A.F.
31	ADW4206	Bulkhead 1 1/2" 11K1 A.F.
32	ADW4206	Adaptor 1 1/4" NPTF - 1/2" 10K1 A.F.
33	ADW4206	TEE 1 1/4" 11K1 A.F.
34	ADW4206	TEE 1 1/2" 11K1 A.F.
35	ADW4206	Adaptor 1 1/4" NPTF - 1/2" 11K1 A.F.
36	ADW4206	Adaptor 1 3/4" NPTF - 1/4" 11K1 A.F.



CHEMICAL ENGINEERING COAL LIQUEFACTION PROJECT UNIVERSITY OF NORTH DAKOTA - GRAND FORKS	
<b>AREA I</b>	
ENG. J. RINDT	DRN. BY J. BETCHER
DATE: 11-7-79	DRG. NO. 1 FY79-TO-4-II

Figure A-5. Detail diagram of the UND autoclave facilities high pressure hydraulic oil supply system.



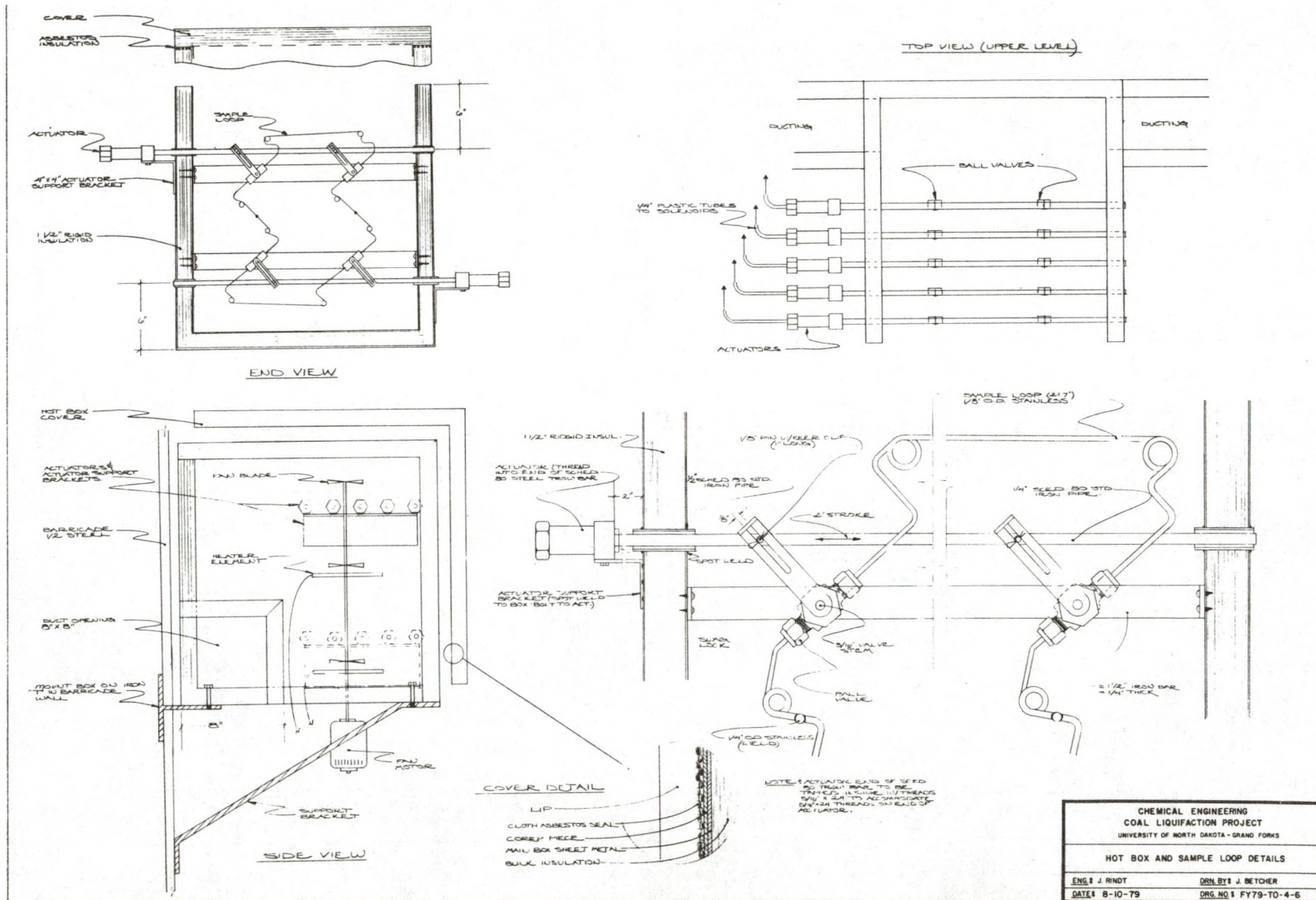
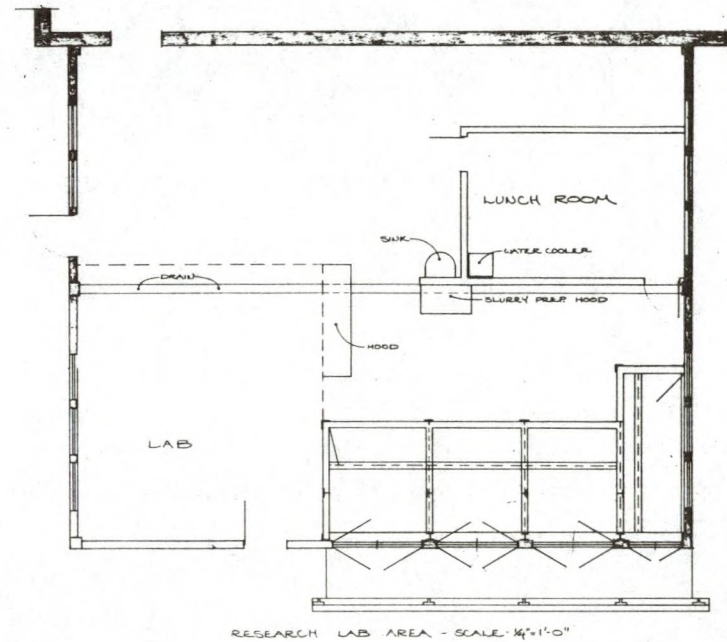
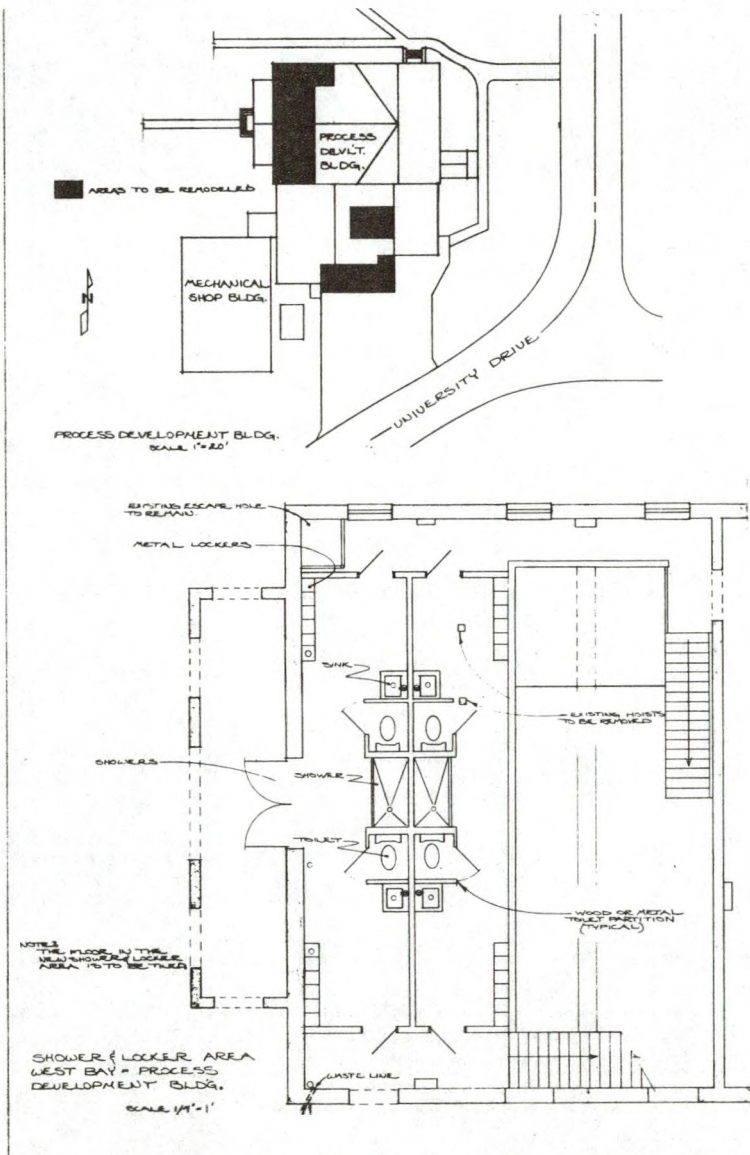


Figure A-7. Drawing of UND autoclave facilities GC sample loops and hot box.

CHEMICAL ENGINEERING COAL LIQUIFICATION PROJECT UNIVERSITY OF NORTH DAKOTA - GRAND FORKS	
HOT BOX AND SAMPLE LOOP DETAILS	
ENG: J. RINDT DATE: 8-10-79	DRN BY: J. BETCHER DRG. NO: FY79-TO-4-6



PROCESS DEVELOPMENT BLDG. FLOORPLAN SCALE 1/4"=1'-0"

CHEMICAL ENGINEERING COAL LIQUIFICATION PROJECT UNIVERSITY OF NORTH DAKOTA - GRAND FORKS	
LUNCH ROOM AND SHOWER FACILITIES - PDU BUILDING	
ENGR. J. RINDT	ENGR. R. J. BETCHER
DATE: 5-29-79	DRG. NO.: FY79-TO-4-3

Figure A-8. Floor plan of the UND building housing the autoclave facilities.

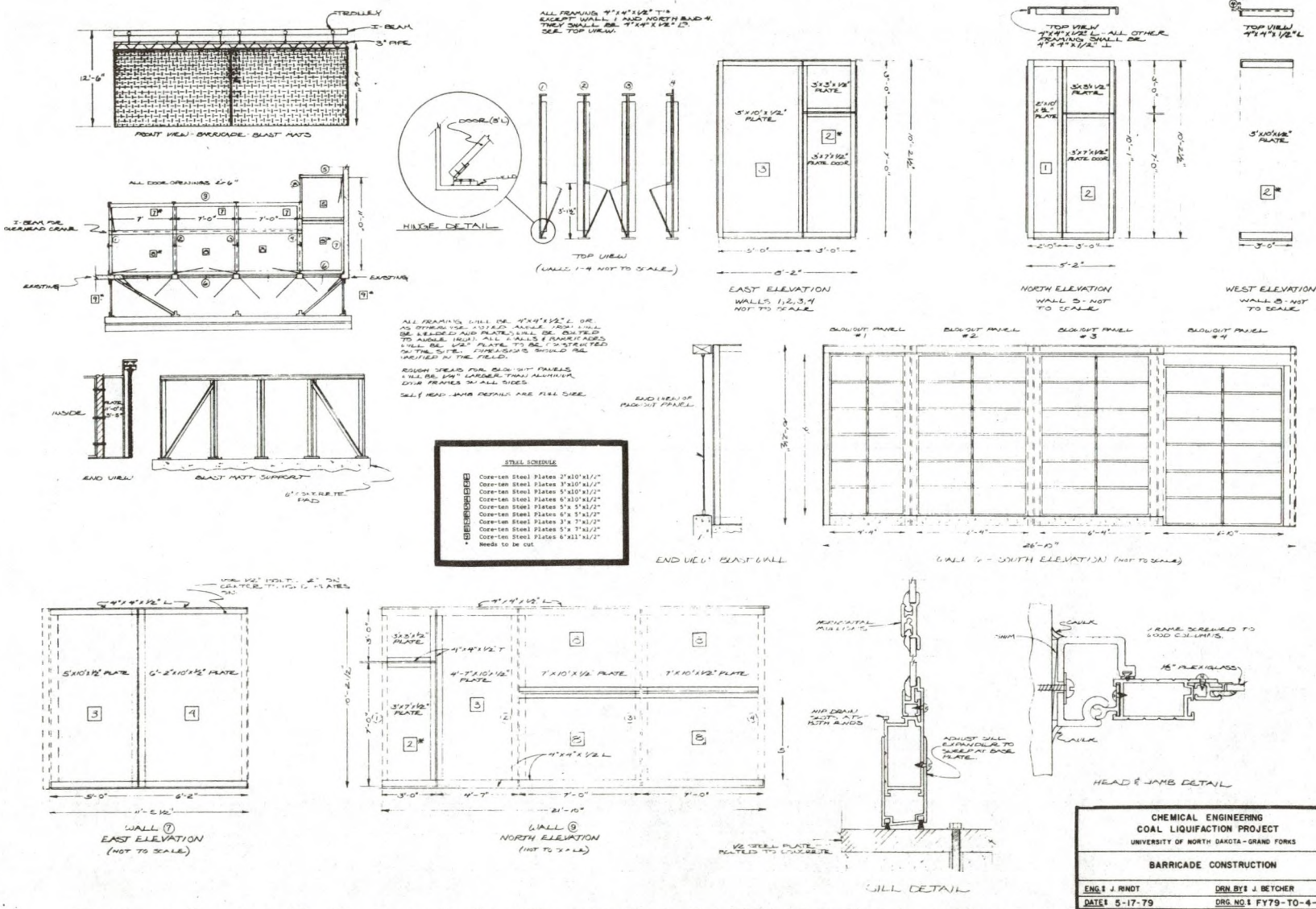


Figure A-9. Detail diagram of the UND autoclave facility barricade system.



## REFERENCES

1. Wilson, W., Knudson, C., Baker, G., and Owens, T., Severson, D., Application of Liquefaction Process to Low-Rank Coals. DOE publication for Presentation at the 1979 Lignite Symposium, May 30-31, 1979, Grand Forks, ND, p. 34
2. Knudson, C., Private Communication, 1979, Available on request from C. Knudson, Grand Forks Energy Technology Center, Grand Forks, ND
3. U.S. Department of Health, Education, and Welfare, Recommended Health and Safety Guidelines for Coal Gasification Pilot Plants, DHEW (NIOSH) Publication #78-120, p. 239

APPENDIX B

SAMPLE YIELD CALCULATIONS FOR RUN B-1

Calculate initial pressure in reactor.<sup>1</sup>

$$P(1) = P(1) + (P(3) * 0.49131)$$

$$= 400 + (29.03 * 0.49131)$$

$$P(1) = 414.2627 \text{ psig}$$

$$C(1) = ((273 + T(1)) / 293) * (Y1 / 100 * (M1 * P(1) + R1) + ((1 - Y1 / 100) * (P(1) * M2 + R2)))$$

$$C(1) = ((273 + 20) / 293) * (100 / 100 * (0.023776 * 414.2627 - 0.137239) + ((1 - 100 / 100) * (414.2627 * 0.024935 - 0.030819)))$$

$$= (1) * ((9.7123) + (0) * (10.2988))$$

$$= 9.7123 \text{ g-moles}$$

H2S Pressure

$$P(2) = P(2) + (P(3) * 0.49131)$$

$$= 0 + (29.03 * 0.49131)$$

$$= 14.2627 \text{ psig}$$

$$C(2) = ((273 + T(2)) / 293) * (Y1 / 100 * (M1 * P(2) + R1) + ((1 - Y1 / 100) * (P(2) * M2 + R2)))$$

$$= ((273 + 0) / 293) * (100 / 100 * (0.023776 * 14.2627 - 0.137239) + ((1 - 100 / 100) * (14.2627 * 0.024935 - 0.030819)))$$

$$= (0.9317) * ((0.2019) + (0) * (0.3248))$$

$$= 0.1881 \text{ g-moles}$$

Moles of H2 in Feed Gas Corrected for Temperature and Pressure

$$C(3) = (1) - C(2)$$

$$= 9.7123 - 0.1881$$

$$= 9.5242 \text{ g-moles}$$

Moles of Reactant Gas

$$N1 = C(3) + C(4)$$

$$= 9.5242 + 0$$

$$= 9.5242 \text{ g-moles}$$

Mole Percent of H2S in Reactant Gas

$$H2 = (C(4) / N1) * 100$$

$$= (0 / 9.5242) * 100$$

$$= 0$$

Mole Percent of H2 in Reactant Gas

$$H3 = ((Y1 / 100 * C(3)) / N1) * 100$$

$$= ((100 / 100 * 9.5242 / 9.5242) * 100)$$

$$= 100$$

Mole Percent CO in Reactant Gas

$$C1 = 100 - H2 - H3$$

$$= 100 - 0 - 100$$

$$= 0$$

---

<sup>1</sup>See Appendix E for meaning of the symbols used.

Mass of Reactant Gas

$$\begin{aligned} M7 &= N1C1/100*28.01 + N1*H3/100*2.016 + N1*H2/100*34.08 \\ &= 9.5242*0/100*28.01 + 9.5242*100/100*2.016 + 9.5242*0/100*34.08 \\ &= 0 + 19.2008 + 0 \\ &= 19.2008 \text{ grams} \end{aligned}$$

Moles of Product Gas

$$\begin{aligned} N2 &= P(3)*V1/25.01512 \\ &= 29.03*8.891/25.01512 \\ &= 10.3180 \text{ g-moles} \end{aligned}$$

Mass of Product Gas

$$\begin{aligned} M8 &= N2*61/100*44.01 + N2*62/100*30.07 + N2*63/100*44.09 + N2*64/100*1.016 + N2* \\ & \quad 65/100*16.04 + N2*66/100*28.01 + N2*67/100*34.08 + N2*68/100*17.08 \\ &= 10.318*8.925/100*44.01 + 10.318*0.395/100*30.07 + 10.318*0.193/100* \\ & \quad 44.09 + 10.318*86.436/100*2.016 + 10.318*1.064/100*16.04 + 10.318*1.88/100* \\ & \quad 28.01 + 10.318*0.164/100*34.08 + 10.318*0.02/100*17.08 \\ &= 68.4174 \text{ grams} \end{aligned}$$

Mass of Water in Slurry

$$\begin{aligned} X1 &= M4/(7 + (2/((100 - @5 - @4)/100))*@4/100)) \\ &= 724.14/(7 + (2/((100 - 29.31 - 7.91)/100))) \\ &= 99.8540 \text{ grams} \end{aligned}$$

Mass of HAO-61 Slurry

$$\begin{aligned} H6 &= 4*X1 \\ &= 4*99.8540 \\ &= 399.4160 \text{ grams} \end{aligned}$$

Mass of MAF Lignite in Slurry

$$\begin{aligned} L1 &= 2*X1 \\ &= 2*99.8540 \\ &= 199.7080 \text{ grams} \end{aligned}$$

Mass of Water out of Reactor

$$\begin{aligned} X2 &= @2/100*M6 + @3/100*M5 \\ &= 0.128/100*536.87 + 95.17/100*124.34 \\ &= 119.0216 \text{ grams} \end{aligned}$$

Mass of Distillates out of Reactor

$$\begin{aligned} D1 &= S1/100*M6 - @2/100*M6 + (100 - @3)/100*M5 \\ &= 77.2/100*536.87 - 0.128/100*536.87 + (100 - 95.17)/100*124.34 \\ &= 419.7821 \text{ grams} \end{aligned}$$

Mass of SRL out of Reactor

$$\begin{aligned} S3 &= (S2 - S1)/100*M6 \\ &= (92.17 - 77.20)/100*536.87 \\ &= 80.3694 \text{ grams} \end{aligned}$$

Mass of IOM out of Reactor

$$\begin{aligned} I1 &= (100 - S2)/100*M6 - M6*(@1/100) \\ &= (100 - 92.17)/100*536.87 - 536.87*(3.58/100) \\ &= 22.8170 \text{ grams} \end{aligned}$$

Mass of Ash in Endpot

$$\begin{aligned} A2 &= M6 * (\theta_1 / 100) \\ &= 536.87 * (3.58 / 100) \\ &= 19.2199 \text{ grams} \end{aligned}$$

Mass of Ash in Slurry

$$\begin{aligned} A1 &= (2 * X1 / ((100 - \theta_4 - \theta_5) / 100)) * \theta_4 / 100 \\ &= (2 * 99.8540 / ((100 - 7.91 - 2931) / 100)) * 7.91 / 100 \\ &= 25.1623 \text{ grams} \end{aligned}$$

Total Mass into Reactor

$$\begin{aligned} I2 &= M7 + L1 + X1 + A1 + H6 \\ &= 19.2008 + 199.7080 + 99.8540 + 25.1623 + 399.4160 \\ &= 743.3411 \text{ grams} \end{aligned}$$

Total Mass out of Reactor

$$\begin{aligned} O &= M8 + X2 + D1 + S3 + I1 + A2 \\ &= 68.4174 + 119.0216 + 419.7821 + 80.3694 + 22.8170 + 19.2199 \\ &= 729.6274 \text{ grams} \end{aligned}$$

Percent Closure

$$\begin{aligned} C6 &= O / I2 * 100 \\ &= 729.6274 / 743.3411 * 100 = 98.1551 \end{aligned}$$

Net Percent Distillates per MAF Lignite

$$\begin{aligned} D &= ((D1 - 4 * X1) / (2 * X1)) * 100 \\ &= ((419.7821 - 4 * 99.8540) / (2 * 99.8540)) * 100 \\ &= 10.1979 \end{aligned}$$

Net Percent SRL per MAF Lignite

$$\begin{aligned} S &= S3 / (2 * X1) * 100 \\ &= 80.3694 / (2 * 99.8540) * 100 \\ &= 40.2435 \end{aligned}$$

Net Percent Gas per MAF Lignite

$$\begin{aligned} G &= ((M8 - M7) / (2 * X1)) * 100 \\ &= ((68.4174 - 19.2008) / (2 * 99.8540)) * 100 \\ &= 24.6443 \end{aligned}$$

Net Percent IOM per MAF Lignite

$$\begin{aligned} I &= (I1 / (2 * X1)) * 100 \\ &= (22.8170 / (2 * 99.8540)) * 100 \\ &= 11.4252 \end{aligned}$$

Net Percent Water per MAF Lignite

$$\begin{aligned} H &= ((X2 - X1) / (2 * X1)) * 100 \\ &= ((119.0216 - 99.8540) / (2 * 99.8540)) * 100 \\ &= 9.5978 \end{aligned}$$

Net Percent Ash per MAF Lignite

$$\begin{aligned} A &= ((A2 - A1) / (2 * X1)) * 100 \\ &= ((19.2199 - 25.1623) / (2 * 99.8540)) * 100 \\ &= -2.9755 \end{aligned}$$

Percent Conversion

$$\begin{aligned} C7 &= 100 - I \\ &= 100 - 11.4252 \\ &= 88.5748 \end{aligned}$$

Total Net Yield per MAF Lignite

$$\begin{aligned} T &= D + S + G + I + H + A \\ &= 10.1979 + 40.2435 + 24.6443 + 11.4252 + 9.5978 - 2.9755 \\ &= 93.1332 \end{aligned}$$

Normalize Data

Weight of Endpot

$$\begin{aligned} M6 &= M6 * 100 / C6 \\ &= 536.87 * 100 / 98.1551 \\ &= 546.9609 \text{ grams} \end{aligned}$$

Mass Condensate in Cold Traps

$$\begin{aligned} M5 &= M5 * 100 / C6 \\ &= 124.34 * 100 / 98.1551 \\ &= 126.6771 \text{ grams} \end{aligned}$$

Mass or Product Gas

$$\begin{aligned} M8 &= M8 * 100 / C6 \\ &= 68.4174 * 100 / 98.1551 \\ &= 69.7034 \text{ grams} \end{aligned}$$

Mass Water out of Reactor

$$\begin{aligned} X2 &= @2 / 100 * M6 + @3 / 100 * M5 \\ &= 0.128 / 100 * 546.9609 + 9517 / 100 * 126.6771 \\ &= 121.2587 \text{ grams} \end{aligned}$$

Mass Distillates out of Reactor

$$\begin{aligned} D1 &= S1 / 100 * M6 - @2 / 100 * M6 + (100 - @3) / 100 * M5 \\ &= 77.2 / 100 * 546.9609 - 0.128 / 100 * 546.9609 + (100 - 95.17) / 100 * 126.6771 \\ &= 427.6722 \text{ grams} \end{aligned}$$

Mass SRL out of Reactor

$$\begin{aligned} S3 &= (S2 - S1) / 100 * M6 \\ &= (92.17 - 77.20) / 100 * 546.9609 \\ &= 81.8800 \text{ grams} \end{aligned}$$

Mass of IOM out of Reactor

$$\begin{aligned} I1 &= (100 - S2) / 100 * M6 - M6 * (@1 / 100) \\ &= (100 - 92.17) / 100 * 546.9609 * (358 / 100) \\ &= 23.2458 \text{ grams} \end{aligned}$$

Mass of Ash in Endpot

$$\begin{aligned} A2 &= M6 * (@1 / 100) \\ &= 546.9609 * (3.58 / 100) \\ &= 19.5812 \text{ grams} \end{aligned}$$

Mass of Ash in Slurry

$$\begin{aligned} A1 &= (2 * X1 / ((100 - @4 - @5) / 100) * @4 / 100) \\ &= (2 * 99.8540 / ((100 - 7.91 - 29.31) / 100) * 7.91 / 100) \\ &= 25.1623 \text{ grams} \end{aligned}$$

Total Mass into Reactor

$$\begin{aligned} I2 &= M7 + L1 + X1 + A1 + H6 \\ &= 19.2008 + 199.7080 + 99.8540 + 25.1623 + 399.4160 \\ &= 743.3411 \text{ grams} \end{aligned}$$

Total Mass out of Reactor

$$\begin{aligned} O &= M8 + X2 + D1 \text{ S3 } I1 + A2 \\ &= 69.7034 + 121.2587 + 427.6722 + 81.8800 + 23.2458 + 19.5812 \\ &= 743.3413 \text{ grams} \end{aligned}$$

Percent Closure

$$\begin{aligned} C6 &= O / I2 * 100 \\ &= 743.3413 / 743.3411 * 100 \\ &= 100.0000 \end{aligned}$$

Net Percent Distillates per MAF Lignite

$$\begin{aligned} D &= ((D1 - 4 * X1) / (2 * X1)) * 100 \\ &= ((427.6722 - 4 * 99.8540) / (2 * 99.8540)) * 100 \\ &= 14.1488 \end{aligned}$$

Net Percent SRL per MAF Lignite

$$\begin{aligned} S &= S3 / (2 * X1) * 100 \\ &= 81.8800 / (2 * 99.8540) * 100 \\ &= 40.9999 \end{aligned}$$

Net Percent Gas per MAG Lignite

$$\begin{aligned} G &= ((M8 - MT) / (2 * X1)) * 100 \\ &= ((69.7034 - 19.2008) / (2 * 99.8540)) * 100 \\ &= 25.2882 \end{aligned}$$

Net Percent IOM Per MAF Lignite

$$\begin{aligned} I &= (I1 / (2 * X1)) * 100 \\ &= (23.2458 / (2 * 99.8540)) * 100 \\ &= 11.6399 \end{aligned}$$

Net Percent Water per MAF Lignite

$$\begin{aligned} H &= ((X2 - X1) / (2 * X1)) * 100 \\ &= ((121.2587 - 99.8540) / (2 * 99.8540)) * 100 \\ &= 10.7180 \end{aligned}$$

Net Percent Ash per MAF Lignite

$$\begin{aligned} A &= ((A2 - A1) / (2 * X1)) * 100 \\ &= ((19.5812 - 25.1623) / (2 * 99.8540)) * 100 \\ &= -2.7946 \end{aligned}$$

Percent Conversion

$$\begin{aligned} C7 &= 100 - I \\ &= 100 - 11.6399 \\ &= 88.3601 \end{aligned}$$

Total Net Yield per MAF Lignite

$$\begin{aligned} T &= D + S + G + I + H + A \\ &= 14.1488 + 40.9999 + 25.2882 + 11.6399 + 10.7180 - 2.7946 \\ &= 100.0002 \end{aligned}$$



APPENDIX C  
RUN CONDITIONS

## RUN CONDITIONS

Run Number	Temperature History	Lignite Moisture	Lignite Ash	Initial Pressure	Maximum Pressure	Feed Gas
B-1	Cold Charge Heat to 420°C Slow Cooling	29.31%	7.91%	400	3880	H <sub>2</sub>
B-2	Hot Charge at 320°C Heat to 420°C Quench Residence time = 29 min	19.91%	7.85%	400	4025	H <sub>2</sub>
B-3	Hot Charge at 360°C Heat to 420°C Hold at 420°C until total time = 29 min	29.14%	7.93%	400	3950	H <sub>2</sub>
B-4	Cold Charge Heat to 420°C Slow Cooling	29.39%	7.90%	400	3325	CO-H <sub>2</sub>
B-5	Hot Charge at 320°C Heat to 420°C Hold at 420°C until total time = 29 min	29.96%	7.84%	400	3900	CO-H <sub>2</sub>
B-6	Repeat of B-4	29.95%	7.84%	400	3900	CO-H <sub>2</sub>

## RUN 1

Run #, Date of Run, Atm Press, H2 Press, H2S Press, % H2 in Feed Gas

B-1 , 41682 , 29.03 , 400, 0 , 100

H2-CO Temp, H2S Temp, Mass Slurry In, Mass Condensate Out

20.0 , 0.00 , 724.1 , 124.34

Vol Gas Out, Mass Endpot, % Ash in Endpot, % H2O in Lignite

8.90 , 536.9 , 3.58 , 29.31

% H2O in Endpot, % Cyclohex Soluble, % THF Soluble

0.13 , 77.20 , 92.17

% H2O in Condensate, % Ash in Lignite

95.17 , 7.91

Product Gas Composition CO2, C2H6, C3H8, H2, CH4, CO, H2S, NH3

8.93, 0.40, 0.19, 86.44, 1.06, 1.88, 0.16, 0.02

## RUN 2

Run #,	Date of Run,	Atm Press,	H2 Press,	H2S Press,	% H2 in Feed Gas			
B-2,	42782,	29.50,	400,	0,	100			
H2-CO Temp,	H2S Temp,	Mass Slurry In,	Mass Condensate Out					
20,	0 ,	716.5 ,	12.60					
Vol Gas Out,	Mass Endpot,	% Ash in Endpot,	% H2O in Lignite					
8.77 ,	678.2,	3.51 ,	29.91					
% H2O in Endpot,	% Cyhex Soluble,	% THF Soluble						
16.21 ,	75.25 ,	86.68						
% H2O in Condensate,	% Ash in Lignite							
92.67 ,	7.85							
Product Gas Composition	CO2,	C2H6,	C3H8,	H2,	CH4,	CO,	H2S,	NH3
	7.04,	0.06,	0.01,	89.75,	0.39,	0.22,	0.15,	0.01

## RUN 3

Run #,	Date of Run,	Atm Press,	H2 Press,	H25 Press,	% H2 in Feed Gas			
B-3,	50482,	28.91 ,	400 ,	0 ,	100			
H2-CO Temp,	H2S Temp,	Mass Slurry In,	Mass Condensate Out					
20 ,	0 ,	726.7 ,	2.03					
Vol Gas Out,	Mass Endpot,	% Ash in Endpot,	% H2O in Lignite					
8.27 ,	677.9 ,	3.67 ,	29.14					
% H2O in Endpot,	% Cyhex Soluble,	% THF Soluble						
16.13 ,	78.33 ,	91.78						
% H2O in Condensate,	% Ash in Lignite							
95.0 ,	7.93							
Product Gas Composition	CO2,	C2H6,	C3H8,	H2,	CH4,	CO,	H2S,	NH3
	4.50,	0.52,	0.00,	97.02,	0.86,	0.15,	0.19,	0.04

## RUN 4

Run #,	Date of Run,	Atm Press,	H2 Press,	H25 Press,	% H2 in Feed Gas			
B-4,	51882,	28.96,	400 ,	0 ,	44.36			
H2-CO Temp,	H2S Temp,	Mass Slurry In,	Mass Condensate Out					
20 ,	0 ,	719.9 ,	105.88					
Vol Gas Out,	Mass Endpot,	% Ash in Endpot,	% H2O in Lignite					
7.92 ,	532.7 ,	4.12 ,	29.39					
% H2O in Endpot,	% Cyhex Soluble,	% THF Soluble						
0.14 ,	76.29 ,	93.09						
% H2O in Condensate,	% Ash in Lignite							
100 ,	7.90							
Product Gas Composition	CO2,	C2H6,	C3H8,	H2,	CH4,	CO,	H2S,	NH3
	17.42,	0.48,	0.00,	50.66,	1.43,	28.01,	0.09,	0.03

## RUN 5

Run #, Date of Run, Atm Press, H2 Press, H2S Press, % H2 in Feed Gas

B-5, 52182 , 29.26 , 400 , 0 , 44.36

H2-CO Temp, H2S Temp, Mass Slurry In, Mass Condensate Out

20 , 0 , 705.7 , 3.89

Vol Gas Out, Mass Endpot, % Ash in Endpot, % H2O in Lignite

9.17 , 643.9 , 4.04 , 29.96

% H2O in Endpot, % Cycchex Soluble, % THF Soluble

8.95 , 78.66 , 92.65

% H2O in Condensate, % Ash in Lignite

100 , 7.84

Product Gas Composition CO2, C2H6, C3H8, H2, CH4, CO, H2S, NH3

29.71, 0.53, 0.00, 41.81, 0.70, 26.45, 0.18, 0.02

## RUN 6

Run #,	Date of Run,	Atm Press,	H2 Press,	H25 Press,	% H2 in Feed Gas			
B-6,	60882,	29.19 ,	400 ,	0 ,	44.36			
H2-CO Temp,	H2S Temp,	Mass Slurry In,	Mass Condensate Out					
20 ,	0 ,	722.9 ,	105.97					
Vol Gas Out,	Mass Endpot,	% Ash in Endpot,	% H2O in Lignite					
9.80 ,	544.1 ,	3.27 ,	29.55					
% H2O in Endpot,	% Cyhex Soluble,	% THF Soluble						
0.16 ,	75.70 ,	93.39						
% H2O in Condensate,	% Ash in Lignite							
56.25 ,	7.84							
Product Gas Composition	CO2,	C2H6,	C3H8,	H2,	CH4,	CO,	H2S,	NH3
	22.98,	0.27,	0.01,	46.13,	0.73,	28.87,	0.20,	0.20



APPENDIX D  
COMPUTER RUN SUMMARIES

RUN NUMBER            B1                            DATE                            41682

INPUT  
.....

COMPONENT	GRAMS IN
MAF LIGNITE	199.708
H2O	99.854
ASH	25.162
HAO-61	399.416
GAS	19.201
TOTAL	743.340

.....  
OUTPUT

COMPONENT	GRAMS OUT	NET YIELDS WTZ MAF LIG
H2O	119.022	9.598
OIL	419.782	10.198
SRL	80.369	40.243
IOH	22.817	11.425
ASH	19.220	-2.976
GAS	68.417	24.644
TOTAL	729.627	93.133

% CLOSURE                            98.155  
% CONVERSION                        88.575

.....  
NORMALIZED  
OUTPUT

COMPONENT	GRAMS OUT	NET YIELDS WTZ MAF LIG
H2O	121.259	10.718
OIL	427.672	14.149
SRL	81.880	41.000
IOH	23.246	11.640
ASH	19.581	-2.795
GAS	69.703	25.288
TOTAL	743.340	100.000

% CLOSURE                            100.000  
% CONVERSION                        88.360

.....  
TIME 0.2 SECS

RUN NUMBER            B2                            DATE                            42782

INPUT

.....

COMPONENT	GRAMS IN
MAF LIGNITE	197.594
H2O	98.797
ASH	24.921
HAD-61	395.188
GAS	19.201
TOTAL	735.701

OUTPUT

.....

COMPONENT	GRAMS OUT	NET YIELDS WT% MAF LIG
H2O	121.619	11.550
OIL	401.356	3.122
SRL	77.523	39.233
IOM	66.535	33.673
ASH	23.806	-0.564
GAS	52.843	17.026
TOTAL	743.682	104.039

% CLOSURE                            101.085  
% CONVERSION                        66.327

NORMALIZED  
OUTPUT

.....

COMPONENT	GRAMS OUT	NET YIELDS WT% MAF LIG
H2O	120.314	10.890
OIL	397.049	0.942
SRL	76.691	38.812
IOM	65.821	33.311
ASH	23.551	-0.694
GAS	52.276	16.739
TOTAL	735.701	100.000

% CLOSURE                            100.000  
% CONVERSION                        66.689

.....  
TIME 0.2 SECS

RUN NUMBER            B3                            DATE                            50482

INPUT  
.....

COMPONENT	GRAMS IN
MAF LIGNITE	200.418
H2O	100.209
ASH	25.255
HAO-61	400.837
GAS	19.201
TOTAL	745.920

OUTPUT

COMPONENT	GRAMS OUT	NET YIELDS WT% MAF LIG
H2O	111.265	5.516
OIL	425.177	12.145
SRL	87.792	43.804
IOH	30.846	15.391
ASH	24.880	-0.187
GAS	40.527	10.641
TOTAL	720.486	87.309

% CLOSURE                            96.590  
% CONVERSION                        84.609

NORMALIZED  
OUTPUT

COMPONENT	GRAMS OUT	NET YIELDS WT% MAF LIG
H2O	115.193	7.476
OIL	440.186	19.633
SRL	90.891	45.351
IOH	31.935	15.934
ASH	25.758	0.251
GAS	41.958	11.355
TOTAL	745.920	100.000

% CLOSURE                            100.000  
% CONVERSION                        84.066

TIME 0.2 SECS

RUN NUMBER            B4                            DATE                            51882

INPUT  
.....

COMPONENT	GRAMS IN
MAF LIGNITE	198.534
H2O	99.267
ASH	25.011
HAO-61	397.068
GAS	268.109
TOTAL	987.988

OUTPUT

COMPONENT	GRAMS OUT	NET YIELDS WT% MAF LIG
H2O	106.636	3.712
OIL	405.648	4.321
SRL	89.495	45.078
IOM	14.863	7.486
ASH	21.948	-1.543
GAS	155.312	-56.815
TOTAL	793.901	2.240

% CLOSURE                            80.355  
% CONVERSION                        92.514

NORMALIZED  
OUTPUT

COMPONENT	GRAMS OUT	NET YIELDS WT% MAF LIG
H2O	132.706	16.843
OIL	504.817	54.272
SRL	111.374	56.098
IOM	18.496	9.316
ASH	27.313	1.160
GAS	193.282	-37.690
TOTAL	987.989	100.000

% CLOSURE                            100.000  
% CONVERSION                        90.684

TIME 0.2 SECS

RUN NUMBER            85                            DATE                            52182

INPUT

.....

COMPONENT	GRAMS IN
MAF LIGNITE	194.620
H2O	97.310
ASH	24.531
HAO-61	389.240
GAS	161.280
TOTAL	866.980

OUTPUT

.....

COMPONENT	GRAMS OUT	NET YIELDS WT% MAF LIG
H2O	61.515	-18.392
OIL	448.835	30.621
SRL	90.076	46.283
IOM	21.312	10.950
ASH	26.012	0.761
GAS	232.292	36.487
TOTAL	880.041	106.711

% CLOSURE                            101.507  
% CONVERSION                        89.050

NORMALIZED  
OUTPUT

.....

COMPONENT	GRAMS OUT	NET YIELDS WT% MAF LIG
H2O	60.602	-18.861
OIL	442.173	27.198
SRL	88.739	45.596
IOM	20.995	10.788
ASH	25.626	0.563
GAS	228.844	34.716
TOTAL	866.980	100.000

% CLOSURE                            100.000  
% CONVERSION                        89.212

TIME 0.2 SECS

RUN NUMBER            86                            DATE                            60882

INPUT

COMPONENT	GRAMS IN
MAF LIGNITE	199.398
H2O	99.699
ASH	24.968
HAO-61	398.795
GAS	161.279
TOTAL	884.139

OUTPUT

COMPONENT	GRAMS OUT	NET YIELDS WTZ MAF LIG
H2O	60.457	-19.680
OIL	457.359	29.370
SRL	96.242	48.267
IOM	18.171	9.113
ASH	17.790	-3.600
GAS	222.194	30.549
TOTAL	872.213	94.019

% CLOSURE                            98.651  
% CONVERSION                        90.887

NORMALIZED  
OUTPUT

COMPONENT	GRAMS OUT	NET YIELDS WTZ MAF LIG
H2O	61.283	-19.266
OIL	463.612	32.506
SRL	97.558	48.927
IOM	18.420	9.238
ASH	18.034	-3.478
GAS	225.232	32.073
TOTAL	884.139	100.000

% CLOSURE                            100.000  
% CONVERSION                        90.762

TIME 0.2 SECS

APPENDIX E

COMPUTER PROGRAM FOR RUN CALCULATIONS



## LIST OF SYMBOLS IN MATERIAL BALANCE PROGRAM

D0	Date of Run
P8	Run Number
P(1)	H <sub>2</sub> or Synthesis Gas Pressure (psig)
P(2)	H <sub>2</sub> S Pressure (psig)
P(3)	Barometric Pressure (inches of Hg)
Y1	Percent H <sub>2</sub> in Feed Gas
T(1)	H <sub>2</sub> or Synthesis Gas Temperature (°C)
T(2)	H <sub>2</sub> S Temperature (°C)
C(1)	Calibration Factor
C(2)	Calibration Factor
C(3)	Moles of H <sub>2</sub> in Feed Gas
C(4)	Moles of H <sub>2</sub> S in Feed Gas
N1	Moles of Feed Gas
H2	Mole Percent of H <sub>2</sub> S in Feed Gas
H3	Mole Percent of H <sub>2</sub> in Feed Gas
C1	Mole Percent CO in Feed Gas
M7	Moles of Feed Gas
N2	Moles of Product Gas
M8	Mass of Product Gas
X1	Mass of Water in Slurry
H6	Mass of HAO-61 in Slurry
L1	Mass of MAF Lignite in Slurry
X2	Mass of Water Out of Reactor
D1	Mass of Distillates Out of Reactor
S3	Mass of SRL Out of Reactor
I1	Mass of IOM Out of Reactor
A2	Mass of Ash in Endpot
A1	Mass of Ash in Slurry
I2	Total Mass into Reactor
O	Total Mass Out of Reactor
C6	Percent Closure
D	Net Percent Distillates per MAF Lignite
S	Net Percent SRL for MAF Lignite

G Net Percent Gas Per MAF Lignite  
I Net Percent IOM Per MAF Lignite  
H Net Percent Water Per MAF Lignite  
A Net Percent Ash Per MAF Lignite  
CT Percent Conversion  
T Total Net Yield Per MAF Lignite  
M4 Mass of Slurry in Autoclave  
M5 Mass Condensate in Cold Traps  
V1 Volume of Product Gas  
M6 Mass of Endpot  
@1 Percent Ash in Endpot  
@2 Percent Water in Endpot  
S1 Percent of Endpot that is Cyclohexane Soluble  
S2 Percent of Endpot that is THF Soluble  
@3 Percent Water in Condensate  
G1 Mole Percent CO<sub>2</sub> in Product Gas  
G2 Mole Percent C<sub>2</sub>H<sub>6</sub> in Product Gas  
G3 Mole Percent C<sub>3</sub>H<sub>8</sub> in Product Gas  
G4 Mole Percent H<sub>2</sub> in Product Gas  
G5 Mole Percent CH<sub>4</sub> in Product Gas  
G6 Mole Percent CO in Product Gas  
G7 Mole Percent H<sub>2</sub>S in Product Gas  
G8 Mole Percent NH<sub>3</sub> in Product Gas  
@5 Percent H<sub>2</sub>O in Lignite

```

5 PRINT "RUN #, DATE OF RUN, ATM PRESS, H2-CO PRESS, H2S PRESS, XH2
IN FEED GAS"
10 INPUT P$,D0,P(3),P(1),P(2),Y1
15 PRINT "H2-CO TEMP, H2S TEMP, MASS SLURRY IN, MASS CONDENSATE OUT"
20 INPUT T(1),T(2),M4,M5
25 PRINT "VOLUME GAS OUT, MASS ENDPOT OUT, XASH IN ENDPOT, XH2O IN LIG
NITE"
30 INPUT V1,M6,@1,@5
35 PRINT "XH2O IN ENDPOT, XCYCLOHEXANE SOLUBLE, XTHF SOLUBLE"
40 INPUT @2,S1,S2
45 PRINT "XH2O IN CONDENSATE, XASH IN LIGNITE"
50 INPUT @3,@4
70 PRINT "PRODUCT GAS COMPOSITION: CO2,C2H6,C3H8,H2,CH4,CO,H2S,NH3"
80 INPUT G1,G2,G3,G4,G5,G6,G7,G8
90 M1= 2.377600E-02
100 M2= 2.493500E-02
110 M3= 1.985780E-02
120 R1= -1.372390E-01
130 R2= -3.081900E-02
140 R5= 2.653510E-02
150 FOR A6=1 TO 2
160 P(A6)= P(A6) + (P(3)*0.49131)
170 C(A6)= ((273+T(A6))/293)*(Y1/100*(M1*P(A6)+R1)+((1-Y1/100)*(P(A6)*M
2+R2)))
180 NEXT A6
190 C(3)= C(1)-C(2)
200 IF T(2)=0 THEN GOTO 220
210 C(4)= ((273+T(1))/294)*(M3*P(2)+R5)
220 N1= C(3)+C(4)
230 H2= (C(4)/N1)*100
240 H3= ((Y1/100*C(3))/N1)*100
250 C1= 100 -H2-H3
260 M7= N1*C1/100*28.01+N1*H3/100*2.016+N1*H2/100*34.08
270 N2= P(3)*V1/25.01512
280 M8= N2*G1/100*44.01+N2*G2/100*30.07+N2*G3/100*44.09+N2*G4/100*2.016
+N2*G5/100*16.04+N2*G6/100*28.01+N2*G7/100*34.08+N2*G8/100*17.08
290 X1= M4/(7+(2/((100-@5-@4)/100)*@4/100))
293 H6= 4*X1
297 L1= 2*X1
300 X2= @2/100*M6+@3/100*M5
310 D1= S1/100*M6-@2/100*M6+(100-@3)/100*M5
320 S3= (S2-S1)/100*M6
330 I1= (100-S2)/100*M6-M6*(@1/100)
335 A2= M6*(@1/100)
337 A1= (2*X1/((100-@4-@5)/100)*@4/100)
340 I2= M7+L1+X1+A1+H6
350 O= M8+X2+D1+S3+I1+A2
360 C6= O/I2*100
370 D= ((D1-4*X1)/(2*X1))*100

```

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380 S=S3/(2*X1)*100
390 G= ((M8-M7)/(2*X1))*100
400 I= (I1/(2*X1))*100
410 H= ((X2-X1)/(2*X1))*100
415 A= ((A2-A1)/(2*X1))*100
420 C7= 100-I
455 T= D+S+G+I+H+A
460 IF Z=1 THEN 675
470 PRINT
480 PRINT "RUN NUMBER", P$, "DATE", D0
490 PRINT
495 :          #####
500 PRINT "INPUT"
510 PRINT "....."
520 PRINT
530 PRINT "COMPONENT          GRAMS IN"
540 PRINT "....."
550 PRINT "MAF LIGNITE",
560 PRINT USING 495,L1
570 PRINT "H2O",
580 PRINT USING 495,X1
590 PRINT "ASH",
600 PRINT USING 495,A1
610 PRINT "HAO-61",
620 PRINT USING 495,H6
630 PRINT "GAS",
640 PRINT USING 495,M7
650 PRINT "  TOTAL",
660 PRINT USING 495,I2
670 PRINT "....."
"
673 IF Z=0 THEN 680
675 PRINT "NORMALIZED"
680 PRINT "OUTPUT"
690 PRINT "....."
700 PRINT "          GRAMS          NET YIELDS"
710 PRINT "COMPONENT  OUT          WIZ MAF LIG"
720 PRINT "....."
730 :#####          ###.###
740 PRINT "H2O",
750 PRINT USING 730,X2,H
760 PRINT "OIL",
770 PRINT USING 730,D1,D
773 PRINT "SRL",
777 PRINT USING 730,S3,S
780 PRINT "IOM",
790 PRINT USING 730,I1,I
800 PRINT "ASH",
810 PRINT USING 730,A2,A
820 PRINT "GAS",
830 PRINT USING 730,M8,G

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```
840 PRINT "  TOTAL",
850 PRINT USING 730,0,I
860 PRINT
870 :      ###.###
880 PRINT "% CLOSURE",
890 PRINT USING 870,C6
900 PRINT "% CONVERSION",
910 PRINT USING 870,C7
920 PRINT "....."
"
930 IF Z=1 THEN 1000
940 M6= M6*100/C6
950 M5= M5*100/C6
960 M8= M8*100/C6
970 Z=1
980 GOTO 300
1000 STOP
1010 END
```

APPENDIX F  
LIST OF ABBREVIATIONS

MAF	Moisture Ash Free
°C	Degrees Celcius
°F	Degrees Fahrenheit
psig	Pounds per square inch, guage
psi	Pounds per square inch
SRC	Solvent Refined Coal
SRL	Solvent Refined Lignite
UND	University of North Dakota
PDU	Process Development Unit
CPU	Continuous Process Unit
PETC	Pittsburgh Energy Technology Center
GFETC	Grand Forks Energy Technology Center
COSTEAM	Carbon Monoxide and Steam
ASTM	American Society of Testing and Materials
APHA	American Public Health Association
IOM	Insoluble Organic Matter
HAO-61	Hydrogenated Anthracene Oil from Run Number 61
AO-4	Anthracene Oil - Batch Number 4
THF	Tetrahydrofuran
mL	Milliliters
min	Minute
Hg	Mercury

LIST OF REFERENCES



## LIST OF REFERENCES

1. Whitehurst, D. D., T. O. Mitchell, and M. Farcasiu. Coal Liquefaction The Chemistry and Technology of Thermal Processes. Academic Press Inc., New York, 1980, 378 pp
2. Conn, A. L. Conversion of Coal to Oil and Gas. Chem. Eng. Prog., 77, No. 5, 11-20 (1981)
3. Wu, W. R. K. and H. H. Storch. Hydrogenation of Coal and Tar. U.S. Bur. Mines Bulletin, No. 633, 1-10 (1968)
4. Berkowitz, N. An Introduction to Coal Technology. Academic Press Inc., New York, 1979, 345 pp
5. Pott, A. and H. Broche. The Solution of Coal by Extraction Under Pressure. Fuel 13, 91-95, 125-128, 154-157 (1934)
6. Elliott, M. A. Chemistry of Coal Utilization. John Wiley & Sons Inc., New York, 1981, 2374 pp
7. Appell, H. R. and I Wender. The Hydrogenation of Coal with Carbon Monoxide and Water. Div. Fuel Chem. Preprints, Am. Chem. Soc., 12, No. 3, 220-222 (1968)
8. Appell, H. R., I. Wender, and R. D. Miller. Solubilization of Low Rank Coal with Carbon Monoxide and Water. Chem. and Ind., 47, 1703 (1969)
9. Severson, D. E., D. R. Skidmore, and D. S. Gleason. Solution-Hydrogenation of Lignite in Coal-Derived Solvents. AIME Trans., 247, 133-136 (June 1970)
10. Descie, S. N. Thermal Dissolution of Lignite Under Hydrogen Pressure. Masters Thesis, University of North Dakota, 1969
11. Severson, D. E., A. M. Souby, and W. R. Kube. Laboratory Studies on Solution-Hydrogenation of Lignite. Symp. on Tech. and Use of Lignite, Grand Forks, N. Dak., 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, 18, 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. Preprints, Am. Chem. Soc., 22, No. 6, 161-182 (1977)
14. Severson, D. E., A. M. Souby, and J. J. Harris. Process Development of Lignite Liquefaction. Symp. on Tech. and Use of Lignite, Grand Forks, N. Dak., May 18-19, 1977, GFERC/IC-77/1 NTIS, Springfield, Virg., 1978, pp. 159-192
15. Severson, D. E. Solvent Refined Lignite Process Development. DOE/FF/4189-15, U.S. DOE, NTIS, Oak Ridge, Tenn., 1979, 96 pp
16. Appell, H. R., E. C. Moroni, and R. D. Miller. COSTEAM Liquefaction of Lignite. Div. Fuel Chem. Preprints, Am. Chem. Soc., 20, No. 1, 58-65 (1975)
17. Willson, W. G., C. L. Knudson, G. G. Baker, T. C. Owens, and D. E. Severson. Application of Liquefaction Processes to Low-Rank Coals. Inc. Eng. Chem., Prod. Res. Dev., 18, No. 4, 297-310 (1979)
18. Sondreal, E. A., C. L. Knudson, J. E. Schiller, and T. H. May. Development of the COSTEAM Process for Liquefaction of Lignite and Western Subbituminous Coals. Symp. on Tech. and Use of Lignite, Grand Forks, N. Dak., May 18-19, 1977, GFERC/IC-77/1 NTIS, Springfield, Virg., 1978, pp. 129-158
19. Souby, A. M., T. C. Owens, and D. E. Severson. Laboratory Batch Liquefaction of Low Rank Coals. Div. Fuel Chem. Preprints, Am. Chem. Soc., 25, No. 1, 233-244 (1980)
20. Gorin, E., H. E. Lebowitz, C. H. Rice, and R. T. Struck. A Synthetic Fuels Process. Proc. Eighth World Pet. Cong., 4, 43-53 (1970)
21. Curran, G. P., R. T. Struck, and E. Gorin. Mechanism of the Hydrogen-Transfer Process to Coal and Coal Extract. Ind. Eng. Chem., Proc. Des. Dev., 6, No. 2, 166-173 (1967)
22. Spencer, D. Advanced Coal Liquefaction. EPRI Journal, 1, No. 3, 31-34 (1982)
23. 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)
24. Appell, H. R., E. C. Moroni, and R. D. Miller. COSTEAM Liquefaction of Lignite. Div. Fuel Chem. Preprints, Am. Chem. Soc., 20, No. 1, 58-65 (1975)
25. Appell, H. R., E. C. Moroni, and R. D. Miller. Hydrogenation of Lignite with Synthesis Gas. Energy Sources, 3, No. 2, 163-175 (1977)

26. Franson, M. A. H. Standard Methods for the Examination of Water and Wastewater. R. R. Connelly & Sons Company, New York, 15th Ed. 1981, 1134 pp
27. Crockford, H. D., J. W. Nowell, H. W. Baird, and F. W. Getzon. Laboratory Manual of Physical Chemistry. John Wiley and Sons Inc., New York, 2nd Ed., 1975, pp. 44-49
28. Hanson, G. B. Microreactor Studies of Lignite Liquefaction. Masters Thesis, University of North Dakota, 1982
29. Culpon, Jr., D. H. A Kinetic Model for the Liquefaction of Lignite In A Continuous Stirred Tank Reactor. Masters Thesis, Texas A & M University, 1982
30. Handwerk, J. B., R. M. Baldwin, J. O. Golden, J. H. Gory. CO-STEAM Liquefaction In A Bath Autoclave. Div. Fuel Chem. Preprints, Am. Chem. Soc., 20, No. 1, 26-46 (1975)
31. Fu, Y. C. and E. G. Illig. Catalytic Coal Liquefaction Using Synthesis Gas. Div. Fuel Chem. Preprints, Am. Chem. Soc., 20, No. 1, 47-57 (1975)