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LIGNITE LIQUEFACTION USING CARBON MONOXIDE AND WATER IN A HOT-CHARGE BATCH AUTOCLAVE

by
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Bachelor of Science in Chemical Engineering,
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Lignite Liquefaction Using Carbon Monoxide and Water In a Hot-Charge Batch Autoclave

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The University of North Dakota, 1982

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The effects of temperature history on the liquefaction of lignite were investigated using the UND hot-charge, time-sampled batch autoclave facility. Approximately two hundred grams of moisture- and ash-free lignite, one hundred grams of water, four hundred grams of hydrogenated anthracene oil solvent and carbon monoxide gas were reacted in both cold-charge and hot-charge experiments. The oil yields and overall conversions were determined by extraction with cyclohexane and tetrahydrofuran, respectively. Maximum operating pressures ranged from 3670 psig to 3925 psig and the maximum reaction temperature was approximately 420°C in all cases.

Oil yields and overall conversion ranged from 16.0 percent to 34.3 percent and 80.6 percent to 92.5 percent, respectively. Increasing the hot-charge temperature above 320°C significantly increased the oil yields and conversions. The increased oil yields and conversions were due to the increase in time at temperatures above 360°C. Increasing the hot-charge temperature above 360°C did not change the oil yields or conversions. The oil yields and conversions for the cold-charge, slow-cooling runs were found to be similar to the results obtained by hot-charging the feed slurry at temperatures above 360°C. The unchanged conversions and oil yields were the result of the time above 360°C being longer than necessary for the liquefaction reactions to reach completion.

This Thesis submitted by Michael L. Swanson 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)

Wayne R Kube

Wm. J. Lawrence

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

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Title Lignite Liquefaction Using Carbon Monoxide and Water in a Hot-Charge Batch Autoclave

Department Chemical Engineering

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Signature Mike Swanson

Date August 12, 1982

CONTENTS

FIGURES

TABLES

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ABSTRACT

The effects of temperature history on the liquefaction of lignite were investigated using the UND hot-charge, time-sampled batch autoclave facility. Approximately two hundred grams of moisture- and ash-free lignite, one hundred grams of water, four hundred grams of hydrogenated anthracene oil solvent and carbon monoxide gas were reacted in both cold-charge and hot-charge experiments. The oil yields and overall conversions were determined by extraction with cyclohexane and tetrahydrofuran, respectively. Maximum operating pressures ranged from 3670 psig to 3925 psig and the maximum reaction temperature was approximately 420°C in all cases.

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CHAPTER I

INTRODUCTION

Even with the current glut of oil and natural gas on the market, our national energy problems still exist. Approximately thirty-three percent of our crude oil requirements were being imported which caused a massive energy trade deficit of fifty billion dollars in 1981 (1).[1] This trade deficit has been economically damaging to the United States economy. The U.S. also finds itself in a dangerous position since most of the imported oil comes from a politically unstable region of the world (Middle East). It is estimated that the world's oil reserves will be depleted by the year 2100 if the world's oil consumption continues at such high rates (2). This has caused the search for alternative sources of energy to increase dramatically in the United States. The high energy density and ease of transporting make liquid fossil fuels the most preferable alternate energy source.

One source of these liquid fuels is available in the United States' large coal resources which are estimated at well over 2000 billion tons (3). Of these resources, North Dakota contains 350 billion tons of lignite making the state the largest single reservoir of coal in the U.S. (4). Because of its high reactivity and moisture content, relative abundance and its ease in strip mining, lignite is especially suitable

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[1] Numbers in parentheses refer to items on the List of References at the end of this paper.

for certain coal liquefaction processes.

From a chemical viewpoint, the principal differences between coal and petroleum are ultimately all due to the much lower hydrogen to carbon (H/C) ratio of coal (approximately 0.7 as against more than 1.2 for petroleum); and it is therefore possible to transform coal into liquid hydrocarbons by direct hydrogenation. Most direct coal liquefaction processes react coal, a hydrogen donor solvent, and large quantities of hydrogen gas in the presence of a catalyst at high temperature and pressure. The high temperatures cause the coal to fragment and the resulting coal radicals react with hydrogen to produce lower molecular weight molecules with higher H/C ratios. The second generation direct liquefaction processes currently being developed are descendent from the Bergius hydrogenation process developed in pre-World War II Germany (5). Some of these major liquefaction processes include the H-Coal, Exxon Donor Solvent (EDS), Synthoil, and the Solvent Refined Coal (SRC) processes (6).

In order to eliminate the need for expensive hydrogen gas that the above liquefaction processes require, the CO-STEAM process was developed using carbon monoxide or synthesis gas (a mixture of carbon monoxide and hydrogen) and the inherent moisture present in the coal to produce lower molecular weight molecules and increase the H/C ratio. The use of carbon monoxide and steam has been found to give similar or better conversions and oil yields for low-rank coals than does hydrogen under similar conditions (7).

The intended purpose of this research is to determine the effects of temperature history on the liquefaction of a North Dakota lignite using carbon monoxide and steam.

CHAPTER II

HISTORY OF THE CO-STEAM PROCESS

Early work concerning the use of carbon monoxide and water to liquefy coal dates back to 1921 when F. Fischer reported that higher yields of ether-soluble material could be obtained from coal when carbon monoxide and water were used than when hydrogen was used under similar conditions (8). Fischer suggested that the higher conversions were caused by the liberation of nascent hydrogen generated by the water-gas shift reaction. Relatively low conversions (∿35%) and the discovery of the Fischer-Tropsch reaction caused the carbon monoxide and water approach to be ignored after 1925.

Research in the use of carbon monoxide and water to liquefy low rank coals was begun at the Pittsburgh Energy Technology Center (PETC) in 1968 using batch autoclave experiments (9,10). The objective of this research was to convert lignite into low-sulfur fuel oil. Appell and co-workers at the PETC reported that conversions of lignite into benzene soluble material were higher using carbon monoxide and steam than those obtained using hydrogen at similar conditions. The use of carbon monoxide and steam was also found to give higher rates of solubilization than was possible when using hydrogen even at higher pressures. At short contact times (approximately 10 min.), the solubilization of lignite using carbon monoxide and steam was found to occur at approximately twice the rate as compared with using hydrogen. It was also reported that the rate of formation of benzene-soluble material using carbon monoxide and water decreased with increasing rank of the coal and with increasing oxidation of the coal (11). The conversion of lignite increased with the carbon monoxide and steam partial pressures up to an initial cold pressure of 1500 psig (11). This initial pressure usually resulted in an operating pressure near 5000 psig. The optimal temperature range was found to be 380-400°C (11).

It was postulated that the increased conversions of the lignite were caused in part by: "A) hydrogenation with activated hydrogen produced "in situ" by the water-gas shift reaction, B) the introduction of alkyl groups, C) the unique ability of carbon monoxide to cleave certain types of bonds or to inhibit condensation reactions leading to benzene-insoluble materials" (12). The inherent alkaline material in the lignite was found to react with carbon monoxide to produce formates which can donate hydrogen to the lignite. The reactivity of carbon monoxide was later thought to be because of its ability to remove cross-linking more than any ability to cleave bonds in lignite (7).

In subsequent work at the PETC, carbon monoxide was replaced with synthesis gas. Synthesis gas was used to reduce the cost of the reaction gas and to improve the viscosity and molecular weight characteristics of the product slurry so that recycle operations could be used in a continuous process. Increasing the temperature and pressure reduced the viscosity of the product slurry but also gave reduced oil yields. Optimal conditions for obtaining acceptable oil yields with the desired low viscosity was a temperature of 450°C and operating pressures greater than 3000 psig (7).

A 4 lb/hr continuous process unit (CPU) was developed and run at the PETC using carbon monoxide, and 0.7 H_2 : 1.0 CO and 3.0 H_2 : 1.0 CO mole ratios of synthesis gas (13). Higher pressures increased oil yields using any of the three reducing gases. Increasing the temperature above 400°C was reported to adversely affect the oil yields for the carbon monoxide atmosphere and have no effect on the oil yields using the 0.7 H_2 : 1.0 CO synthesis gas. The oil yields increased with increasing temperature using the 3.0 H_2 : 1.0 CO synthesis gas.

The Colorado School of Mines did research using carbon monoxide and steam in the liquefaction of a West Virginia bituminous coal (14). Results indicated that conversion of coal to a benzene-soluble material increased with increasing reaction temperature in the investigated temperature range of 375 to 475°C. The removal of sulfur was found to increase with increasing temperature but the removal was not as high as was found when using hydrogen.

Berg and his group at Montana State University have also been researching coal liquefaction using carbon monoxide (3,15,16). The results obtained indicated that increasing pressure and temperature will increase the conversion of subbituminous coal to a benzene-soluble material. Increasing temperature also caused the gas yield to increase. Prior oxidation of the coal was found to decrease conversions. Sodium carbonate and other alkaline materials were shown to catalyze the water-gas shift reaction and solubilization reactions thereby increasing the conversions.

Work on solvent-hydrogenation of lignite at the University of North Dakota (UND) was initiated in 1965 under the sponsorship of the Great Northern Railway Co. (now merged into the Burlington Northern)(17).

From 1965 to 1970, batch autoclave runs using carbon monoxide and/or hydrogen were carried out in an effort to provide engineering data necessary for the construction of a CPU. After interim support from the Pittsburg and Midway Coal Mining Company, a 5 year contract was negotiated in April of 1972 between the UND Chemical Engineering Department and the U.S.D.I. Office of Coal Research for a comprehensive research program of lignite technology (17). This program (Project Lignite) operated a 0.6 ton coal/day process development unit (PDU) designed for the continuous donor solvent liquefaction of lignite in a carbon monoxide and/or hydrogen atmosphere to produce a low sulfur and ash fuel (4,18). This fuel known as Solvent Refined Lignite (SRL) had a melting point between 150 and 200°C and could either be catalytically hydrogenated to a premium liquid fuel or used directly as a boiler fuel.

The CO-STEAM liquefaction research facilities established at the Grand Forks Energy Technology Center (GFETC) since 1975 have included a unique hot-charge and time-sampled batch autoclave system, a five lb. coal/hr CPU for studying lined out operation in various reactor flow configurations, and an array of analytical instrumentation for determining elemental and molecular compositions (19,20). From the work done on the hot-charge time-sampled batch autoclave, the average molecular weight of the product slurry was found to decrease with increasing temperature. Increasing temperature also caused an increase in gas production (methane) most of which came from the solvent and not the coal itself. This highlights the fact that residence times should be kept to a minimum to avoid losses of solvent to the production of methane. Experimental work was also done on the rates of reaction using carbon monoxide and/or hydrogen. This work showed that carbon monoxide undergoes reactions with lignite that are kinetically more favorable than those using hydrogen in the temperature range of 350 to 480°C (21). It was also shown that the rate controlling step in the liquefaction process appeared to be the rate of chemical reaction (19).

Various runs were made on the CPU to determine the effects of temperature and slurry-coal concentration on the liquefaction yields and product quality (20). The results indicated that the coal-slurry concentration had no effect on the conversion of lignite into a tetrahydrofuran (THF) soluble material. The conversion of lignite was found to increase with increasing temperature using a equimolar synthesis gas. Increasing the reaction temperature resulted in a decrease in the average molecular weight of the product slurry and an increase in the yield of hydrocarbon gases (20).

CHAPTER III

EXPERIMENTAL PROCEDURES AND CALCULATIONS

Materials

The lignite used in this investigation was obtained from the Indian Head Mine of the North American Coal Company located near Zap in Mercer County, North Dakota. The size distribution, proximate and ultimate analyses of the Zap lignite are given in Table 1. To keep the fraction of water present in the feed slurry constant, the as-received lignite was air dried from approximately 31.5 weight percent to 30.0 weight percent moisture. After air drying the lignite was double-wrapped in plastic bags with as much air as possible forced out of the bags. This was done to prevent additional drying and air oxidation of the lignite.

The solvent used in the feed slurry was a catalytically hydrogenated anthracene oil produced during Run 61 (HAO-61) from the CPU at the GFETC. Analyses of HAO-61 and the initial anthracene oil obtained from batch number four (AO-4) are shown in Table 2.

The histological grade THF, reagent grade cyclohexane, and C.P. grade methanol used in the extractions of both the lignite product and condensate from the cold traps were purchased from the Fischer Scientific Company. The carbon monoxide was obtained in 1500 psig cylinders from the Linde Division of Union Carbide.

TABLE 1

ANALYSES OF ZAP LIGNITE

Size Distribution

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

Proximate Analysis [a]

Constituent	Percent
Volatile Matter	33.6
Moisture	30.6
Fixed Carbon	28.1
Ash	7.7
TOTAL	100.0

[a] as received basis

Ultimate Analysis [b]

Constituent	Percent
Carbon	62.1
Hydrogen	3.81
Nitrogen	1.02
Sulfur	1.03
Oxygen (by diff.)	21.0
Ash	11.1

[b] moisture free basis

TABLE 2

ANALYSIS 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	1.11	1.05
Element Analysis		
Carbon, Wt. %	90.2	90.3
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 equipment used was UND's hot-charge, time-sampled batch autoclave system which has been described in more detail in Appendix 1. Appendix 1 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. The system consisted of a one gallon magnetically stirred autoclave rated at 5,100 psi at 510°C which had been adapted to allow the charging of the feed slurry to the preheated and pressurized autoclave. The autoclave had also been modified to allow samples of both the liquid and gas products to be obtained throughout the run. An in-line gas chromatograph had been installed to allow the time-sampled gases to be analyzed without intermediate handling. The slurry charge vessel was a one gallon stainless steel accumulator equipped with a movable 4-inch piston and was rated at 10,000 psi at room temperature. The gas compression system consisted of two 2.5 gallon piston accumulators also rated at 10,000 psi at room temperature. A high-pressure, positive-displacement, packed-plunger, metering hydraulic pump was used to supply the high pressure oil to both the slurry charge and gas compression systems. The quench vessel used was a 2.5 gallon autoclave.

Slurry Preparation

The moisture and ash contents of the Zap lignite were determined by the American Society for Testing Materials (ASTM) procedures D3173 and D3174, respectively (22,23). From the moisture and ash determinations, enough feed slurry with a water to moisture- and ash-free (MAF) lignite to HAO-61 solvent ratio of 100 : 200 : 400 was prepared allowing a total of approximately 200 gms MAF lignite to be charged. The feed slurry was prepared using a top loading balance which weighed accurately to the nearest tenth of a gram.

Procedure for Cold-Charge Runs

In cold-charge runs, the feed slurry was placed directly into the autoclave and the autoclave was then sealed and evacuated of gas. Sufficient carbon monoxide to obtain an initial pressure of 1050 psig was charged to the autoclave and the heaters and stirrer were turned on. To have the temperature peak at the desired maximum temperature of 420°C in the slow cooling runs, it was necessary to shut the heat off 8°C before reaching 420°C. The insulation was removed from the head of the autoclave as soon as the maximum temperature was reached and the autoclave was allowed to cool to 204°C at which point the product gas was removed.

After the product gas was removed, 400 psig of dry nitrogen was charged to the autoclave and the autoclave was allowed to cool overnight. The next day, the product slurry was placed in a preweighed sample container and the remaining product slurry residue was collected using preweighed disposable wipes.

Procedure for Hot-Charge Runs

In hot-charge runs, the autoclave was sealed, evacuated, and heated to the hot-charge temperature. The carbon monoxide was slowly metered into the preheated autoclave and the autoclave temperature was allowed to stabilize 20°C over the desired hot-charge temperature to compensate for the temperature drop which occurred when the feed slurry was charged to the autoclave. The reaction time for the hot charge runs was defined as starting when the feed slurry had reached a temperature 20°C below the desired hot-charge temperature. This reaction time was determined by measuring the time necessary for the first run (M4), which was hot-charged at 320°C, to heat up from 300°C to 420°C. The reaction time was determined to be 34 minutes and this time was used as the reaction time for the remaining two hot-charge runs. The second run was hot-charged at 360°C and heated to 420°C. The product slurry was then held at 420°C until the total time above 340°C was 34 minutes. The third run was hot-charged at 420°C and held above 400°C for the 34 minute reaction time. After the 34 minute reaction period, the product slurry was discharged from the bottom of the autoclave into a quench vessel where the slurry and gas were cooled to room temperature in a matter of minutes.

After allowing 30 minutes for the product slurry and gas temperatures to stabilize, the product gas was withdrawn from the quench vessel. The product slurry was removed from the quench vessel and stored in a preweighed sample container. Residue remaining in the quench vessel and the autoclave was then collected using preweighed disposable wipes. Feed slurry remaining in the charge vessel and valves was also collected using preweighed disposable wipes in order to determine the mass of slurry charged to the autoclave.

Analytical Procedure

The product slurry and gas were analyzed according to the flowsheet shown in Figure 1. The product gas was analyzed within 24 hours of a run to reduce any air dilutions that might occur because of an undetected leak in the sample bag. The product slurry was well mixed using a Fischer steadi-speed adjustable stirrer to ensure a uniform sample was obtained for each analysis. In between analyses, the air-tight sample container was kept sealed to prevent oxidation or drying of the product slurry.

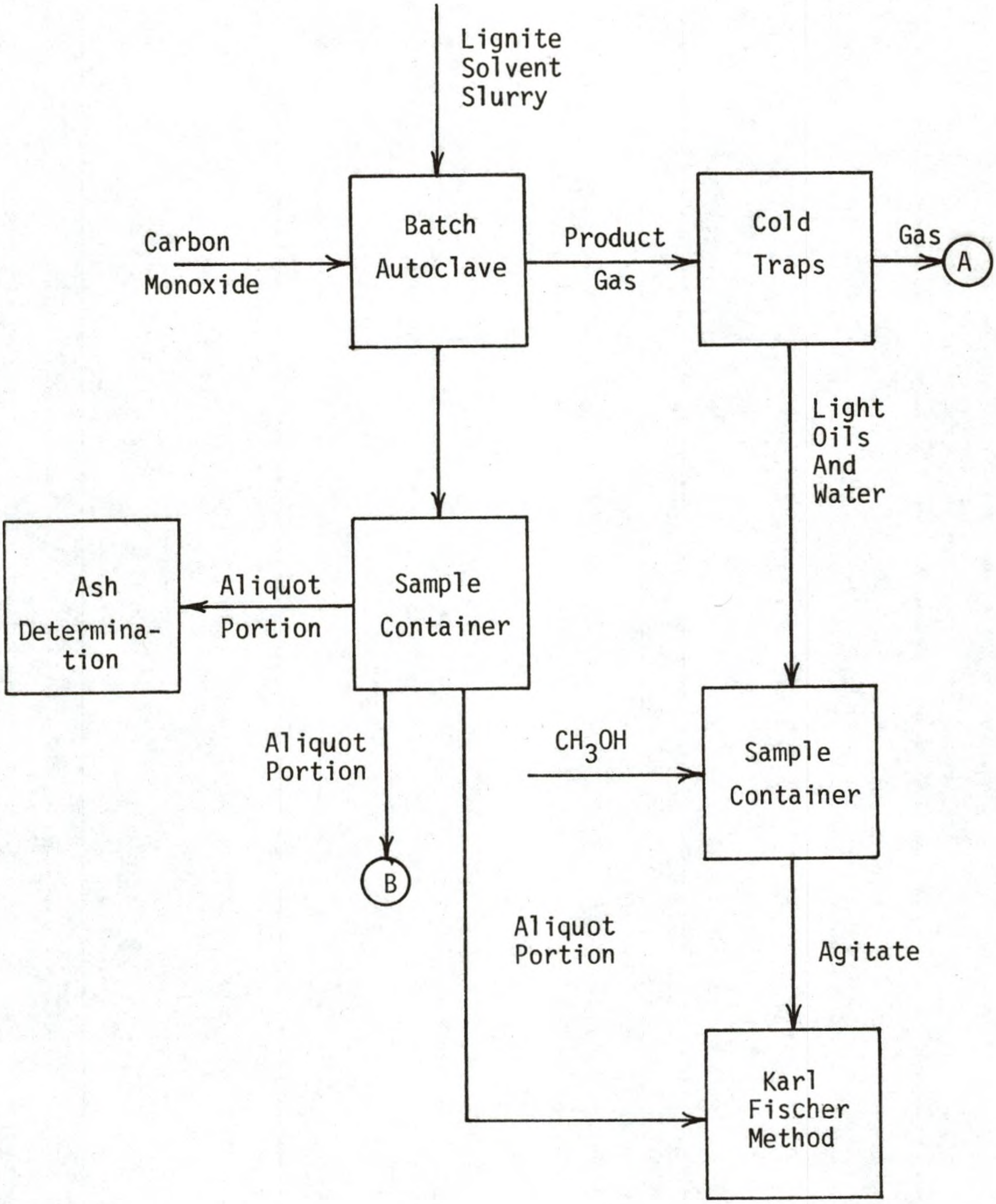


Figure 1. Analytical Procedure Flowsheet.

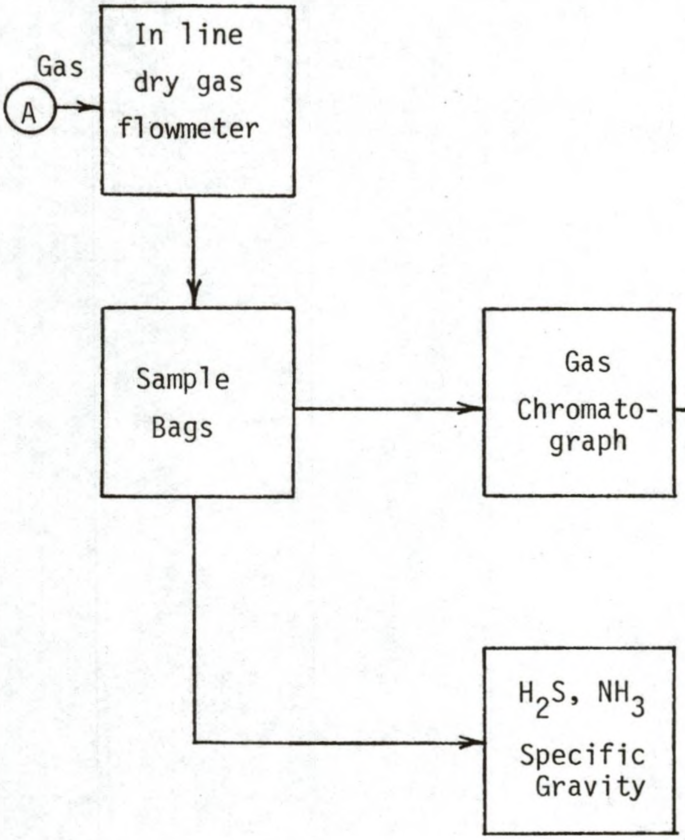


Figure 1. Continued.

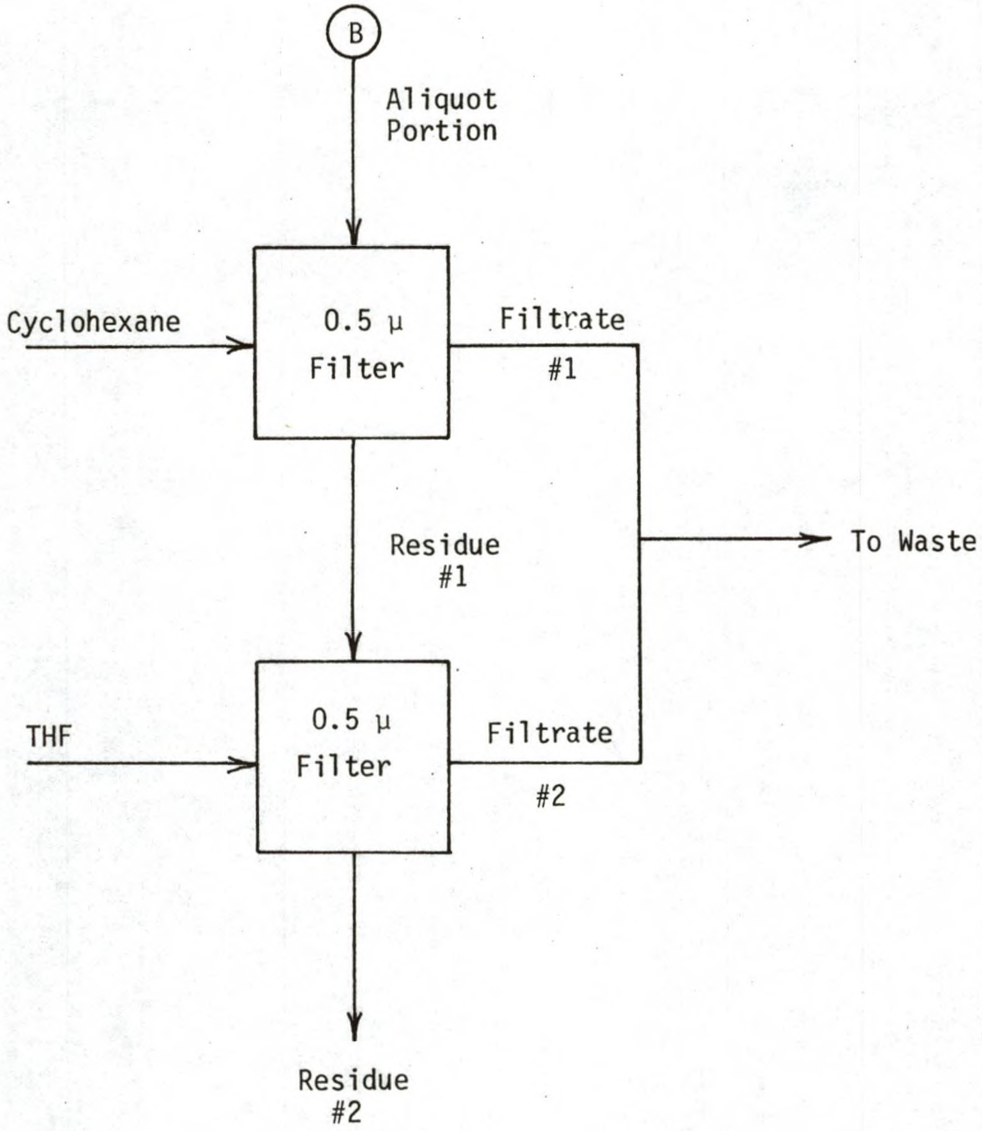


Figure 1. Continued.

Product Gas Analysis

The product gases were bled from the autoclave or quench vessel through three cold traps in series; the first was in an ice bath while the next two were in isopropanol-dry ice baths. Then the gas was drawn through a dry gas flowmeter and totalizer into a 15 cu. ft. sample bag. A Hewlett Packard F and M Scientific 700 Laboratory Chromatograph with Porapak Q and 5 Å Mole Sieve columns were used to determine the concentrations of H_2, N_2, O_2, CO, CO_2, CH_4, C_2H_6 and C_3H_8 in the product gas. Hydrogen sulfide was determined using a modified ASTM D2385 method (24). Ammonia was measured using a modified Nessler's method for ammonia determination in aqueous solution (25). Specific gravity of the gas was determined by the Reanault method using a gas density bulb (26). Appendix 2 contains a summary of the laboratory procedures used in the hydrogen sulfide, ammonia, and specific gravity determinations. After all analyses were performed, the volume of the remaining gas was measured using a Scientific Precision Co. Wet-test flowmeter to compare the volume with that measured using the in-line volumetric flowmeter.

Cyclohexane and THF Extractions

Approximately 1.0 grams of product slurry weighed to the nearest 0.1 mg was extracted with approximately 100 ml. of cyclohexane. The extract was pressure filtered through a preweighed 0.5 micron filter (Millipore, type FH) using dry nitrogen gas at 25 psig. The residue was washed with additional cyclohexane (approximately 100 ml.) until the wash liquid was clear. The filter cake was then dried in an oven at 105°C for 15 minutes and weighed. The residue and filter paper from the cyclohexane extraction were extracted with 100 ml. of THF and then pressure filtered through another preweighed 0.5 micron filter using dry nitrogen gas at 25 psig. The filter cake was then washed with more THF (about 100 ml.) until the wash liquid was clear. The filter cake and filter papers were dried in an oven at 105°C for 15 minutes and reweighed.

Moisture Determinations

The Karl Fischer method was used to determine the moisture content of both the product slurry and the condensate collected in the cold traps (27). In determining the moisture content of the product slurry, a known weight of the slurry was added to an anhydrous chloroform-methanol solution using a disposable pipet. This solution was then titrated with Karl Fischer reagent until the water-free endpoint was reached. For the slow-cooling runs in which the gas was taken off at 204°C thereby causing the product slurry to contain little water, the titrations were performed in a Photovolt Aquatest IV automatic titrator. The quenched runs which would contain more water were titrated using a standard 50 ml. buret.

In determining the moisture content of the condensate from the cold traps, the condensate was mixed with reagent grade methanol of known moisture content in approximately a 1 : 15 weight ratio. The condensate and methanol were well mixed and allowed to sit for several hours to ensure that all the water was absorbed by the methanol. A known weight of the methanol was added to the anhydrous chloroform-methanol solution using a micropipette. This solution was titrated with Karl Fischer reagent in a Photovolt Aquatest IV automatic titrator. All titrations were repeated until a minimum of three trials differing by less than two percent were obtained.

Net Yield and Overall Conversion Calculations

The net yield of oil was defined as the weight ratio of the MAF cyclohexane solubles to the MAF lignite charged expressed as a percent. Cyclohexane was chosen as the solvent based on previous work done at UND (28). It was found that the oil yield determined by the extraction with cyclohexane closely correlated to the oil yield determined by microdistillation at 250°C and 1 torr.

The net yield of SRL was the weight of material soluble in the THF but insoluble in cyclohexane expressed as a percent of the MAF lignite charged.

The net yield of insoluble organic matter (IOM) was defined as the weight of the ash-free portion of the THF insolubles expressed as a weight percent of the MAF lignite charged.

The water, ash, and gas net yields were defined by the following general equation:

$$\text{Net Yield} = \frac{\text{mass component out} - \text{mass component in}}{\text{mass MAF lignite in}}\ (100)$$

The overall conversion was defined as 100 minus the IOM net yield or from the following equation:

$$\text{conversion} = \frac{\text{mass MAF lignite in} - \text{mass MAF THF insolubles out}}{\text{mass MAF lignite in}}\ (100)$$

CHAPTER IV

RESULTS AND DISCUSSION

The experimental conditions for each run are summarized in Table 3. A summary of the net yields, overall conversions, and the material balance closure for each run is given in Table 4. The sample net yield calculations for run M1 are shown in Appendix 3. The computer program used to perform the net yield calculations and the definitions of the symbols used in the program are shown in Appendix 4. Appendix 5 contains the data sheets and the computer printouts for each run.

Reproducability

The closure of the material balance was used to check the experimental technique. Closures ranged between 95.1 to 105.0 percent. Net yields were normalized by assuming that any loss or "gain" of material would be proportionally distributed between the products. The normalization calculations are also shown in Appendix 3. As a check on experimental technique, two identical cold-charge, slow-cooling runs (M1 and M2) were made to compare the results obtained. Since the water and oil net yields were drastically different, another cold-charge, slow-cooling run (M7) was performed and the results were found to closely agree with the results obtained in run M1. Subsequently, the results from run M2 were disregarded, although the results were shown in Table 4. The discrepancy in run M2 was probably the result of a faulty moisture determination on the cold trap condensate.

TABLE 3

SUMMARY OF EXPERIMENTAL CONDITIONS

Run #	Temperature History	Lignite Moisture	Lignite Ash	Maximum Temp. (°C)	Maximum Pressure (psig)
M1	Cold charge heat to 420°C slow cooling	29.9%	7.98%	419°C	3750
M2	Cold charge heat to 420°C slow cooling	30.7%	7.88%	417°C	3670
M3	Cold charge heat to 420°C quench immediately	30.3%	7.93%	420°C	3675
M4	Hot charge at 320°C heat to 420°C quench immediately reaction time = 34 min.	29.9%	7.97%	420°C	3750
M5	Hot charge at 360°C heat to 420°C hold at 420°C until total reaction time = 34 min., quench	29.9%	7.97%	423°C	3890
M6	Hot charge at 420°C hold at 420°C until total reaction time = 34 min., quench	29.5%	8.03%	420°C	3925
M7	Cold charge heat to 420°C slow cooling	28.7%	8.11%	418°C	3840

TABLE 4

SUMMARY OF NORMALIZED NET YIELDS, OVERALL CONVERSIONS AND MATERIAL BALANCE CLOSURES FOR EACH RUN

		Normalized Yields Wt % of MAF Lignite							
Run #	Temeprature History	H_2O	Oil	SRL	IOM	Ash	Gas	% Conversion	% Closure
M1	cold charge heat to 420°C slow cooling	-27.9	27.1	37.7	7.5	0.1	55.4	92.5	95.1
M2	cold charge heat to 420°C slow cooling	-10.6	10.2	41.7	8.2	-0.6	51.0	91.8	105.0
M3	cold charge heat to 420°C quench immediately	-12.8	16.0	45.7	19.4	-0.5	32.2	80.6	100.6
M4	hot charge at 320°C heat to 420°C quench immediately	- 9.9	19.1	45.4	16.4	-0.1	29.0	83.6	100.6
M5	hot charge at 360°C heat to 420°C hold at 420°C until reaction time = 34 min. quench	-20.5	26.9	40.5	9.1	0.1	43.9	90.9	99.8
M6	Hot charge at 420°C hold at 420°C until reaction time = 34 min. quench	-22.2	34.3	40.2	8.9	0.2	38.6	91.1	97.3
M7	cold charge heat to 420°C slow cooling	-28.2	24.6	43.1	8.6	0.4	51.4	91.4	99.1

Effects of Temperature History

The cold-charge, slow-cooling runs (M1 and M7) remained at reaction temperatures greater than 380°C for a considerably longer period of time than the hot-charge runs. In spite of the longer reaction time, the oil yields and overall conversions of the cold-charge runs remained essentially unchanged while the gas yields and the consumption of water (negative net yields) increased as compared to similar results obtained from the hot-charge runs M5 and M6. The similar conversions agree with the results obtained by Appell and co-workers in which it was reported that the solubilization reaction is essentially complete in 15 to 20 minutes at 380 to 400°C in the presence of a good donor solvent (11). Since the slurry of hot charge runs M5 and M6 were above 380°C for at least 26 minutes, the reaction times were long enough to have caused the liquefaction reactions to reach completion.

The effects of quenching can be seen by comparing the cold charge run that was quenched immediately at 420°C (M3) with the cold charge, slow-cooling runs (M1 and M7). The results show that when the product slurry was quenched, the overall conversion and the net yields of the oil and gas and the consumption of water all decreased. These results were due to the fact that the product slurry only spent approximately 10 minutes in the 380 to 420°C temperature range instead of the necessary 15 to 20 minutes for the solubilization reactions to have reached completion.

The reason for hot-charging the feed slurry to the preheated autoclave was to considerably reduce the time necessary for the slurry to reach the hot-charge temperature. This reduced the effects of any reactions which were occurring while the slurry was being slowly heated to the hotter reaction temperature. Figure 2 shows a temperature versus time schematic for a typical hot-charge run (M6 in this case). It can be seen that in approximately 2.4 minutes, the slurry had reached the desired reaction temperature.

Based on the small differences between the conversions and the net yields of the cold charge run (M3) and the run hot charged at 320°C (M4) where both runs were quenched immediately upon reaching 420°C, it appears that the liquefaction reactions including the water-gas shift reaction were kinetically unreactive below 320°C. The small differences in the product gas composition for these two runs as shown in Table 5 also tend to support the observation that the water-gas shift reaction is relatively inactive below 320°C. Prior studies have also shown the inactivity of the water-gas shift reaction below 325°C (2). Since the equilibrium constant for the water-gas shift reaction increases with decreasing temperature and the rate of reaction decreases with decreasing temperature, the rate of reaction for the water-gas shift reaction was found to be the controlling step below 320°C.

The effects of increasing the hot charge temperature can be found by comparing runs M4, M5 and M6. Increasing the hot charge temperature from 320 to 360°C (runs M4 and M5 respectively) increased the overall conversion, oil and gas net yields and the consumption of water while decreasing the net SRL yields. The increased oil yields and conversions were due to the increased time the product slurry of run M5 was above 360°C as compared to the product slurry of run M4. This result suggests that the liquefaction reactions are kinetically more favorable above 360°C.

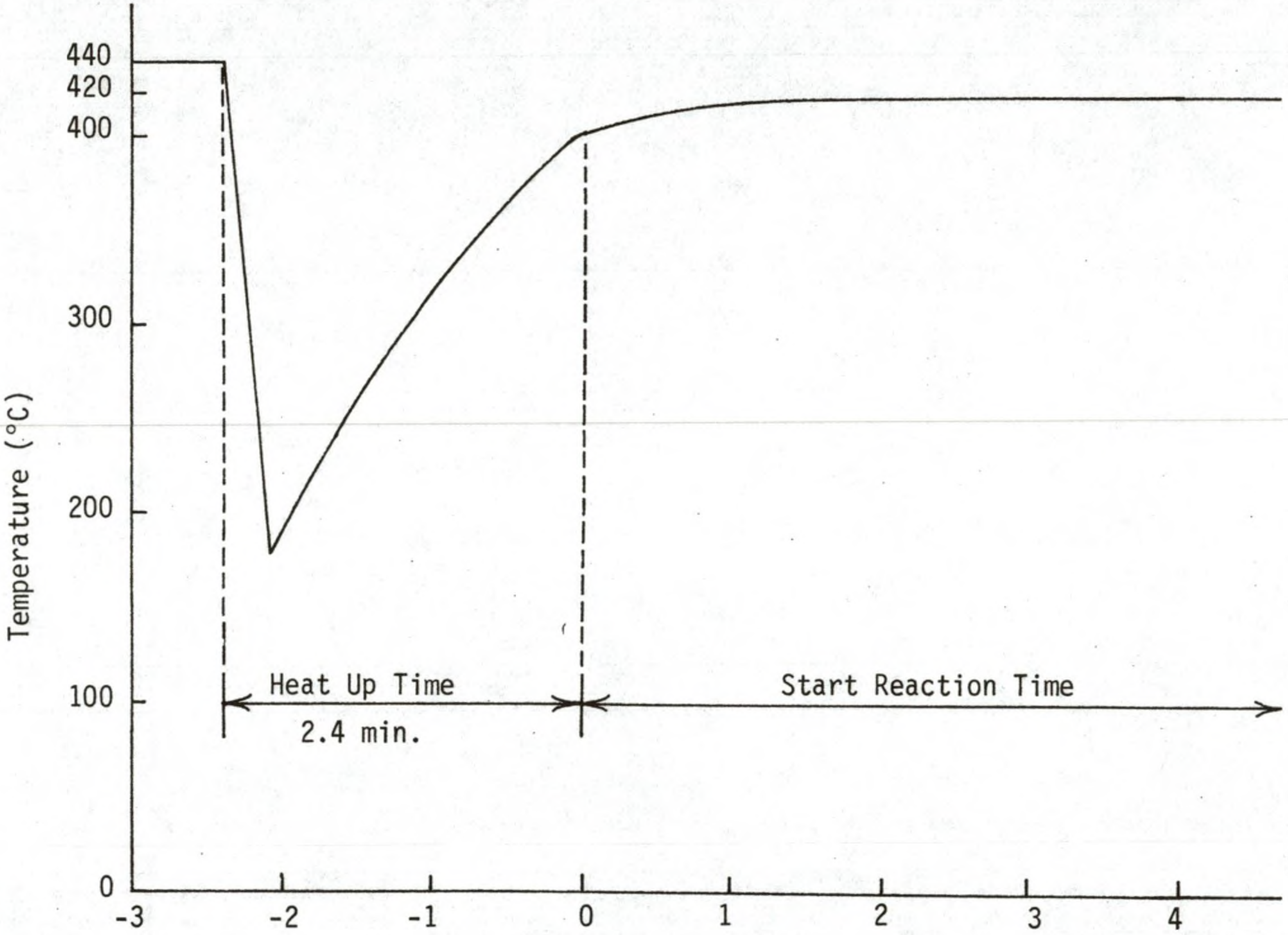


Figure 2. Typical Temperature Versus Time Effect of Hot-Charging on the Feed Slurry for Run 6.

TABLE 5

PRODUCT GAS COMPOSITION FOR RUNS M3 AND M4

	Normalized Airfree Composition, mol % Run	
Component	M3	M4
CO_2	23.5	23.3
C_2H_6	0.13	0.08
C_3H_8	0.04	0.04
H_2	8.93	6.37
CH_4	0.57	0.53
CO	66.8	69.5
H_2S	0.01	0.18
NH_3	0.01	0.01
TOTAL	100.00	100.00

Increasing the hot-charge temperature from 360 to 420°C did not significantly change the conversion or the net yields of SRL, IOM, and water, but it did appear to have decreased the gas yields and increased the oil yields. These results disagree with previous results reported in literature which state that increasing the time at higher reaction temperatures should increase the gas yields at the expense of the oil yields (7,11,19). When the normalized net yields of run M5 were compared to the net yields of run M6 which were calculated by assuming all the lost material in the material balance was from the product gas as shown in Table 6, the net yields were essentially the same for both runs. This similarity was consistent with the previous results obtained from literature. Based on the probability that most of the lost material was from the product gas, it would be concluded that increasing the hot-charge temperature from 360 to 420°C had an insignificant effect on the overall conversions and oil yields. This insignificant difference between the two runs was caused by the extended time both slurries were above the reaction temperature of 380°C. The times for both runs were well above the reaction time necessary for the liquefaction reactions to reach completion. A comparison of the normalized net yields for run M6 and the net yields for run M6 determined by assuming all lost material was product gas is given in Appendix 6.

TABLE 6

NORMALIZED NET YIELDS FOR RUN M5 AND THE NET YEILDS CALCULATED FOR RUN M6 ASSUMING ALL LOST MATERIAL WAS PRODUCT GAS

			Net Yields as Wt % MAF Lignite Charged				
Run #	H_2O	Oil	SRL	IOM	Ash	Gas	% Conversion
M5	-20.5	26.9	40.5	9.1	0.1	43.9	90.9
M6	-22.9	28.0	39.1	8.7	-0.2	47.3	91.3

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

1) The liquefaction reaction rates including that of the water-gas shift reaction below 320°C were sufficiently slow so as to be unimportant.

2) The conversions and oil yields of the cold-charge, slow-cooling runs were similar to the conversions and oil yields obtained for runs hot-charged above 360°C because the product slurry was above 380°C for a sufficiently long time to allow the liquefaction reactions to reach completion.

3) Increasing the hot-charge temperature from 320 to 360°C resulted in increased oil yields and conversions.

4) Increasing the hot-charge temperature from 360 to 420°C did not change the conversions nor the oil yields.

Recommendations

1) In order to determine more precisely the effects of hot-charging at 360°C and heating to 420°C, one run should be made hot-charging at 360°C and quenching the product slurry as soon as it reaches 420°C. Also a hot-charge at 420°C for the same reaction time should be run for comparative purposes.

2) Using carbon monoxide as the reducing gas, the minimum

reaction time necessary for the liquefaction reaction to reach completion should be determined.

3) Because of the necessity to condense larger volumes of water in slow-cooling runs, the free volume for condensate collection should be increased.

APPENDICES

APPENDIX 1

UND HOT-CHARGE TIME-SAMPLED 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.(1)

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 temperature 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, Environmental and Safety Program.

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

Figure 3 is an overall flow diagram of the components of the autoclaves and primary support equipment. Figure 3 is broken down into seven areas as indicated by the dotted lines.

Figure 4 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 5 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 6 is a detailed diagram of Area IV, the gas compression system. The major components of this system are two 1-½ 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 3. 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 versitility as the slurry charge system with the additional advantage of continuous feed capability when the two accumulators are used alternately.

Figure 7 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 3 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 8 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 measurements 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 quantity 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 9 is a drawing of the GC sample loops and hot box. The sample loops are also shown diagramatically in Figure 3, 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 10 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.(2) 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 10. Figure 11 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 ½-inch steel cables. The barricade itself is constructed of ½-inch Cor-ten steel plate. The barricade and blast mats have been calculated to provide protection against shrapnel.

Federal health and safety guidelines(3) 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 10, 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.

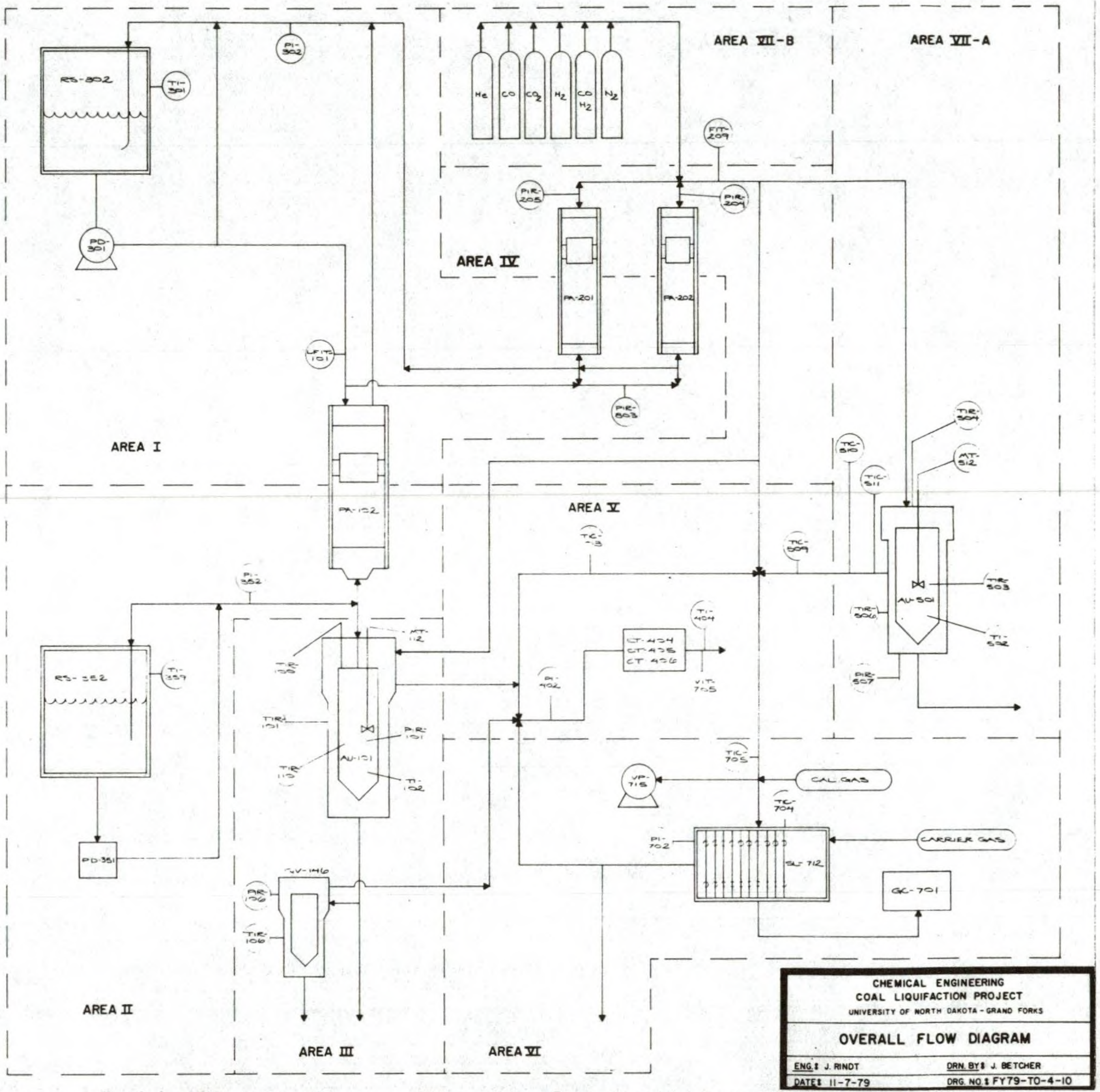
AU - AUTOCLAVE
AC - CARRIER GAS CLEANUP APPARATUS
BA - BLATTER ACCUMULATOR
CP - COLLECTION PORT
CT - COLD TRAP
CV - CHECK VALVE
DV - DOUBLE VALVE
FI - FLOW INDICATOR
FIT - FLOW INDICATOR TOTALIZER
FL - FILTER
FM - FINE METERING VALVE
FV - FLUSH VALVE
GC - GAS CHROMOTOGRAPH
GV - GAS SAMPLING VALVE
MT - MAGNETIC TACKOMETER PICKUP
PA - PISTON ACCUMULATOR
PD - POSITIVE DISPLACEMENT PUMP
PG - BACK PRESSURE REG. VALVE
PI - PRESSURE INDICATOR
PIR - PRESSURE INDICATOR RECORDER
PR - PRESSURE RELIEF VALVE
PV - SOLENOID VALVE
QV - QUENCH VESSEL
RS - RESERVOIR
RSI - REMOTE SAMPLE INLET
RV - PRESSURE REGULATOR VALVE
RVS - REMOTE VACUUM SOURCE
SB - SURGE CHECK BYPASS VALVE
SC - SURGE CHECK VALVE
SL - SAMPLE LOOP
SP - SURGE PISTON ACCUMULATOR
ST - SAMPLE TRAY
SV - SHUTOFF VALVE
TI - TEMPERATURE INDICATOR
TIC - TEMPERATURE INDICATOR CONTROLLER
TIR - TEMPERATURE INDICATOR RECORDER

PIPE SCHEDULE

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

FITTING SCHEDULE

1	20BFX9966	Bulkhead	(9/16" - 20K) A.E.
2	20M96K6	Adaptor	(9/16" 20K + 3/8" 20K) A.E.
3	6M49N6	Adaptor	(1/4" NPTM - 9/16" 20K) A.E.
4	6M49N6	Adaptor	(1/4" NPTM - 9/16" 20K) A.E.
5	6M129N6	Adaptor	(3/4" NPTM - 9/16" 20K) A.E.
6	6MX912K8	Adaptor	(3/4" NPTF - 9/16" 20K) A.E.
7	6M46N6	Adaptor	(1/4" NPTM + 3/8" 20K) A.E.
8	20M96K6	Adaptor	(9/16" 20K - 3/8" 20K) A.E.
9	6M169N6	Adaptor	(1" NPTM - 9/16" 20K) A.E.
10	20BFX666	Bulkhead	(3/8" 20K) A.E.
11	6M126N6	Adaptor	(3/4" NPTM - 3/8" 20K) A.E.
12	20M96B6	Adaptor	(9/16" 60K + 3/8" 20K) A.E.
13	60M94B3	Adaptor	(9/16" 60K - 1/4" 60K) A.E.
14	6M166N6	Adaptor	(1" NPTF - 3/8" 20K) A.E.
18	6M126N6	Adaptor	(3/4" NPTM + 3/8" 20K) A.E.
19	60BF4433	Bulkhead	(1/4" 60K) A.E.
20	6M166N3	Adaptor	(1" NPTM - 3/8" 60K) A.E.
21	60M46B3	Adaptor	(1/4" 60K + 3/8" 60K) A.E.
22	6M44N3	Adaptors	(1/4" NPTM + 1/4" 60K) A.E.
23	6M84N3	Adaptors	(1/2" NPTM - 1/4" 60K) A.E.
24	6M44B2	Adaptor	(1/4" 60K + 1/4" 10K) A.E.
25	QC6-B-600/QC6-D-600	Quick Connect	(3/8") Swagloc
26	601PC-4	Adaptor	(3/8" 11K - 1/4" 11K) Swagloc
27	601-PC/4R6-316/6TMT4-316	Adaptor	(3/8"-1/4"-TLE-1/8") Swagloc
28	6M42D1	Adaptor	(1/4" 10K - 1/8" 10K) A.E.
29	CT-4440	Tee	(1/4" 60K) A.E.
30	15M42B1	Adaptor	(1/4" 60K + 1/8" 11K) A.E.
31	15BF2211	Bulkhead	(1/8" 11K) A.E.
32	6M26C8	Adaptor	(1/4" NPTF - 1/8" 10K) A.E.
33	ST2220	Tee	(1/8" 11K) A.E.
34	SS-2-TSW-3	Weld Tee	(1/8" 11K) Cajon
35	6M44N2	Adaptor	(1/4" NPTM - 1/4" 11K) A.E.
36	6M412D8	Adaptor	(3/4" NPTF + 1/4" 11K) A.E.



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

OVERALL FLOW DIAGRAM

ENG.: J. RINDT
DRN. BY: J. BETCHER
DATE: 11-7-79
DRG. NO.: FY79-TO-4-10

Figure 3. Overall Flow Diagram of the UND Hot-Charge, Time-Sampled Batch Autoclave Facility.

AU - AUTOCLAVE
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Ⓒ	1/4" O.D.	Plastic Instrument Tubing
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Ⓖ	1/8" O.D.	11,000 psi (S.S.)
Ⓗ	1/4" O.D.	11,000 psi (S.S.)

FITTING SCHEDULE

1	20BFX9966	Bulkhead	(9/16" - 20K) A.E.
2	20M96K6	Adaptor	(9/16" 20K + 1/4" 20K) A.E.
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4	6M49N6	Adaptor	(1/4" NPTM - 9/16" 20K) A.E.
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6	6MX912K8	Adaptor	(3/4" NPTF - 9/16" 20K) A.E.
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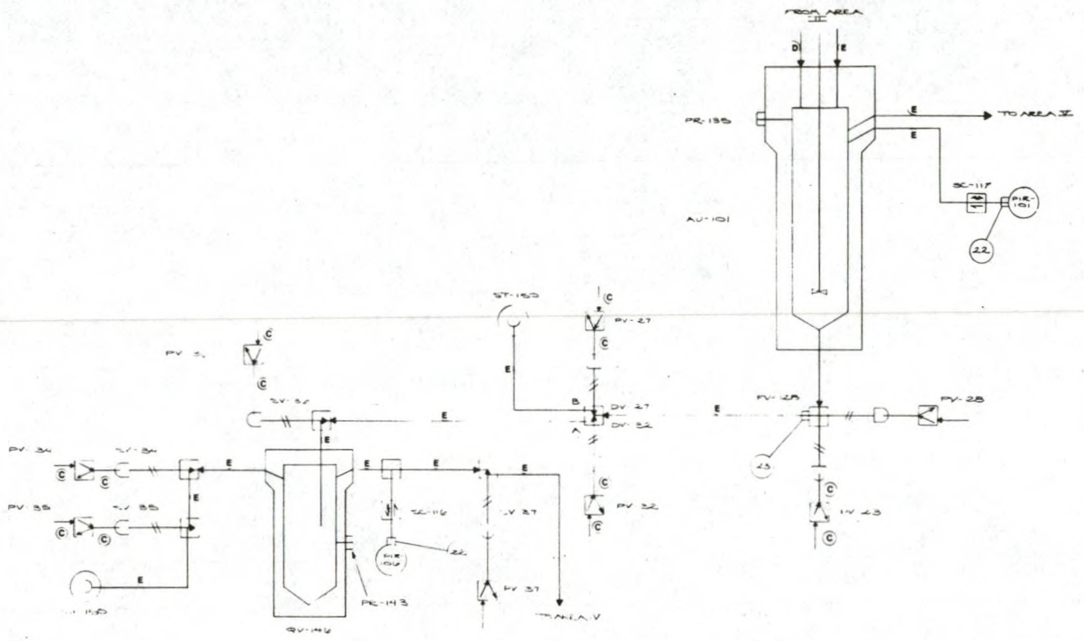
22	6M44N3	Adaptors	(1/4" NPTM - 1/4" 60K) A.E.
23	6M84N3	Adaptors	(1/2" NPTM - 1/4" 60K) A.E.
24	6M44B2	Adaptor	(1/4" 60K + 1/4" 10K) A.E.
25	QC6-B-600/QC6-D-600	Quick Connect	(1/8") Swagloc
26	601PC-4	Adaptor	(3/8" 11K - 1/4" 11K) Swagloc
27	601-PC/4R6-316/6TMT4-316	Adaptor	(3/8"-1/4"-TEE-3/8") Swagloc
28	6M42D1	Adaptor	(1/4" 10K - 1/8" 10K) A.E.
29	CT-4440	Tee	(1/4" 60K) A.E.
30	15M42B1	Adaptor	(1/4" 60K + 1/8" 11K) A.E.
31	15BF2211	Bulkhead	(1/8" 11K) A.E.
32	6M26C8	Adaptor	(1/4" NPTF - 1/8" 10K) A.E.
33	ST2220	Tee	(1/8" 11K) A.E.
34	SS-2-TSW-3	Weld Tee	(1/8" 11K) Cajon
35	6M44N2	Adaptor	(1/4" NPTM - 1/4" 11K) A.E.
36	6M412D8	Adaptor	(3/4" NPTF + 1/4" 11K) A.E.



CHEMICAL ENGINEERING
COAL LIQUIFACTION PROJECT
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AREA III

ENG.: J. RINDT
DRN. BY: J. BETCHER
DATE: 11-7-79
DRG. NO.: FY79-TO-4-13

Figure 4. Detailed Diagram of Area III, the UND Hot-Charge Autoclave Components.

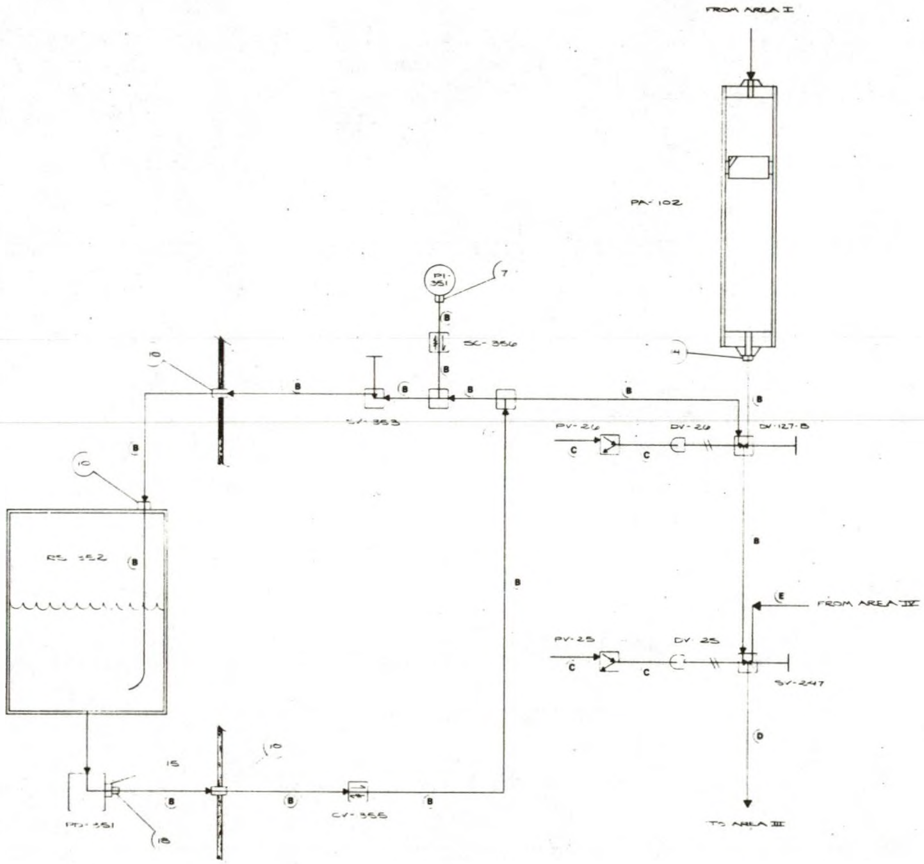
AO - AUTOCLAVE
AC - CARRIER GAS CLEANUP APPARATUS
BA - BLATTER ACCUMULATOR
CP - COLLECTION PORT
CT - COLD TRAP
CV - CHECK VALUE
DV - DOUBLE VALUE
FI - FLOW INDICATOR
FIT - FLOW INDICATOR TOTALIZER
FL - FILTER
FM - FINE METERING VALUE
FV - FLUSH VALUE
GC - GAS CHROMOTOGRAPH
GV - GAS SAMPLING VALUE
MT - MAGNETIC TACKOMETER PICKUP
PA - PISTON ACCUMULATOR
PD - POSITIVE DISPLACEMENT PUMP
PG - BACK PRESSURE REG. VALUE
PI - PRESSURE INDICATOR
PIR - PRESSURE INDICATOR RECORDER
PR - PRESSURE RELIEF VALVE
PV - SOLENOID VALVE
QV - QUENCH VESSEL
RS - RESERVOIR
RSI - REMOTE SAMPLE INLET
RV - PRESSURE REGULATOR VALVE
RVS - REMOTE VACUUM SOURCE
SB - SURGE CHECK BYPASS VALVE
SC - SURGE CHECK VALVE
SL - SAMPLE LOOP
SP - SURGE PISTON ACCUMULATOR
ST - SAMPLE TRAY
SV - SHUTOFF VALVE
TI - TEMPERATURE INDICATOR
TIC - TEMPERATURE INDICATOR CONTROLLER
TIR - TEMPERATURE INDICATOR RECORDER

PIPE SCHEDULE

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

FITTING SCHEDULE

1	20MFX9966	Bulkhead	(9/16" - 20K) A.E.
2	20M96K6	Adaptor	(9/16" 20K + 3/8" 20K) A.E.
3	6M49M6	Adaptor	(1/4" NPTM - 9/16" 20K) A.E.
4	6M49M6	Adaptor	(1/4" NPTM + 9/16" 20K) A.E.
5	6M129M6	Adaptor	(3/4" NPTM - 9/16" 20K) A.F.
6	6MX912K8	Adaptor	(3/4" NPTF + 9/16" 20K) A.E.
7	6M46M6	Adaptor	(1/4" NPTM - 3/8" 20K) A.E.
8	20M96K6	Adaptor	(9/16" 20K - 3/8" 20K) A.E.
9	6M169M6	Adaptor	(1" NPTM + 9/16" 20K) A.E.
10	20BFX666	Bulkhead	(3/8" 20K) A.E.
11	6M126M6	Adaptor	(3/4" NPTM - 3/8" 20K) A.E.
12	20M96B6	Adaptor	(9/16" 60K - 3/8" 20K) A.E.
13	60M94B3	Adaptor	(9/16" 60K - 1/4" 60K) A.E.
14	6M166M6	Adaptor	(1" NPTF - 3/8" 20K) A.E.
18	6M126M6	Adaptor	(3/4" NPTM + 3/8" 20K) A.E.
19	60BF4433	Bulkhead	(1/4" 60K) A.E.
20	6M166N3	Adaptor	(1" NPTM + 3/8" 60K) A.E.
21	60M46B3	Adaptor	(1/4" 60K - 3/8" 60K) A.E.
22	6M44N3	Adaptors	(1/4" NPTM - 1/4" 60K) A.E.
23	6M84N3	Adaptors	(1/2" NPTM - 1/4" 60K) A.E.
24	6M44B2	Adaptor	(1/4" 60K + 1/4" 10K) A.E.
25	QC6-B-600/QC6-D-600	Quick Connect	(3/8") Swagloc
26	601PC-4	Adaptor	(3/8" 11K + 1/4" 11K) Swagloc
27	601-PC/4R6-316/6TMT4-316	Adaptor	(3/8"-1/4"-TEE-1/8") Swagloc
28	6M42D1	Adaptor	(1/4" 10K + 1/8" 10K) A.E.
29	CT-4440	Tee	(1/4" 60K) A.E.
30	15M42B1	Adaptor	(1/4" 60K + 1/8" 11K) A.E.
31	15BF2211	Bulkhead	(1/8" 11K) A.E.
32	6M26C8	Adaptor	(1/4" NPTF + 1/8" 10K) A.E.
33	ST2220	Tee	(1/8" 11K) A.E.
34	SS-2-TSW-3	Weld Tee	(1/8" 11K) Cajon
35	6M44N2	Adaptor	(1/4" NPTM - 1/4" 11K) A.E.
36	6M412D8	Adaptor	(3/4" NPTF + 1/4" 11K) A.E.



CHEMICAL ENGINEERING
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AREA II

ENG.: J. RINDT
DRN. BY: J. BETCHER
DATE: 11-7-79
DRG. NO.: FY79-TO-4-12

Figure 5. Detailed Diagram of Area II, the Slurry Charge System Components.

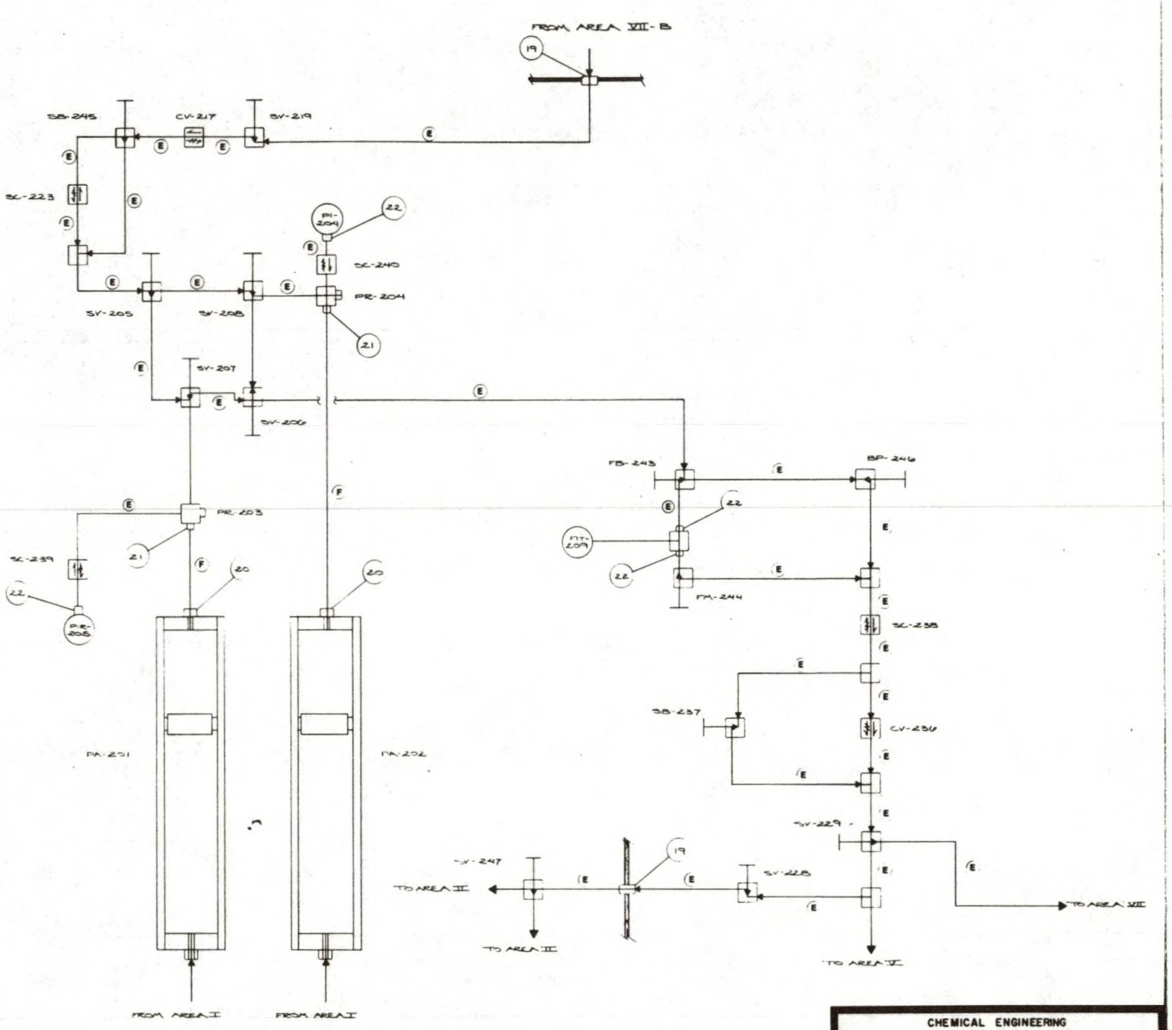
AU - AUTOCLAVE
AC - CARRIER GAS CLEANUP APPARATUS
BA - BLATTER ACCUMULATOR
CP - COLLECTION PORT
CT - COLD TRAP
CV - CHECK VALUE
DV - DOUBLE VALUE
FI - FLOW INDICATOR
FIT - FLOW INDICATOR TOTALIZER
FL - FILTER
FM - FINE METERING VALUE
FV - FLUSH VALUE
GC - GAS CHROMOTOGRAPH
GV - GAS SAMPLING VALUE
MT - MAGNETIC TACHOMETER PICKUP
PA - PISTON ACCUMULATOR
PD - POSITIVE DISPLACEMENT PUMP
PG - BACK PRESSURE REG. VALUE
PI - PRESSURE INDICATOR
PIR - PRESSURE INDICATOR RECORDER
PR - PRESSURE RELIEF VALVE
PV - SOLENOID VALVE
QV - QUENCH VESSEL
RS - RESERVOIR
RSI - REMOTE SAMPLE INLET
RV - PRESSURE REGULATOR VALVE
RVS - REMOTE VACUUM SOURCE
SB - SURGE CHECK BYPASS VALVE
SC - SURGE CHECK VALVE
SL - SAMPLE LOOP
SP - SURGE PISTON ACCUMULATOR
ST - SAMPLE TRAY
SV - SHUTOFF VALVE
TI - TEMPERATURE INDICATOR
TIC - TEMPERATURE INDICATOR CONTROLLER
TIR - TEMPERATURE INDICATOR RECORDER

PIPE SCHEDULE

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

FITTING SCHEDULE

1	20BFX9966	Bulkhead	(9/16" - 20K) A.E.
2	20M96K6	Adaptor	(9/16" 20K + 3/8" 20K) A.E.
3	6M49M6	Adaptor	(1/4" NPTM - 9/16" 20K) A.E.
4	6M49M6	Adaptor	(1/4" NPTM - 9/16" 20K) A.E.
5	6M129M6	Adaptor	(3/4" NPTM + 9/16" 20K) A.E.
6	6MX912K8	Adaptor	(3/4" NPTF + 9/16" 20K) A.E.
7	6M46M6	Adaptor	(1/4" NPTM - 3/8" 20K) A.E.
8	20M96K6	Adaptor	(9/16" 20K + 3/8" 20K) A.E.
9	6M169M6	Adaptor	(1" NPTM + 9/16" 20K) A.E.
10	20BFX666	Bulkhead	(3/8" 20K) A.E.
11	6M126M6	Adaptor	(3/4" NPTM + 3/8" 20K) A.E.
12	20M96B6	Adaptor	(9/16" 60K + 3/8" 20K) A.E.
13	60M94B3	Adaptor	(9/16" 60K + 1/4" 60K) A.E.
14	6M166M6	Adaptor	(1" NPTF + 3/8" 20K) A.E.
18	6M126M6	Adaptor	(3/4" NPTM + 3/8" 20K) A.E.
19	60BF4433	Bulkhead	(1/4" 60K) A.E.
20	6M166M3	Adaptor	(1" NPTM + 3/8" 60K) A.E.
21	60M46B3	Adaptor	(1/4" 60K + 3/8" 60K) A.E.
22	6M44M3	Adaptors	(1/4" NPTM + 1/4" 60K) A.E.
23	6M84M3	Adaptors	(1/2" NPTM + 1/4" 60K) A.E.
24	6M44B2	Adaptor	(1/4" 60K + 1/4" 10K) A.E.
25	QC6-B-600/QC6-D-600	Quick Connect	(3/8") Swagloc
26	601PC-4	Adaptor	(3/8" 11K + 1/4" 11K) Swagloc
27	601-PC/4R6-316/6TMT4-316	Adaptor	(3/8"-1/4"-TEE-1/8") Swagloc
28	6M42D1	Adaptor	(1/4" 10K + 1/8" 10K) A.E.
29	CT-4440	Tee	(1/4" 60K) A.E.
30	15M42B1	Adaptor	(1/4" 60K + 1/8" 11K) A.E.
31	15BF2211	Bulkhead	(1/8" 11K) A.E.
32	6M26C8	Adaptor	(1/4" NPTF + 1/8" 10K) A.E.
33	ST2220	Tee	(1/8" 11K) A.E.
34	SS-2-TSW-3	Weld Tee	(1/8" 11K) Cajon
35	6M44N2	Adaptor	(1/4" NPTM - 1/4" 11K) A.E.
36	6M412D8	Adaptor	(3/4" NPTF + 1/4" 11K) A.E.



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

AREA IV

ENG.: J. RINDT
DRN. BY: J. BETCHER
DATE: 11-7-79
DRG. NO.: FY79-TO-4-14

Figure 6. Detailed Diagram of Area IV, the Gas Compression System Components.

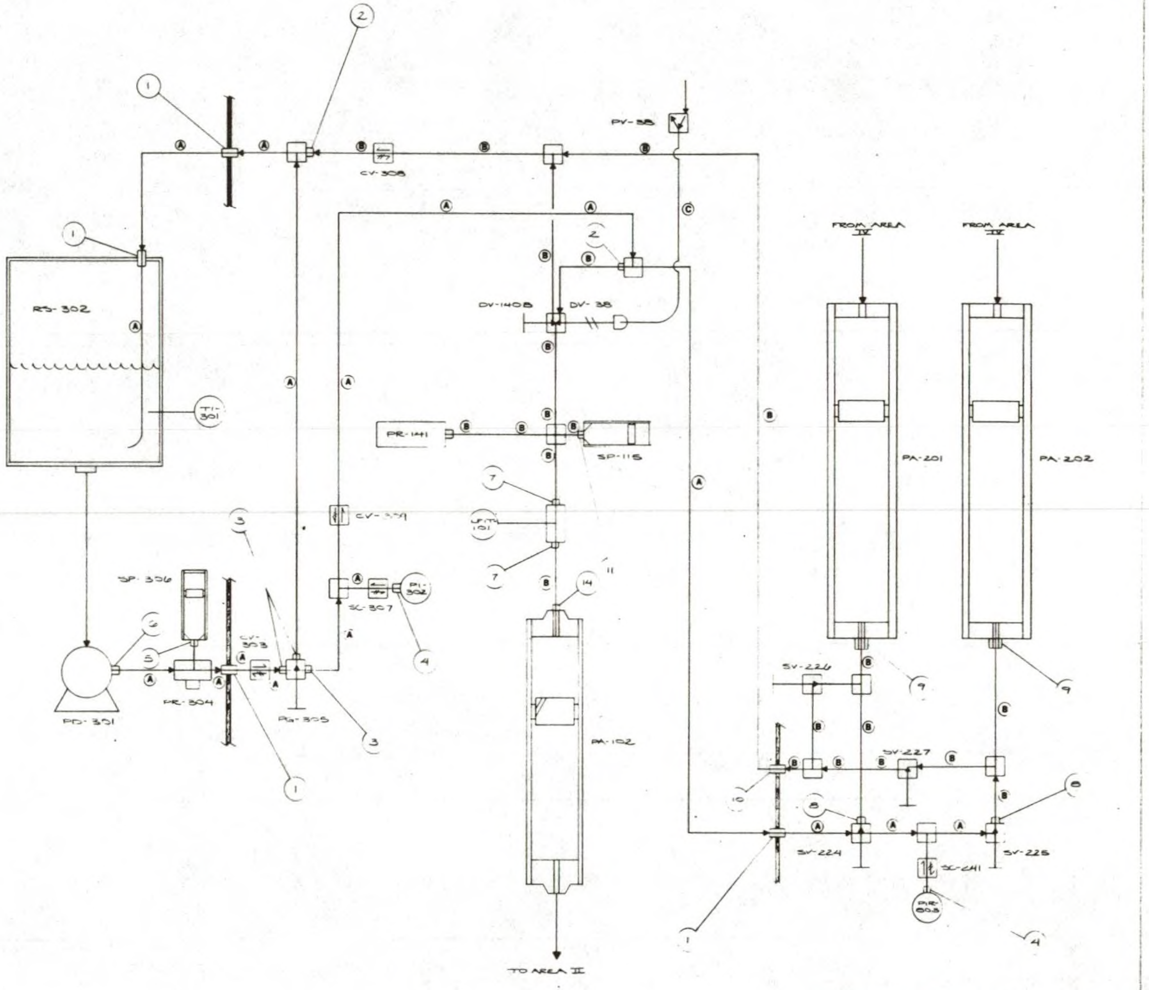
AU - AUTOCLAVE
AC - CARRIER GAS CLEANUP APPARATUS
BA - BLATTER ACCUMULATOR
CP - COLLECTION PORT
CT - COLD TRAP
CV - CHECK VALUE
DV - DOUBLE VALUE
FI - FLOW INDICATOR
FIT - FLOW INDICATOR TOTALIZER
FL - FILTER
FM - FINE METERING VALUE
FV - FLUSH VALUE
GC - GAS CHROMOTOGRAPH
GV - GAS SAMPLING VALUE
MT - MAGNETIC TACKOMETER PICKUP
PA - PISTON ACCUMULATOR
PD - POSITIVE DISPLACEMENT PUMP
PG - BACK PRESSURE REG. VALUE
PI - PRESSURE INDICATOR
PIR - PRESSURE INDICATOR RECORDER
PR - PRESSURE RELIEF VALVE
PV - SOLENOID VALVE
QV - QUENCH VESSEL
RS - RESERVOIR
RSI - REMOTE SAMPLE INLET
RV - PRESSURE REGULATOR VALVE
RVS - REMOTE VACUUM SOURCE
SB - SURGE CHECK BYPASS VALVE
SC - SURGE CHECK VALVE
SL - SAMPLE LOOP
SP - SURGE PISTON ACCUMULATOR
ST - SAMPLE TRAY
SV - SHUTOFF VALVE
TI - TEMPERATURE INDICATOR
TIC - TEMPERATURE INDICATOR CONTROLLER
TIR - TEMPERATURE INDICATOR RECORDER

PIPE SCHEDULE

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

FITTING SCHEDULE

1	20BFX9966	Bulkhead	(9/16" - 20K) A.E.
2	20M96K6	Adaptor	(9/16" 20K + 3/8" 20K) A.E.
3	6M49N6	Adaptor	(1/4" NPTM - 9/16" 20K) A.E.
4	6M49N6	Adaptor	(1/4" NPTM - 9/16" 20K) A.E.
5	6M129N6	Adaptor	(3/4" NPTM - 9/16" 20K) A.E.
6	6MX912K8	Adaptor	(3/4" NPTF - 9/16" 20K) A.E.
7	6M46N6	Adaptor	(1/4" NPTM - 3/8" 20K) A.E.
8	20M96K6	Adaptor	(9/16" 20K - 3/8" 20 , A.E.
9	6M169N6	Adaptor	(1" NPTM - 9/16" 20K) A.E.
10	20BFX666	Bulkhead	(3/8" 20K) A.E.
11	6M126N6	Adaptor	(3/4" NPTM - 3/8" 20K) A.E.
12	20M96B6	Adaptor	(9/16" 60K - 3/8" 20K) A.E.
13	60M94B3	Adaptor	(9/16" 60K - 1/4" 60K) A.E.
14	6M166N6	Adaptor	(1" NPTF - 3/8" 20K) A.E.
18	6M126N6	Adaptor	(3/4" NPTM - 3/4" 20K) A.E.
19	60BF4433	Bulkhead	(1/4" 60K) A.E.
20	6M166N3	Adaptor	(1" NPTM - 3/8" 60K) A.E.
21	60M46B3	Adaptor	(1/4" 60K - 3/8" 60K) A.E.
22	6M44N3	Adaptors	(1/4" NPTM - 1/4" 60K) A.E.
23	6M84N3	Adaptors	(1/2" NPTM - 1/4" 60K) A.E.
24	6M44B2	Adaptor	(1/4" 60K + 1/4" 10K) A.E.
25	QC6-B-600/QC6-0-600	Quick Connect	(3/8") Swagloc
26	601PC-4	Adaptor	(3/8" 11K + 1/4" 11K) Swagloc
27	601-PC/4B6-316/6TMT4-316	Adaptor	(3/8"-1/4"-TLI-1/8") Swagloc
28	6M42D1	Adaptor	(1/4" 10K - 1/8" 10K) A.E.
29	CT-4440	Tee	(1/4" 60K) A.E.
30	15M42B1	Adaptor	(1/4" 60K + 1/8" 11K) A.E.
31	15BF2211	Bulkhead	(1/8" 11K) A.E.
32	6M26C8	Adaptor	(1/4" NPTF - 1/8" 10K) A.E.
33	ST2220	Tee	(1/8" 11K) A.E.
34	SS-2-TSW-3	Weld Tee	(1/8" 11K) Cajon
35	6M44N2	Adaptor	(1/4" NPTM - 1/4" 11K) A.E.
36	6M412D8	Adaptor	(3/4" NPTF + 1/4" 11K) A.E.



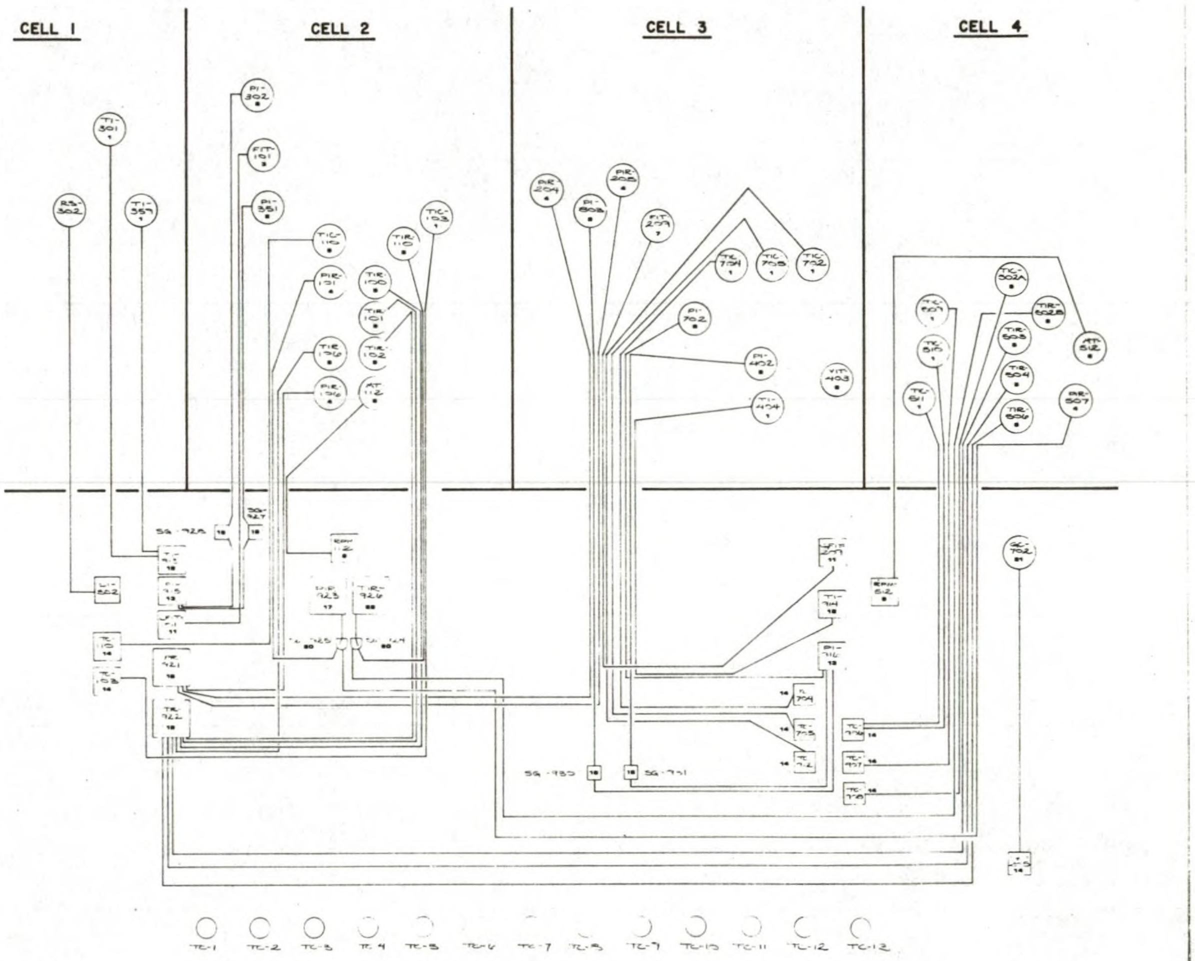
CHEMICAL ENGINEERING
COAL LIQUIFACTION PROJECT
UNIVERSITY OF NORTH DAKOTA - GRAND FORKS

AREA I

ENG.: J. RINDT
DRN. BY: J. BETCHER
DATE: 11-7-79
DRG. NO.: FY79-TO-4-11

Figure 7. Detailed Diagram of Area I, the High-Pressure Hydraulic Oil Supply System Components.

INSTRUMENTATION SCHEDULE	
1	Thermocouple - 2J2M15 Honeywell
2	Pressure Transducer - LFE 2070-7501
3	Flow Indicator (lq) - EFM N/¼/1
4	Pressure Transducer - Century 471-002-38-4[illegible]
5	Thermocouple - In house from Honeywell
6	Magnetic Stirrer Tackometer Pickup - AE AFI-15
7	Flow Indicator (gas) - EFM N/¼/1
8	Dry Gas Meter - Rockwell R-200
9	Tackometer Readout - AE AFP-150
10	Thermocouple Wire - Honeywell
11	Gas Flow Totalizer - EFM T20-TP
12	Temperature Redout - Doric 402-A-1-C
13	Pressure Redout - Doric 412-A
14	Temperature Controllers - Ba
15	Pressure Transducer Module - MII-SIT
16	Chromatography Data Station - P&E [illegible]
17	Continuous Pressure Recorder/Single [illegible] 251-10-000-11-0134- 6-FH000-700-003-055
18	Multipoint Pressure Recorder - Speedomax 251-[illegible]- 055-301
19	Temperature Recorder - Speedomax 251-10-[illegible]
20	Signal Switch
21	Gas Chromatograph - Antek 330



CHEMICAL ENGINERING
COAL LIQUIFACTION PROJECT
UNIVERSITY OF NORTH DAKOTA - GRAND FORKS

INSTRUMENTATION

ENG: J. RINDT DRN. BY: J. BETCHER
DATE: 11-8-79 DRG. NO.: FY79-TO-4-18

Figure 8. Schematic of the Instrumentation for the Hot-Charge System.

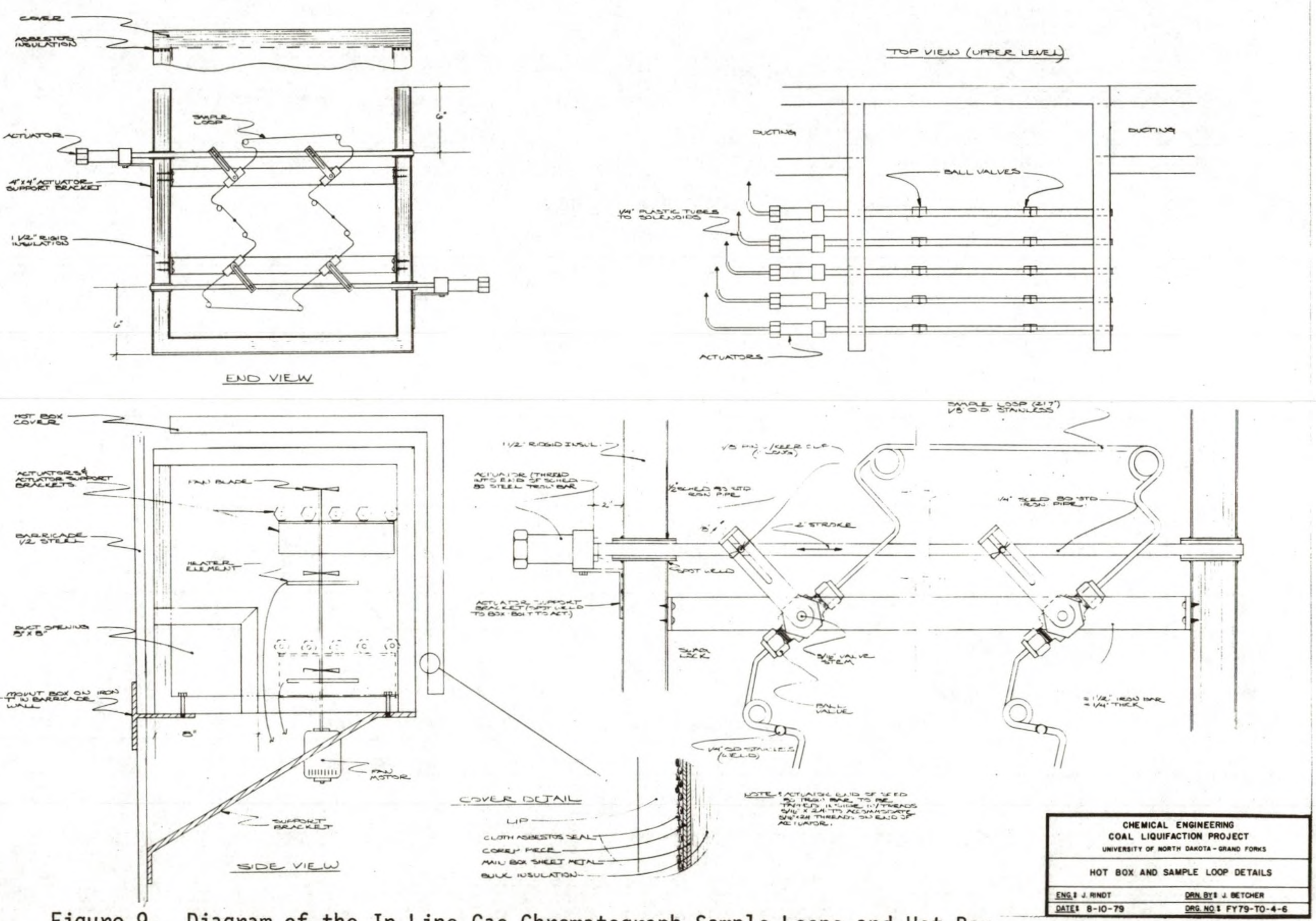


Figure 9. Diagram of the In-Line Gas Chromatograph Sample Loops and Hot Box.

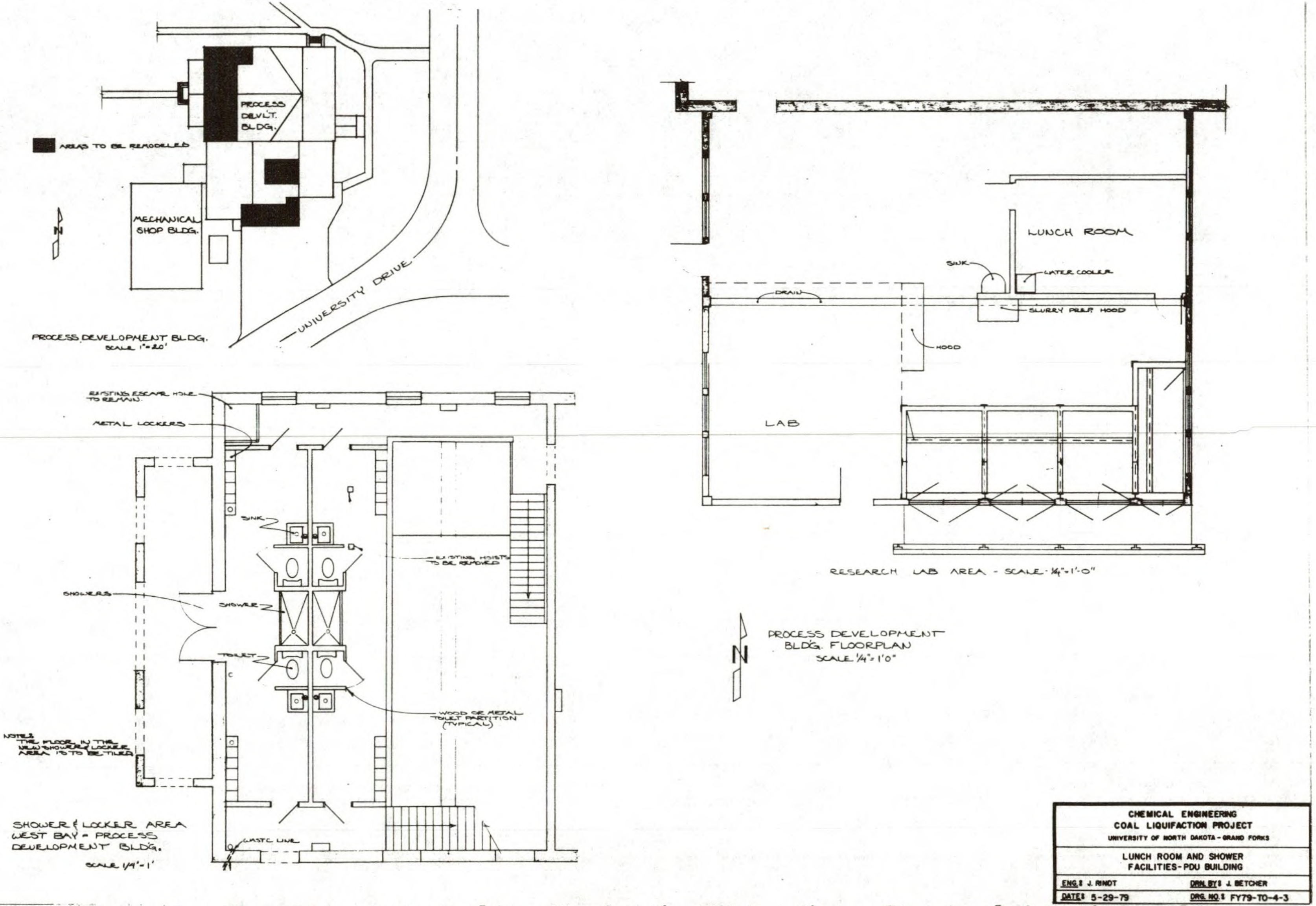
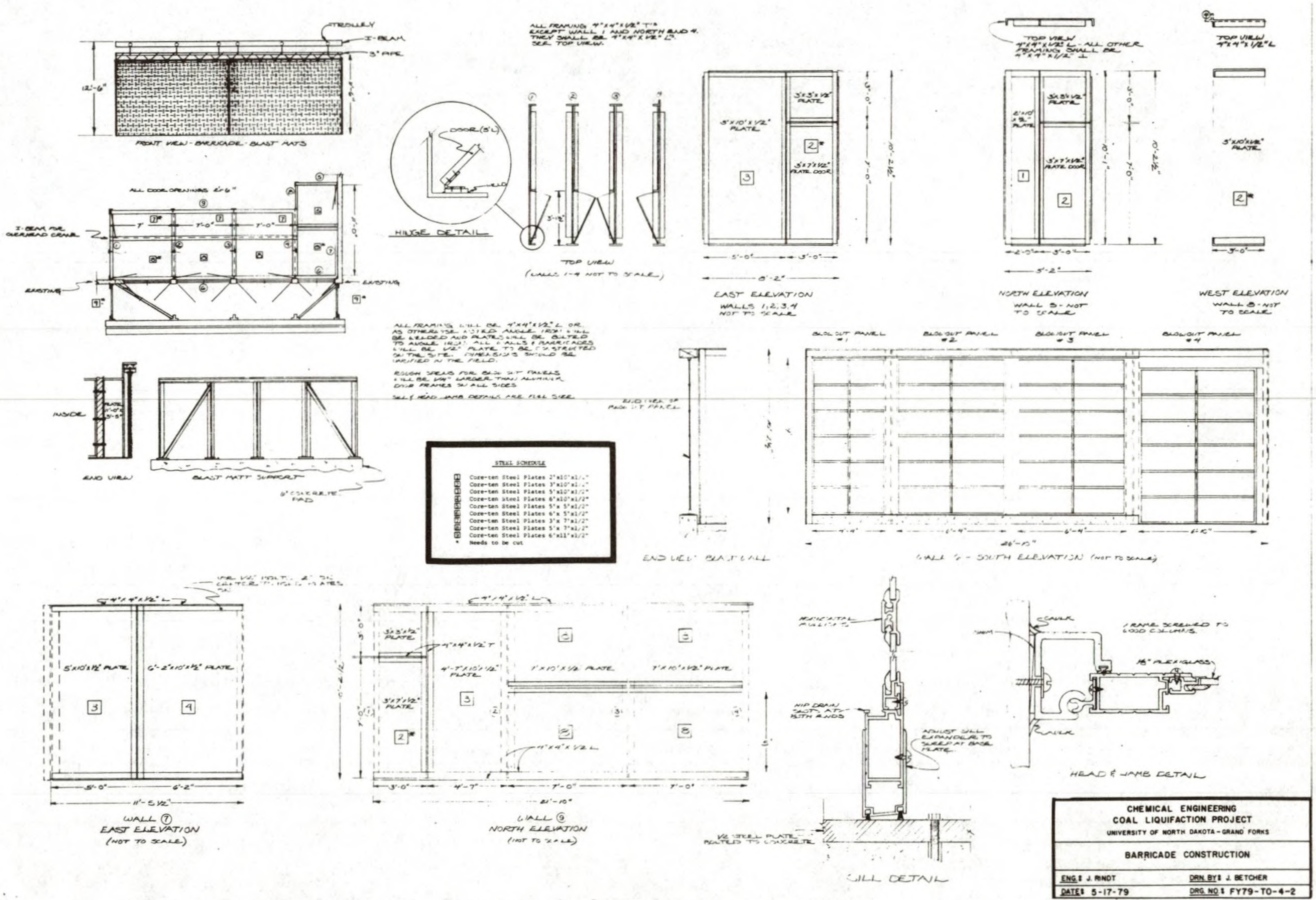


Figure 10. Floor Plan of the Building Housing the UND Hot-Charge Time-Sampled Batch Autoclave facility.



ALL FRAMING: 4"x4"x1/2" T'S EXCEPT WALL 1 AND NORTH END 4. THEY SHALL BE 4"x4"x1/2" L'S. SEE TOP VIEW.

ALL FRAMING WILL BE 4"x4"x1/2" L OR AS OTHERWISE NOTED. ANGLE IRON WILL BE WELDED AND PLATES WILL BE BOLTED TO ANGLE IRON. ALL WALLS & BARRICADES WILL BE 1/2" PLATE TO BE CONSTRUCTED ON THE SITE. DIMENSIONS SHOULD BE VARIFIED IN THE FIELD.

ROUGH OPENINGS FOR BLOWOUT PANELS WILL BE 1/4" LARGER THAN ALUMINUM DOOR FRAMES ON ALL SIDES

SILL & HEAD JAMB DETAILS ARE FULL SIZE

STEEL SCHEDULE

1	Core-ten Steel Plates 2'x10'x1/2"
2	Core-ten Steel Plates 3'x10'x1/2"
3	Core-ten Steel Plates 5'x10'x1/2"
4	Core-ten Steel Plates 6'x10'x1/2"
5	Core-ten Steel Plates 5'x 5'x1/2"
6	Core-ten Steel Plates 6'x 5'x1/2"
7	Core-ten Steel Plates 3'x 7'x1/2"
8	Core-ten Steel Plates 5'x 7'x1/2"
9	Core-ten Steel Plates 6'x11'x1/2"
*	Needs to be cut

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

BARRICADE CONSTRUCTION

ENG: J. RINDT — DRN. BY: J. BETCHER
DATE: 5-17-79 — DRG. NO.: FY79-TO-4-2

Figure 11. Deatiled Diagram of Barricade Structure.

APPENDIX 2

SUMMARY OF PROCEDURES FOR HYDROGEN SULFIDE, AMMONIA, AND SPECIFIC GRAVITY DETERMINATIONS OF THE PRODUCT GAS

DETERMINATION OF HYDROGEN SULFIDE IN SRC PRODUCT GAS (24)

Date: This method covers the determination of hydrogen sulfide in product gas from SRC lab unit. It is applicable on a concentration range of about 0.1 to 7% V/V H_2S. A measured volume of product gas is bubbled through ammoniacal zinc sulfate solution to remove hydrogen sulfide. The amount of hydrogen sulfide in the absorber is then determined iodomectrically.

Special Apparatus:

(a) A 250 ml Erlenmeyer flask with a two-hole rubber stopper carrying (1) a 7-mm diameter glass tube, with a drawn down tip, extending nearly to the bottom of the flask and a (2) short 7-mm diameter glass tube, extending just a little ways on either end of stopper, for exit of excess unabsorbed gas. On the inlet end of the (1) inlet-tube, a 1-ft long rubber injection tube is attached. On the outer end of the (2) exit tube, a small rubber bulb having a small slit cut in it can be used as a 'flap' valve to restrict the rapid flow of gas and to exclude the entrance of air.

(b) Gas Syringes, 500 or 1000 ml - Hamilton Super Syringes No. S-0500 or S-1000.

Reagent Solutions:

(a) Ammoniacal Zinc Sulfate. - Dissolve 50 grams of zinc sulfate heptahydrate in 250 ml of water, and then slowly add 250 ml of concentrated ammonium hydroxide while stirring. Filter off any precipitate that may form upon long standing.

(d) Hydrochloric Acid 1:1. Dilute concentrated HCl with an equal volume of water.

(c) Iodine Solution (0.05N). - Weigh 12.8 gms of resublimed iodine crystals into a 250 ml beaker. Add 40 gms of potassium iodide (KI) and 100 ml of water. Stir until solution is complete, dilute to 2000 ml, mix thoroughly, and store in a brown-glass reagent bottle. (No need to know iodine solution normality exactly if it does not change and the same exact amount volume (25.0 ml) is used in reagent blank and sample determinations.)

(d) Sodium Thiosulfate Standard Solution (0.05N). - Dissolve 25 gms of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in 500 ml water and add 0.01 gm sodium carbonate (Na_2CO_3) to stabilize the solution. Dilute to 2000 ml and mix thoroughly. Standardize verus potassium dichromate or potassium iodate by usual techniques to accuracy of ± 2 ppt.

(e) Starch Solution, 2%. To 250 ml of boiling water, add a cold suspension of 5 gms of soluble starch and 0.025 gms mercuric iodide. Boil for a few minutes to clear. Store in glass-stoppered bottle with undissolved mercuric iodide on bottom.

Sampling

The product gas must be analyzed for H_2S as soon as possible after receipt. Hydrogen sulfide can react with any condensed water vapor or ammonia and can come out of the gas mixture. It may also dissolve in the sides of the bag and be lost. The effect of it possibly combining with carbon monoxide to form carbonyl sulfide (COS) is not known. It would be preferred if the gas could be sampled at the source.

Procedure:

Transfer 30 ml, by graduate, of the ammoniacal zinc sulfate solution to a 250 ml Erlenmeyer flask. Dilute with water to about 150 ml and add a 1 ½ -inch stirring bar. Put in inlet tube-valved stopper. Attach filled gas syringe to injection tube by means of a short piece of glass or stainless steel tubing. While magnetically stirring, slowly inject 100 ml to 1000 ml of product gas depending on H_2S percentage. (Watch for heavy turbidity formation as guide to the volume of gas to use in first test.) (Caution: This part of the test should be done in area free of open flames or sparks. Also if gas contains much carbon monoxide, it should be done in good fume hood!)

Remove syringe from injection tube and record volume of gas injected into flask. Raise rubber stopper just enough to bring end of inlet tube out of solution and wash down the injection tube with about 1 ml of 1:1 HCl and a little water from a wash bottle. Transfer the stirring bar, by means of a thief, from the 250 ml E flask to a 500 ml Erlenmeyer flask containing 25.0 ml of 0.05N iodine solution (by pipet) and 40 ml 1:1 HCl solution (by graduate). While stirring continuously, very slowly pour the contents of the absorbing 250 ml flask into the 500 ml E flask. Rinse the 250 ml flask with about 100 ml water into the 500 ml flask.

As quickly as possible, titrate the solution, while being stirred, with standardized 0.05N sodium thiosulfate solution until the solution is yellow. Then add 2 ml of 2% starch solution and continue titration to a permanent colorless end point. Record volume of sodium thiosulfate required for titration. (Sample solutions are usually turbid at end of titration, blanks are clear.)

Run through above procedure, leaving out gas sample, for reagent blank. The nature of this test is that the blanks are equal to or usually higher than the titration volumes obtained for samples.

The blank and sample tests should be run in duplicate. The blank values do not change very much, therefore only a weekly check is necessary if there have not been any changes in reagents, room temperature, etc. If sample titration volume is less than half of the reagent blank, test should be rerun using less gas sample.

Calculations

$$\frac{(V_b - V_s) \times N_t \times F \times 100}{V\ gas} = \%\ H_2S\ (\text{mole}\ \%)$$

where:

V_b = Volume (mls) of sodium thiosulfate used in blank

V_s = Volume (mls) of sodium thiosulfate used in sample.

N_t = Normality of sodium thiosulfate

Vgas = Volume (mls) of product gas used in test.

F = Factor in milliliters of hydrogen sulfide per milliequivalent of sodium thiosulfate. It is one half of the reciprocal of the molar equivalent of one liter of gas (moles/liter) at temperature and pressure of product gas at time of testing. See "Molar equivalent of one liter of gas at various temperatures and pressures" Table for ease of calculations. An average value of 12.5 can be used in the Kansas City Area.

Revised: July 1971 - REP

Reference Source: ASTM D2385

AMMONIA IN GAS DETERMINATION (25)

1. Turn on Spectronic "20," set at 425 nm, and allow to warm up for thirty minutes.

2. Prepare six ammonia-in-water standards of 1, 2, 3, 5, 8, and 10 ppm ammonia concentration in 500 ml volumetric flasks. (Use 200, 500, and 1000 ppm ammonia stock solution standards for preparation of the 1 to 10 ppm ammonia samples. Note: Before diluting to the mark, add one drop of conc. HCl to each 500 ml volumetric flask.

3. Transfer a portion of each of the six ammonia standards prepared in step 2 to 50 ml volumetric flasks, filling to the mark, and then adding 1 ml of Nessler's Reagent. **Start timer, agitate volumetric flask for several minutes and then allow color development to take place for twenty minutes. Prepare blank in identical manner using acidified demineralized water.

4. Read optical density (o.d.) of ammonia standards at 425 nm on Spectronic "20," using the blank for the 100% transmission adjustment. Note: Be sure to use the same curvette for all readings, rinsing it thoroughly with the next sample to be analyzed before proceeding.

5. Obtain computer tape marked "Least Square Slope Calc.-General," No. 1, and determine the slope value for the x-y plot of concentration vs. optical density.

6. Prepare gas samples for analysis by bubbling 500 cc of the product gas through 150 ml of filtered distilled water. Next add one drop of concentrated HCl and 1 ml of Nessler's Reagent. Stopper flask, set timer, agitate flask for several minutes and allow color development for twenty minutes. Prepare blank in identical manner.

7. After twenty minutes, adjust the 100% transmission setting with the blank, and then read the optical density of the samples. If an optical density reading is higher than 0.7, repeat the analysis on the sample using a higher dilution.(1) Note: Be sure to use the same curvette for all readings, rinsing it thoroughly with the next sample to be analyzed before proceeding.

8. Compute ppm NH_3 in water sample by multiplying the optical density by the slope value obtained in step 5. X dilution factor. From ppm NH_3 in water sample, calculate mol ppm NH_3 in 500 cc gas sample (See worksheet).

9. Record all data in appropriate lab databook and on NH_3 in gas worksheet.

** Adding Nessler's reagent to the flask after diluting to the mark is done to minimize air contact with the sample.

(1) Conversely, if optical density reading is less than 0.1, repeat analysis using a lower dilution.

DETERMINATION OF SPECIFIC GRAVITY (26)

1. Evacuate the glass flask by attaching it to the vacuum pump with the attached rubber hose. Open the flask and turn on the vacuum pump to evacuate the bulb. Evacuate until a constant reading is obtained on the manometer. Close the flask and shut off the vacuum pump. Open the flask to the air and then weigh. Record the weight obtained as the weight of AIR.

2. Again evacuate the glass flask until a steady reading is obtained on the manometer. Close the flask and shut off vacuum pump. Weigh and record the weight of the evacuated flask. Record this as the VACUUM weight.

3. Attach the previously weighed evacuated flask back on to the vacuum apparatus without opening the flask. Connect the gas sample bag to the gas hose provided on the apparatus, making sure all valves are closed. With the flask and bag closed, open the valve mounted on the wall leading to the bag and allow the vacuum to stabilize. Close the wall mounted valve tightly. Open the gas valve on the bag and allow the gas to enter the rubber hose. Maintain a constant vigil on the manometer to insure a constant reading. Slowly close the vacuum valve mounted on the vacuum pump; maintaining a constant manometer reading. Close until the manometer starts to drop, reopen valve to a steady position. At this time, quickly open the wall mounted valve while, simultaneously, closing the valve on the vacuum pump. Now open the ground glass fitting to allow the gas to enter the glass bulb. After thirty seconds close the valves on the bulb and on the gas bag and then disconnect the bulb and weigh and record the weight as that of the gas sample.

4. Repeat the procedure for each sample until reproducible results are achieved. This procedure is to be followed for each sample gas bag for each run. The results are to be recorded for each bag.

5. CALCULATIONS

   a. Subtract the weight of the VACUUM from that of the AIR sample weight.
   b. Subtract the weight of the VACUUM from the weight of the GAS.
   c. Divide the results from a into the results from b.
   d. Repeat for each sample and average the results for each bag.

6. NOTES

   Lintless gloves should be worn at all times to avoid weight gain of the bulb.
   The specific gravity for two different bags of a sample will not necessarily be the same, but, the specific gravity of two samples of the same bag should be within ± .0100 of the average specific gravity.

APPENDIX 3

SAMPLE YIELD CALCULATIONS FOR RUN M1

SAMPLE CALCULATIONS FOR RUN M1

The mass of the gas into the autoclave was determined by calibrating the accumulator into which the reaction gas was compressed. The accumulator was calibrated to use any mixture of CO and H_2 from pure CO to pure H_2. The accumulator was also calibrated to allow H_2S to be added to the mixture.

First the pressure of the reaction gas was converted from gauge to absolute pressure for run 1.

$P(1) = H_2\text{-CO pressure} = \text{gauge pressure} + \text{atmospheric pressure}$

$$P(1) = P(1) + (P(3) * 0.49131) \qquad (160)^2$$

$$P(1) = 380 \text{ psig} + (29.57 \text{ in Hg})(.49131 \frac{\text{psig}}{\text{in Hg}})$$

$$P(1) = 394.528 \text{ psig}$$

The moles of gas in was then determined from the equation

$$C(1) = ((273 + T(1)/293)(Y1/100 * (P(1) * M1 + R1) + ((1 - Y1/100)\ P(1) * M2 + R2))) \qquad (170)$$

where $T(1) = H_2\text{-CO}$ temperature in accumulator, °C

$Y1 = \%\ H_2$ in feed gas

$P(1) = H_2\text{-CO}$ pressure in accumulator, psig

M1 and M2 = Slopes for the H_2 and CO calibration lines, respectively

R1 and R2 = Y-intercepts of the H_2 and CO calibration lines, respectively

for Run 1 T(1) = 19°C, Y1 = 0

$$C1 = (273 + 19/293)((0/100) * (394.5280 \text{ psig} * 2.3776 \times 10^{-2} +$$

---

[2] Numbers in parentheses in this Appendix refer to the line number in the computer program which performs this calculation.

$-1.37239 \times 10^{-1}) + ((1-0/100 * (394.5280 \text{ psig} *$

$2.4935 \times 10^{-2} + -3.0819 \times 10^{-2})))$

$= 9.773$

P(2) = H_2S pressure = gauge pressure + atmospheric pressure

$P(2) = \quad P(2) + P(3) * 0.49131)$ (160)

$= 0 \text{ psig} + (29.57 \text{ in Hg})(0.49131 \frac{\text{psig}}{\text{in Hg}})$

$= 14.528 \text{ psig}$

and from the equation

$C(2) = ((273 + T(2)/293) * Y1/100 * (P(2) * M1 + R1)))$ (170)

$+ ((1-Y/100) * (P(2) * M2 + R2)))$

where T(2) = H_2S temperature in accumulator °C

assume 0 if no H_2S present

Y1 = % H_2 in feed gas

P(2) = H_2S pressure in accumulator, psig

M1 and M2 = slopes for the H_2 and CO calibration lines, respectively.

R1 and R2 = Y intercepts of the H_2 and CO calibration lines, respectively.

for Run 1

since no H_2S is present T(2) = 0

$C(2) = (273 + 0°C/293) * (0/100 * (14.5280 \text{ psig} * 2.3776 \times 10^{-2}$

$+ -1.37239 \times 10^{-1}) + ((1-0/100)* (14.5280 \text{ psig}$

$2.4935 \times 10^{-2} + -3.0819 \times 10^{-2})))$

$C(2) = \ .309$

$C(3) = \ C(1) - C(2) = 9.773 - .309 = 9.464$ (190)

Then $N = C(3) + C(4)$ (220)

where C(4) = moles of H_2S in the gas and is found by the equation

$$C(4) = ((273 + T(1)/294) * (P(2) * M3 + R5)) \qquad (210)$$

where T1 = H_2-CO temperature in accumulator °C

P(2) = H_2S pressures in accumulator psig

M(3) = slope of H_2S calibration line

R(5) = Y intercept of H_2S calibration line

In this case when T(2) = 0

C(4) = 0

Therefore N1 = C(3) = 9.464 moles of gas into the autoclave

since all runs were made using pure CO

the mass of the gas in was

$$\frac{9.464 \text{ g moles CO} \quad 28.01}{\text{g mole CO}} = 265.099 \quad \text{CO in}$$

Grams slurry into the autoclave = 728.1 grams

The slurry consists of H_2O, MAF lignite, and solvent in a 100 : 200 : 400 ratio and the ash present in the lignite. Therefore the actual amounts of H_2O and MAF lignite, HAO-61 and ash into the autoclave were determined as follows:

HAO-61 + MAF lignite H_2O + Ash = Mass slurry into autoclave

$$= 4x + 2x + 1x + \left(\frac{\text{MAF lignite}}{1\text{-}\%\ H_2O \text{ lignite} - \%\text{ Ash lignite}}\right) * (\%\text{ ash lignite})$$

$$= \quad 7x + \left(\frac{2x}{1\text{-}\%\ H_2O \text{ lignite} - \%\text{ Ash lignite}}\right) * (\%\text{ Ash lignite}) \qquad (290)$$

for Run 1

$$7x + \left(\frac{2x}{(1\text{-}29.88/100 - 7.98/100)}\right) * (7.98/100) = 728.1 \text{ gm}$$

$$7x + .25684\ x = 728.1 \text{ gm}$$

$$7.25684\ x = 728.1\ \text{gm}$$

$$x = 100.333\ \text{gm}$$

Therefore the masses of following are

H_2O = 100.333 gm

MAF lignite = (2) 100.333 gm = 200.666

HAO-61 = (4) 100.333 gm = 401.332

and the mass of the ash is

$$\text{Ash} = \left(\frac{\text{MAF lignite}}{(1-\%\ H_2O\ \text{lignite} - \%\ \text{Ash lignite})}\right) * (\%\ \text{Ash lignite})$$

$$= \left(\frac{200.6659}{1-29.88/100 - 7.98/100}\right) * (7.98/100) \qquad (337)$$

$$= 25.769$$

$$\text{Total mass in} = H_2O + \text{MAF lignite} + \text{HAO-61} + \text{Ash} + \text{Gas} \qquad (340)$$

$$= 100.3329 + 200.6659 + 401.3317 + 25.7695 + 265.0995$$

$$= 993.199\ \text{gm}$$

The moles of product gas were determined from the volume measurement on the dry gas meter assuming that the ideal gas law is obeyed. From the ideal gas law

$$PV = nRT \text{ or } n = \frac{PV}{RT} \qquad (270)$$

for Run 1

$$n = \frac{(29.57\ \text{in Hg})(10.169\ \text{ft}^3)}{(.048106 \frac{\text{in Hg ft}^3}{\text{g mole oR}})(520°\text{R})}$$

$$n = 12.0206\ \text{g moles gas out}$$

knowing the product gas composition the mass of the gas out can be determined as follows

Component	Total moles	mole %	MW	
CO_2	(12.0206 moles)	(35.54/100)	(44.01) =	188.0164
C_2H_6	"	(0.47/100)	(30.07) =	1.6989
C_3H_8	"	(0.14/100)	(44.09) =	0.7420
H_2	"	(14.64/100)	(2.016) =	3.5478
CH_4	"	(1.15/100)	(16.04) =	2.2173
CO	"	(47.99/100)	(28.01) =	161.5812
H_2S	"	(0.05/100)	(34.08) =	0.2048
NH_3	"	(0.01/100)	(17.08) =	0.0205
Total				358.028

Mass of the gas out = 358.028 gm (280)

Mass of the Ash out

Ash = (% of Ash of endpot)(Mass endpot) (335)

= (4.52%/100)(544.7g) = 24.620 gm Ash Out

IOM = (1 - % THF soluble) (Mass endpot) - Ash out (330)

= (1-92.84/100)(544.7g) - 24.6204 g

= 14.380 g IOM out

SRL = (% THF soluble - % cyclohexane soluble)(Mass endpot) (320)

= ((92.84 - 79.62)/100) * (544.7g)

= 72.009 g SRL out

H_2O out = (% H_2O in endpot)(Mass endpot)+(% H_2O in condensate)
(Mass Condensate) (300)

H_2O out = (0.147%)(544.7 gm) + (98.24%/100)(42.2 gm)

= 42.258 gm H_2O out

Oil out = (% cyclohexane soluble)(Mass endpot) - (% H_2O in endpot)
(Mass endpot) + (1 - % H_2O in condensate)
(Mass condensate) (310)

Oil out = (79.62 %/100)(544.7 gm) - (0.147 %/100 (544.7 gm)

+ (1-98.24/100)(42.2 gm) = 433.632 gm oil out

Total mass out = Gas + Ash + IOM + SRL + H_2O + Oil (350)

= 358.028 + 24.620 + 14.380 + 72.009 + 42.258 + 433.632

= 944.928 gm out

Net yields/wt % MAF lignite

$$H_2O = \left(\frac{H_2O\text{ out} - H_2O\text{ in}}{\text{MAF lignite in}}\right) 100 \qquad (410)$$

$$= \left(\frac{42.258 - 100.333}{200.666}\right) 100 = -28.941\%$$

$$\text{Oil} = \left(\frac{\text{oil out - solvent HAO-61 in}}{\text{MAF lignite in}}\right) 100 \qquad (370)$$

$$= \left(\frac{433.632 - 401.332}{200.666}\right) 100 = 16.096\%$$

$$\text{SRL} = \left(\frac{\text{SRL out}}{\text{MAF lignite in}}\right) 100 \qquad (380)$$

$$= \left(\frac{72.009}{200.666}\right) (100) = 35.885\%$$

$$\text{IOM} = \left(\frac{\text{IOM out}}{\text{MAF lignite in}}\right) 100 \ (400) = \left(\frac{14.280}{200.666}\right) 100 = 7.166\%$$

$$\text{Ash} = \left(\frac{\text{Ash out - Ash in}}{\text{MAF lignite in}}\right) 100 \qquad (415)$$

$$= \left(\frac{24.620 - 25.769}{200.666}\right) 100 = -0.5726\%$$

$$\text{Gas} = \left(\frac{\text{Gas out - Gas in}}{\text{MAF lignite in}}\right) 100 \qquad (390)$$

$$= \left(\frac{358.029 - 265.099}{200.666}\right) 100 = 46.311$$

$$\text{Total} = H_2O + \text{Oil} + \text{SRL} + \text{IOM} + \text{Ash} + \text{Gas} \qquad (455)$$

$$= -28.941 + 16.096 + 35.885 + 7.166 + -0.573 + 46.311$$

$$= 75.945$$

$$\%\ \text{closure} = \left(\frac{\text{mass out}}{\text{mass in}}\right)\ 100 \qquad (360)$$

$$= \left(\frac{944.928}{993.199}\right)\ 100 \quad = 95.140$$

$$\%\ \text{conversion} = 100 - \text{IOM} \qquad (420)$$

$$= 100 - 7.166 \quad = 92.834$$

Normalized Output

Assuming difference in output is divided proportionally between the gas out, endpot, and cold trap condensate

$$\text{Mass gas out} = \left(\frac{\text{unnormalized mass gas out}}{\%\ \text{closure}}\right)\ 100 \qquad (960)$$

$$= \left(\frac{358.0290}{90.140}\right)\ 100$$

$$= 376.318 \quad \text{gm gas out}$$

$$\text{Mass endpot out} = \left(\frac{\text{unnormalized mass endpot}}{\%\ \text{closure}}\right)\ 100 \qquad (940)$$

$$= \left(\frac{544.7}{95.140}\right)\ 100 = 572.525\ \text{gm endpot out}$$

$$\text{Mass condensate out} = \left(\frac{\text{unnormalized mass condensate}}{\%\ \text{closure}}\right)\ 100 \qquad (950)$$

$$= \left(\frac{42.2\ \text{gm}}{95.140}\right)\ 100 = 44.356\ \text{gm condensate out}$$

Normalized mass out = mass gas out + mass endpot out

+ mass condensate out

= 376.318 + 572.525 + 44.356

= 993.199 gm out

Ash out = (% Ash in endpot)(Mass endpot) (335)

(4.52%/100) (572.525 gm)

= 25.878 gm Ash out

IOM = (1- % THF soluble/100)(Mass endpot) - Ash out (330)

(1- 92.84/100)(572.525 gm) -25.878

= 40.993 - 25.878

= 15.115 gm IOM out

SRL = (% THF soluble - % cyclohexane soluble)(Mass endpot)(320)

= (92.84 - 79.62)/100 (572.525)

= 75.688 gm SRL out

H_2O out = (%H_2O in endpot)(Mass endpot)+(%H_2O in condensate)

(Mass condensate) (300)

= (0.147%/100)(572.525) + (98.24%/100)(44.356)

= 44.417 gm H_2O out

Oil out = (% cyclohexane soluble)(Mass endpot) + (1-% H_2O in

condensate)(Mass condensate) - (% H_2O in endpot) (310)

(Mass endpot)

= (79.62%/100)(572.525 gm) + (1-98.24%/100)(44.356 gm)

- (0.147%/100)(572.525 gm) = 455.783 gm

Gas out = 376.318 gm gas out

Total mass out = Ash + IOM + SRL + H_2O + Oil + Gas (350)

= 25.878 + 15.115 + 75.688 + 44.417 + 455.783 + 376.318

= 993.199 gm

Normalized Net Yields/Wt % MAF Lignite

$$H_2O = \left(\frac{H_2O \text{ out} - H_2O \text{ in}}{\text{Mass MAF lignite in}}\right) 100 \qquad (410)$$

$$= \left(\frac{44.417 - 100.333}{200.666}\right) 100 = -27.865$$

$$\text{Oil} = \left(\frac{\text{Oil out} - \text{HAO-61 Solvent in}}{\text{MAF lignite in}}\right) 100 \qquad (370)$$

$$= \left(\frac{455.783 - 401.332}{200.666}\right) 100 = 27.135$$

$$\text{SRL} = \left(\frac{\text{SRL out}}{\text{MAF lignite in}}\right) 100 \qquad (380)$$

$$= \left(\frac{75.688}{200.666}\right) 100 = 37.718$$

$$\text{IOM} = \frac{\text{IOM out}}{\text{MAF lignite in}} = \left(\frac{15.115}{200.666}\right) 100 \qquad (400)$$

$$= 7.532$$

$$\text{Ash} = \left(\frac{\text{Ash out} - \text{Ash in}}{\text{MAF lignite in}}\right) 100 = \qquad (415)$$

$$\left(\frac{25.878 - 25.769}{200.666}\right) 100 = .054$$

$$\text{Gas} = \left(\frac{\text{Gas out} - \text{Gas in}}{\text{MAF lignite in}}\right) 100 \qquad (390)$$

$$= \left(\frac{376.318 - 265.099}{200.666}\right) 100$$

$$= 55.425$$

$$\text{Total} = H_2O + \text{Oil} + \text{SRL} + \text{IOM} + \text{Ash} + \text{Gas} \qquad (455)$$

$$= -27.865 + 27.135 + 37.718 + 7.532 + .054 + 55.425$$

$$= 100.000$$

$$\% \text{ closure} = \left(\frac{\text{Mass out}}{\text{Mass in}}\right) 100 \qquad (360)$$

$$= \left(\frac{993.199}{993.199}\right) 100 = 100\% \text{ closure}$$

$$\% \text{ conversion} = 100\%\text{-IOM} = 100-7.532 \qquad (420)$$

$$= 92.468$$

APPENDIX 4

SYMBOLS AND COMPUTER PROGRAM USED TO PERFORM NET YIELD CALCULATIONS

Symbols Used in Computer Programs

D0	Date of Run
P0	Run #
P(3)	Barometric pressure, in Hg
P(1)	H2-CO pressure in accumulator, psi
P(2)	H_2S pressure in accumulator, psi
Y1	% H2 in feed gas
T(1)	H2-CO temperature in accumulator, °C
T(2)	H2S temperature in accumulator, °C
M(4)	Mass slurry into autoclave sm
M5	Mass condensate in cold traps sm
V1	Volume product gas out through dry gas meter, ft^3
M6	Mass endpot out, gm
@1	% Ash in endpot
@2	% H_2O in endpot
S1	% cyclohexane soluble of endpot
S2	% THF soluble of endpot
@3	% H_2O in condensate
G1	Mole % CO_2 in product gas
G2	mole % C2H6 in product gas
G3	mole % C3H8 in product gas
G4	mole % H2 in product gas
G5	mole % CH4 in product gas
G6	mole % CO in product gas
G7	mole % H2S in product gas
G8	mole % NH3 in product gas
N1	moles of reactant gas, gmoles
M7	mass of reactant gas, gm
H2	mole % of H2S in reactant gas
H3	mole % of H2 in reactant gas
C1	mole % of CO in reactant gas
N2	moles of product gas, gmoles
MS	Mass of product gas, gm
X1	Mass H_2O in, gm

X2	Mass H_2O out, gm
@4	% ash in lignite
D1	Mass oil out, gm
S3	Mass SRL out, gm
I1	Mass IOM out, gm
I2	Mass in, gm
0	Mass out, gm
C6	% closure
C(AG)	Calibration coefficient
D	New % oil/MAF lignite
S	Net % SRL/MAF lignite
G	Net % gas/MAF lignite
I	Net % IOM/MAF lignite
H	Net % H_2O/MAF lignite
H6	Mass HAO-61 in, gm
L1	Mass MAF lignite in, gm
A1	Mass Ash in, gm
@5	% H_2O in lignite
A2	Mass Ash in endpot, gm
A	Net % Ash/MAF lignite
C7	% conversion

```
  5  PRINT "RUN #, DATE OF RUN, ATM PRESS, H2-CO PRESS, H2S PRESS, %H2
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, %ASH IN ENDPOT, %H2O IN LIG
NITE"
 30 INPUT V1,M6,@1,@5
 35 PRINT "%H2O IN ENDPOT, %CYCLOHEXANE SOLUBLE, %THF SOLUBLE"
 40 INPUT @2,S1,S2
 45 PRINT "%H2O IN CONDENSATE, %ASH 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
```

```
 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 "H20",
 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                  WT% MAF LIG"
 720 PRINT ".............         ........             .............."
 730 :####.###                  ###.###
 740 PRINT "H20",
 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
```

```
 840 PRINT "   TOTAL",
 850 PRINT USING 730,0,T
 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 5

DATA SHEETS AND COMPUTER PRINTOUTS FOR EACH RUN

Data Sheet

Run 1

Run #, Date of Run, Atm Press, H2-CO Press, H2S Press, % H2 in feed gas

M1 , 22382 , 29.57 , 380 , 0 , 0

H2-CO Temp, H2S Temp, Mass Slurry In, Mass condensate out

19 , 0 , 728.1 , 42.2

Vol Gas out, Mass Endpot, % Ash in Endpot, % H_2O in lignite

10.169 , 544.7 , 4.52 , 29.88

% H_2O in Endpot, % cychex soluble, % THF soluble

0.147 , 79.62 , 92.84

% H_2O in condensate, % Ash in lignite

98.24 , 7.98

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

35.54, 0.47, 0.14, 14.64, 1.15, 47.99, 0.05, 0.01

Run 2

Run #, Date of Run, Atm Press, H2-CO Press, H2S Press, % H2 in feed gas

M2 , 30382 , 29.36 , 380 , 0 , 0

H2-CO Temp, H2S Temp, Mass Slurry In, Mass condensate out

20 , 0 , 733.5 , 93.0

Vol Gas out, Mass Endpot, % Ash in Endpot, % H_2O in lignite

10.195 , 568.7 , 4.58 , 30.74

% H_2O in Endpot, % cychex soluble, % THF soluble

0.2065 , 76.79 , 92.36

% H_2O in condensate, % Ash in lignite

88.61 , 7.88

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

43.82, 0.27, 0.08, 9.97, 0.64, 45.14, 0.09, 0.01

Run 3

Run #, Date of Run, Atm Press, H2-CO Press, H2S Press, % H2 in feed gas

M3 , 31082 , 28.78 in Hg, 380 psi , 0 , 0

H2-CO Temp, H2S Temp, Mass Slurry In, Mass condensate out

22°C , 0 , 730.0 gm , 6.2 gm

Vol Gas out, Mass Endpot, % Ash in Endpot, % H_2O in lignite

9.874 ft , 663.1 gm , 3.76 , 30.29

% H_2O in Endpot, % cychex soluble, % THF soluble

10.42% , 76.37 , 90.31

% H_2O in condensate, % Ash in lignite

100 , 7.93

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

23.53, 0.130, 0.035, 8.935, 0.575, 66.78, 0.01, 0.005

Run 4

Run #, Date of Run, Atm Press, H2-CO Press, H2S Press, % H2 in feed gas

M4 , 31682 , 28.93, 380 , 0 , 0

H2-CO Temp, H2S Temp, Mass Slurry In, Mass condensate out

22 , 0 , 721.0 , 5.2

Vol Gas out, Mass Endpot, % Ash in Endpot, % H_2O in lignite

9.396 , 662.0 , 3.84 , 29.92

% H_2O in Endpot, % cychex soluble, % THF soluble

11.71 , 77.48 , 91.20

% H_2O in condensate, % Ash in lignite

50.08 , 7.97

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

23.335, 0.08, 0.04, 6.37, 0.525, 69.455, 0.185, 0.015

Run 5

Run #, Date of Run, Atm Press, H2-CO Press, H2S Press, % H2 in feed gas

M5 , 32382 , 238.63, 380 , 0 , 0

H2-CO Temp, H2S Temp, Mass Slurry In, Mass condensate out

22 , 0 , 723.2 , 3.2

Vol Gas out, Mass Endpot, % Ash in Endpot, % H_2O in lignite

10.313 , 631.5 , 4.06 , 29.91

% H_2O in Endpot, % cychex soluble, % THF soluble

9.05 , 80.29 , 93.06

% H_2O in condensate, % Ash in lignite

50.08 , 7.97

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

26.91, 0.36, 0.095, 8.235, 1.11, 63.15, 0.12, 0.01

Run 6

Run #, Date of Run, Atm Press, H2-CO Press, H2S Press, % H2 in feed gas

M6 , 33182 , 28.91 , 380 , 0 , 0

H2-CO Temp, H2S Temp, Mass Slurry In, Mass condensate out

21 , 0 , 725.0 , 2.2

Vol Gas out, Mass Endpot, % Ash in Endpot, % H_2O in lignite

10.060 , 628.3 , 4.03 , 29.45

% H_2O in Endpot, % cychex soluble, % THF soluble

8.339 , 80.76 , 93.21

% H_2O in condensate, % Ash in lignite

77.94 , 8.03

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

30.52, 0.37, 0.155, 15.320, 1.345, 52.125, 0.16, 0.01

Run 7

Run #, Date of Run, Atm Press, H2-CO Press, H2S Press, % H2 in feed gas

M7 , 60482 , 29.08, 380 , 0 , 0

H2-CO Temp, H2S Temp, Mass Slurry In, Mass condensate out

22 , 0 , 727.28 , 75.25

Vol Gas out, Mass Endpot, % Ash in Endpot, % H_2O in lignite

10.6975 , 543.09 , 4.84 , 28.71

% H_2O in Endpot, % cychex soluble, % THF soluble

0.128 , 76.24 , 92.00

% H_2O in condensate, % Ash in lignite

56.62 , 8.11

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

31.465, 0.295, 0.140, 13.130, 0.960, 53.890, 0.100, 0.040

```
RUN NUMBER        M1                DATE                  22382

INPUT
........

COMPONENT                            GRAMS IN
..........                           .........
MAF LIGNITE                           200.666
H20                                   100.333
ASH                                    25.769
HAO-61                                401.332
GAS                                   265.099
   TOTAL                              993.198
............................................................
OUTPUT
...........
                   GRAMS                         NET YIELDS
COMPONENT          OUT                          WT% MAF LIG
.............      ........                    .............
H20                  42.258                         -28.941
OIL                 433.632                          16.096
SRL                  72.009                          35.885
IOM                  14.380                           7.166
ASH                  24.620                          -0.573
GAS                 358.028                          46.311
   TOTAL            944.927                          75.945

% CLOSURE                    95.140
% CONVERSION                 92.834
............................................................
NORMALIZED
OUTPUT
...........
                   GRAMS                         NET YIELDS
COMPONENT          OUT                          WT% MAF LIG
.............      ........                    .............
H20                  44.417                         -27.865
OIL                 455.783                          27.135
SRL                  75.688                          37.718
IOM                  15.115                           7.532
ASH                  25.878                           0.054
GAS                 376.318                          55.425
   TOTAL            993.198                         100.000

% CLOSURE                   100.000
% CONVERSION                 92.468
............................................................
TIME 0.2 SECS
```

```
RUN NUMBER          M2                  DATE                 30382

INPUT
........

COMPONENT                                GRAMS IN
..........                               .........
MAF LIGNITE                               202.156
H20                                       101.078
ASH                                        25.953
HAO-61                                    404.312
GAS                                       266.032
   TOTAL                                  999.531
............................................................
OUTPUT
...........
                   GRAMS                       NET YIELDS
COMPONENT          OUT                        WT% MAF LIG
............       ........                   .............
H20                  83.582                        -8.655
OIL                 446.123                        20.682
SRL                  88.547                        43.801
IOM                  17.402                         8.608
ASH                  26.046                         0.046
GAS                 387.465                        60.069
   TOTAL           1049.164                       124.552

% CLOSURE                   104.966
% CONVERSION                 91.392
............................................................
NORMALIZED
OUTPUT
...........
                   GRAMS                       NET YIELDS
COMPONENT          OUT                        WT% MAF LIG
............       ........                   .............
H20                  79.628                       -10.611
OIL                 425.018                        10.242
SRL                  84.358                        41.729
IOM                  16.579                         8.201
ASH                  24.814                        -0.563
GAS                 369.135                        51.002
   TOTAL            999.531                       100.000

% CLOSURE                   100.000
% CONVERSION                 91.799
............................................................
TIME 0.2 SECS
```

```
RUN NUMBER        M3                DATE              31082

INPUT
.......

COMPONENT                             GRAMS IN
..........                            .........
MAF LIGNITE                            201.193
H20                                    100.596
ASH                                     25.825
HAO-61                                 402.386
GAS                                    267.891
   TOTAL                               997.891
............................................................
OUTPUT
..........
                   GRAMS                 NET YIELDS
COMPONENT          OUT                  WT% MAF LIG
............      .......              ............
H20                 75.295                 -12.576
OIL                437.314                  17.361
SRL                 92.436                  45.944
IOM                 39.322                  19.544
ASH                 24.933                  -0.443
GAS                333.892                  32.805
   TOTAL          1003.191                 102.634

% CLOSURE                  100.531
% CONVERSION                80.456
............................................................
NORMALIZED
OUTPUT
..........
                   GRAMS                 NET YIELDS
COMPONENT          OUT                  WT% MAF LIG
............      .......              ............
H20                 74.897                 -12.773
OIL                435.004                  16.212
SRL                 91.948                  45.701
IOM                 39.114                  19.441
ASH                 24.801                  -0.509
GAS                332.128                  31.928
   TOTAL           997.891                 100.000

% CLOSURE                  100.000
% CONVERSION                80.559
............................................................
TIME 0.2 SECS
```

```
RUN NUMBER        M4                  DATE                   31682

INPUT
........

COMPONENT                              GRAMS IN
...........                            ..........
MAF LIGNITE                             198.715
H20                                      99.357
ASH                                      25.499
HAO-61                                  397.429
GAS                                     267.895
   TOTAL                                988.894
...............................................................
OUTPUT
...........
                    GRAMS                          NET YIELDS
COMPONENT           OUT                           WT% MAF LIG
.............       ........                      .............
H20                   80.124                            -9.679
OIL                  437.993                            20.413
SRL                   90.826                            45.707
IOM                   32.835                            16.524
ASH                   25.421                            -0.039
GAS                  326.472                            29.478
   TOTAL             993.671                           102.404

% CLOSURE                     100.483
% CONVERSION                   83.476
...............................................................
NORMALIZED
OUTPUT
...........
                    GRAMS                          NET YIELDS
COMPONENT           OUT                           WT% MAF LIG
.............       ........                      .............
H20                   79.739                            -9.872
OIL                  435.887                            19.354
SRL                   90.390                            45.487
IOM                   32.677                            16.444
ASH                   25.299                            -0.101
GAS                  324.903                            28.688
   TOTAL             988.894                           100.000

% CLOSURE                     100.000
% CONVERSION                   83.556
...............................................................
TIME 0.2 SECS
```

```
RUN NUMBER        M5                DATE              32382

INPUT
........

COMPONENT                            GRAMS IN
..........                           .........
MAF LIGNITE                           199.322
H20                                    99.661
ASH                                    25.573
HAO-61                                398.644
GAS                                   267.887
   TOTAL                              991.087
...........................................................
OUTPUT
...........
                  GRAMS                 NET YIELDS
COMPONENT         OUT                  WT% MAF LIG
............      ........             .............
H20                58.753                  -20.523
OIL               451.478                   26.507
SRL                80.643                   40.458
IOM                18.187                    9.125
ASH                25.639                    0.033
GAS               354.904                   43.656
   TOTAL          989.603                   99.256

% CLOSURE                 99.850
% CONVERSION              90.875
...........................................................
NORMALIZED
OUTPUT
...........
                  GRAMS                 NET YIELDS
COMPONENT         OUT                  WT% MAF LIG
............      ........             .............
H20                58.841                  -20.479
OIL               452.154                   26.846
SRL                80.763                   40.519
IOM                18.214                    9.138
ASH                25.677                    0.052
GAS               355.436                   43.923
   TOTAL          991.086                  100.000

% CLOSURE                100.000
% CONVERSION              90.862
...........................................................
TIME 0.2 SECS
```

```
RUN NUMBER          M6                DATE                33182

INPUT
........

COMPONENT                              GRAMS IN
...........                            .........
MAF LIGNITE                             199.810
H20                                      99.905
ASH                                      25.663
HAO-61                                  399.621
GAS                                     266.958
   TOTAL                                991.957
............................................................
OUTPUT
...........
                   GRAMS                    NET YIELDS
COMPONENT          OUT                      WT% MAF LIG
............       .......                  ............
H20                 54.109                     -22.920
OIL                455.506                      27.969
SRL                 78.223                      39.149
IOM                 17.341                       8.679
ASH                 25.320                      -0.172
GAS                334.751                      33.929
   TOTAL           965.250                      86.634

% CLOSURE                    97.308
% CONVERSION                 91.321
............................................................
NORMALIZED
OUTPUT
...........
                   GRAMS                    NET YIELDS
COMPONENT          OUT                      WT% MAF LIG
............       .......                  ............
H20                 55.606                     -22.171
OIL                468.109                      34.277
SRL                 80.388                      40.232
IOM                 17.821                       8.919
ASH                 26.021                       0.179
GAS                344.013                      38.564
   TOTAL           991.957                     100.000

% CLOSURE                   100.000
% CONVERSION                 91.081
............................................................
TIME 0.2 SECS
```

```
RUN NUMBER          M7                  DATE                    60482

INPUT
........

COMPONENT                                GRAMS IN
..........                               .........
MAF LIGNITE                               200.443
H20                                       100.221
ASH                                        25.730
HAO-61                                    400.886
GAS                                       267.899
   TOTAL                                  995.178
.............................................................
OUTPUT
...........
                   GRAMS                     NET YIELDS
COMPONENT          OUT                       WT% MAF LIG
............       .......                   ............
H20                 43.302                       -28.397
OIL                446.000                        22.507
SRL                 85.591                        42.701
IOM                 17.162                         8.562
ASH                 26.286                         0.277
GAS                367.507                        49.694
   TOTAL           985.846                        95.344

% CLOSURE                   99.062
% CONVERSION                91.438
.............................................................
NORMALIZED
OUTPUT
...........
                   GRAMS                     NET YIELDS
COMPONENT          OUT                       WT% MAF LIG
............       .......                   ............
H20                 43.712                       -28.192
OIL                450.222                        24.613
SRL                 86.401                        43.105
IOM                 17.324                         8.643
ASH                 26.534                         0.402
GAS                370.986                        51.430
   TOTAL           995.178                       100.000

% CLOSURE                  100.000
% CONVERSION                91.357
.............................................................
TIME 0.2 SECS
```

APPENDIX 6

COMPARISON BETWEEN THE NORMALIZED NET YIELDS OF RUN M6 AND THE NET YIELDS FOR RUN M6 CALCULATED BY ASSUMING ALL LOST MATERIAL WAS PRODUCT GAS

TABLE 7

COMPARISON BETWEEN THE NORMALIZED NET YIELDS OF RUN M6 AND THE NET YIELDS FOR RUN M6 CALCULATED BY ASSUMING ALL LOST MATERIAL WAS PRODUCT GAS

	Net Yields as Wt % MAF lignite Charged						
Run #	H_2O	Oil	SRL	IOM	Ash	Gas	% Conversion
M6a	-22.2	34.3	40.2	8.9	0.2	38.6	91.1
M6b	-22.9	28.0	39.1	8.7	-0.2	47.3	91.3

a - Normalized Net Yields

b - Net Yields determined by assuming all lost material was product gas

APPENDIX 7

LIST OF ABBREVIATIONS

APPENDIX 7

LIST OF ABBREVIATIONS

U.S.	United States
H/C	Hydrogen to Carbon Ratio
EDS	Exxon Donor Solvent Process
SRC	Solvent Refined Coal Process
CO-STEAM	Carbon Monoxide and Steam
PETC	Pittsburgh Energy Technology Center
Min.	Minute
psig	Pounds per square inch, gauge
°C	Degrees Celcius
CPU	Continuous Process Unit
lb.	Pound
hr.	Hour
UND	University of North Dakota
Co.	Company
U.S.D.I.	United States Department of Interior
PDU	Process Development Unit
SRL	Solvent Refined Lignite
GFETC	Grand Forks Energy Technology Center
THF	Tetrahydrofuran
HAO-61	Hydrogenated Anthracene Oil from Run 61
AO-4	Anthracene Oil - Batch 4
C.P.	Chemical Pure
psi	Pounds per square inch
gal.	Gallon
ASTM	American Society of Testing Materials
MAF	Moisture-Ash free
cu.	Cubic
ft.	Feet
Å	Angstrom
gm, gms	Grams
Mg	Milligrams
IOM	Insoluble Organic Matter
et al.	And others

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