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The Effect of Two Catalysts (Ammonia and Quinoline) on The Liquefaction of North Dakota Lignite

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THE EFFECT OF TWO CATALYSTS (AMMONIA AND QUINOLINE)
ON THE LIQUEFACTION OF NORTH DAKOTA LIGNITE

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has been done.

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This thesis meets the standards for appearance and con-
forms to the style and format requirements of the Graduate
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ABSTRACT

The effects of two disposable catalysts on liquefaction of lignite were evaluated by comparison of overall conversions and yields of distillate. All experiments were conducted using a tubing bomb reactor at temperatures ranging from 400^OC to 440^OC for a period of twenty minutes.

Using ammonia, a reaction temperature of 420^OC and a reducing gas of either synthesis gas or carbon monoxide gave maximum yield of distillate. While ammonia promotes the formation of distillate, overall conversion is not increased. Quinoline had no significant effect on the overall conversion and distillate yield, except in the presence of hydrogen at 420^OC.

Chapter I

INTRODUCTION

During the past decade the unpredictable international energy market and large increases in petroleum prices have had profound effects on the economic life of the United States and other nations. In spite of this, world demand for petroleum is growing and world oil supplies face the danger of depletion in the next few decades (1). Thus the energy strategies of industrial nations turn towards developing alternative sources of liquid fuels. One such alternative is direct liquefaction of coal.

Coal is the most abundant fossil resource and must play a key role in supplying energy and chemicals for the future in the United States. Low-rank coals represent a major hydrocarbon resource for the United States (2). The extensive low-rank coal resources of the United States are located in the Western states, with 98 percent of the lignite in North Dakota and Montana (3). In addition to its abundance, the lignite of North Dakota has some characteristics such as low cost, high moisture content, and high reactivity that make it attractive for certain liquefaction processes.

Most direct liquefaction processes react coal and a solvent (or a process derived oil) with hydrogen gas in the presence of a catalyst at elevated temperatures and pressures ($\underline{4}$). One important drawback in direct liquefaction of coal is the use of commercial catalysts that are expensive, have short life, and cannot be recovered or regenerated. The answer to this problem is to find low cost disposable catalysts.

The major objective of this investigation was to study the effect of two disposable catalysts (ammonia and quinoline) on lignite liquefaction using pure carbon monoxide gas, pure hydrogen gas, and a synthesis gas (H_2/CO) at three temperatures; 400, 420, 440°C. The added catalyst was five percent of moisture-ash-free (maf) coal when using anthracene oil as the vehicle solvent.

Ammonia and quinoline were chosen as lignite liquefaction catalysts for this study because they are basic nitrogen compounds, and thus, may reduce hydrogen consumption, and may be selective in the production of liquids.

Chapter II

LITERATURE SURVEY

Very little is reported in current literature concerning the effect of basic nitrogen catalysts on lignite liquefaction yields. However, there is a considerable body of work on lignite liquefaction using other catalysts. It has also been reported that reaction temperature, reaction pressure, reaction time, and type of reducing gas are parameters affecting the yields of coal-derived liquid products and the overall conversion.

2.1 DIRECT LIQUEFACTION

Coal hydrogenation studies done by Fisher and Eisner (5) and by Pelipetz, Kuhn, Friedman and Storch (6) indicated optimum coal conversions using hydrogen at operating pressures of 4,000 to 6,000 psig and a reaction time of three hours. Del Bel, Friedman, Yavorsky and Wender (7) studied the effects of temperature, pressure and reaction time on lignite hydrogenation using carbon monoxide and steam and synthesis gas ($CO+H_2$) as reducing gases. Using carbon monoxide and steam, a maximum conversion of 72.6 percent of the maf-coal was obtained at $400^{\circ}C$, 4000 psig, two hours reaction time, and 8.5 percent added water. As the temperature was in-

was that the formation of benzene-soluble material proceeded much more rapidly than when hydrogen was used. In their report, they also indicated that even when the operating pressure of hydrogen was significantly higher than the combined pressure of steam and carbon monoxide the conversion of low-rank coal using carbon monoxide and steam was greater than that obtained using hydrogen. The behavior of carbon monoxide and water with lignite is unusual because of the relatively low optimum temperature of 380° to $400^\circ C$. Above $400^\circ C$ the overall conversion decreased because of the carbonization of some of the reactive compounds formed in the liquefaction reaction. In order to reduce cost, they conducted a experiment using synthesis gas instead of carbon monoxide. With two parts by weight of solvent containing 20 percent isoquinoline for each part of coal and water, it was possible to operate near 3,000 psi using either synthesis gas or carbon monoxide. Overall conversions of 85 percent of the maf-coal and oil yields of 74 percent were obtained (12).

Knudson, Willson, and Baker (13) conducted batch studies on the liquefaction kinetics of Beulah lignite and indicated that carbon monoxide undergoes reactions with lignite that are kinetically more favorable than those with hydrogen. They also reported the reactions are favorable to liquefaction throughout the temperature range 350°-$480^\circ C$.

Willson, Severson, Knudson, Owens, Baker, Farnum and Sou-
by (14) compared liquefaction yields for Big Brown Texas
lignite with those for North Dakota lignite. Their report
indicated an increase in both SRL (Solvent Refined Lignite)
and overall conversion of Zap lignite when synthesis gas was
used instead of hydrogen. In the presence of carbon monox-
ide and water, higher SRL yields and overall conversions
were obtained. These results indicated that the ability to
donate hydrogen and stabilize weak free radicals when using
carbon monoxide-water was better than when using pure hydro-
gen. As the temperature increased from 380° to $460^O C$, the
total conversion increased from 46 to 98 percent of the
maf-coal. Batch autoclave studies showed that SRL and dis-
tillate yields peaked at $420^O C$ using synthesis gas (H_2/CO)
as a reducing gas.

Willson, Knudson, Baker, Owens, and Severson (15) inves-
tigated the effects of temperature and slurry coal concen-
tration on product yields and quality for the liquefaction
of Beulah lignite. They reported that increasing the coal
concentration in the feed slurry had no effect on the THF-
insoluble residue at temperatures of either $480^O C$ or $500^O C$.
Runs were made at 440°, 460°, and $480^O C$ using anthracene oil
as a solvent and synthesis gas as a reducing gas to deter-
mine the effect of temperature. Increasing the temperature
from 440° to $480^O C$ resulted in an increase in overall con-
version from 64 percent to 80 percent and an increase in

distillate yield when the temperature increased from 440° to 460°C followed by a decrease when the temperature reached 480°C.

Work done by Sondreal, Knudson, Schiller and May (16) indicated that CO-steam liquefaction in general involves two kinds of reactions: 1) reactions occuring over the entire range of temperature investigated (350°-380°C) involve a rapid consumption of carbon monoxide and production of a major portion of the total yield of product oil (either soluble oil or distillate oil), and 2) reactions which occur rapidly at or above about 460°C which involve depolymerization of the heavy ends that have been solublized but not liquefied.

Spencer (17) indicated that coal liquefaction improves at hydrogen partial pressures of 1000-1200 psia and liquefaction temperatures of 418°-427°C. The liquid yields were much higher than anticipated for this temperature range.

2.2 CATALYSTS IN DIRECT LIQUEFACTION

Overall conversion and liquid product yield may be favorably affected by a variety of catalysts. Appell, Wender and Miller (11,12) reported that the CO-steam reaction was promoted by addition of sodium formate, alkali carbonates, iron oxide, metal chlorides or basic nitrogen. They also indicated that the benefit of basic nitrogen in compounds was to

increase the boiling point of the solvent. The increase in conversion may be the result of more materials from the solvent remaining in the liquid phase at operating conditions.

Spencer (17) reported that materials containing basic nitrogen compounds are beneficial in coal liquefaction based on work done at the Pennsylvania State University. Experiments were conducted on various sequential elution solvent chromatography (SESC) fractions of solvent refined coal. Results show there is little or no coke formed in the fraction that contains basic nitrogen compounds and some phenolic compounds. They explained that in this fraction condensation and polymerization were prevented from occuring because of the presence of basic nitrogen compounds. They also indicated that quinoline and tetrahydroquinoline are good hydrogen donor agents.

Fu and Lllig (18) have liquefied bituminous coal by hydrotreating with synthesis gas ($CO+H_2$) at temperatures of 400°-450^OC and operating pressures of 3,000-4,000 psi in the presence of cobalt molybdate catalyst, steam, and recycle oil. They reported 94 percent conversion. On comparing liquefaction of coal with synthesis gas with liquefaction of coal using pure hydrogen and cobalt molybdate catalyst, they reported that the optimum liquefaction temperature was in the range of 425°-450^OC. There was no significant difference in the overall conversion and the oil yield in the

temperature range of $425^{\circ}-450^{\circ}C$ with cobalt molybdate as a catalyst.

Early work by Wright and Severson (19) on the catalyst activity of coal minerals clearly illustrated that the naturally occuring minerals in lignite had a catalytic effect on the reaction of hydrogen or carbon monoxide with lignite.

Souby, Severson, and Kube (20) reported that cations, particularly those of sodium and calcium, have a catalytic effect on solution-hydrogenation of lignite. They also indicated that cations occuring naturally in North Dakota lignite may be sufficient for good solution characteristics.

Mobil Research and Development Corporation (21), investigating coal conversion in basic nitrogen heterocyclic compounds, concluded that quinoline and benzoquinoline were good hydrogen transfer agents because they are basic nitrogen heterocyclic compounds. Like pyrene, quinoline reacts readily with molecular hydrogen and forms an active H-donor, tetrahydroquinoline. The effect of temperature on the overall conversion of Monterey coal was investigated using quinoline as a solvent in the presence of hydrogen gas. It was reported that for an increase in temperature from 750° to $800^{\circ}F$ at constant pressure (1000 psig), the overall conversion dropped from 85.5 to 63.6 percent of the maf-coal.

Tomita, Tano, Oikawa, and Tamai (22) investigated two
Japanese and three German coals ranging in rank from high-
volatile C bituminous coal to semi-anthracite which were
treated with liquid ammonia as a non-aqueous solvent at
373°K and 10 MPa. Results showed that the atomic H/C ratio
of the extract by liquid ammonia was larger than that of the
parent coal indicating a less aromatic nature of the ex-
tract.

Matida, Nishiyama, and Tamai (23) investigated a Japanese
bituminous coal which was treated with liquid ammonia at
temperatures to 120°C. They reported that many nitrogen-
containing compounds are powerful solvents for coal. Con-
sidering the basic nature and small molecular dimension, am-
monia can penetrate a coal particle and loosen the coal
structure when contact is made at elevated temperatures.
Thus, compounds of small molecular units can escape from the
coal particle. Although ammonia is far from being a degrad-
ing or reactive solvent, it may well react with some of the
functional groups in coal. Impregnation of catalyst into
microcracks may prevent coal particles from caking, and
therefore, preserve the effective surface area for reaction.

Harris, Simons, and Lagowski (24) conducted extraction
experiments on Rockdale (Texas) lignite using pure liquid
ammonia. They concluded that the original lignite structure
can be modified and lignite separated into its component
moieties by treatment with liquid ammonia.

The current literature review indicates a lack of data on catalytic liquefaction of lignite using basic nitrogen catalyst such as alpha-picoline, beta-collidine, quinoline, iso-quinoline, ammonia, 2-phenylypyridine, 7,8-benzoquinoline pyridine, and 1,2,3,4-tetrahydroquinoline. Thus, it is seen that it will be important to determine the effect of basic nitrogen catalysts on yields of lignite liquefaction.

Chapter III

EXPERIMENTAL

3.1   MATERIALS

The lignite used in all experiments was obtained by the
University of North Dakota Energy Research Center (UNDERC)
from the Indian Head Mine of the North American Coal Company
near Zap in Mercer county. It was pulverized at the UNDERC.
Size distribution, proximate analysis and ultimate analysis
are given in Table 1.

The anthracene oil solvent used in the feed slurry was
obtained from Crowley Tar & Chemical. Anthracene oil from
batch number four (AO_4) was used in this study; analyses are
shown in Table 2.

Quinoline, obtained from Crowley Tar & Chemical, was C.P.
grade. Ammonia, carbon monoxide, hydrogen and synthesis gas
(51.8 mole % CO and 48.2 mole % H_2) were obtained from the
Linde Division of Union Carbide.

The following chemicals were also used: histological
grade tetrahydrofuran (THF), reagent grade cyclohexane and
C.P. grade methanol.

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TABLE 1

ANALYSES OF ZAP LIGNITE[*]

Size Distribtion

U.S. Mesh 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 (after pulverization)

Ultimate Analysis[b]

Constituent	Percent
Carbon	62.1
Hydrogen	3.8
Nitrogen	1.0
Sulfur	1.0
Oxygen (by diff.)	21.0
Ash	11.1
Total	100.0

[b] moisture free basis

[*] data obtained from UNDERC

TABLE 2

ANALYSES OF SOLVENT*

Solvent	AO_4(1)
ASTM D-1160 Distillation @ 5 torr	
IBP, $^{\circ}$C	94
Vol. % off at, $^{\circ}$C	135
10	146
20	163
30	175
40	185
50	195
60	205
70	217
80	231
90	251
95	276
Max. Temp., $^{\circ}$C	288
Vol. % off at Max. Temp.	96.5
Calculated from ASTM D-1160	
IBP - 120°C Fraction, Wt. %	3.1
120 - 260°C Fraction, Wt. %	85.0
260° - Max. Temp. Fraction, Wt. %	7.6
Vacuum Bottoms, Wt. %	4.3
Density, gms/ml @ RT	1.1
Elemental analysis, Wt. %	
Carbon	90.2
Hydrogen	5.9
Nitrogen	0.8
Sulfur	0.7
Oxygen (by difference)	2.4
H/C Ratio	0.79

(1) As received anthracene oil from Crowley Tar & Chemical

* data obtained from UNDERC

3.2   EQUIPMENT

The apparatus used was UNDERC's tubing bomb reactor sys-
tem which is capable of running twelve separate tests at the
same time. The complete tubing bomb apparatus is shown in
Figure 1. The reactor design and experimental procedures
are similar to those used by Curran, Struck, and Gorwin (25)
and Neavel (26). Each tubing bomb consists of a 16-inch
long 316 stainless steel tube, 0.359-inch O.D., 0.312-inch
I.D., equipped with Autoclave Engineer's cone and thread
fittings. One end of each tubing bomb is sealed with an end
plug and the other end is connected via a reducing coupler
to a 23.0-inch long, 0.25-inch O.D., 0.625-inch I.D. support
neck terminating in a three port metering valve (Autoclave
Engineers). Details of the tubing bomb assembly are shown
in Figures 2 and 3.

The tubing bomb reactors were agitated up and down at a
frequency of 240 cycles per minute by the mechanism shown in
Figure 4. A fluidized sand bath was used to heat the reac-
tors. The sand was heated using three band heaters wrapped
around the outside of sand bath. The heaters were connected
to a Barber Colman series 120 analog setpoint controller.

Temperatures were continuously measured and recorded us-
ing type J thermocouples connected to a Leeds and Northup
Speedmax W twelve point chart recorder. The lift apparatus
used for elevating the reactors is shown in Figure 5.

Figure 1:   Complete tubing bomb apparatus
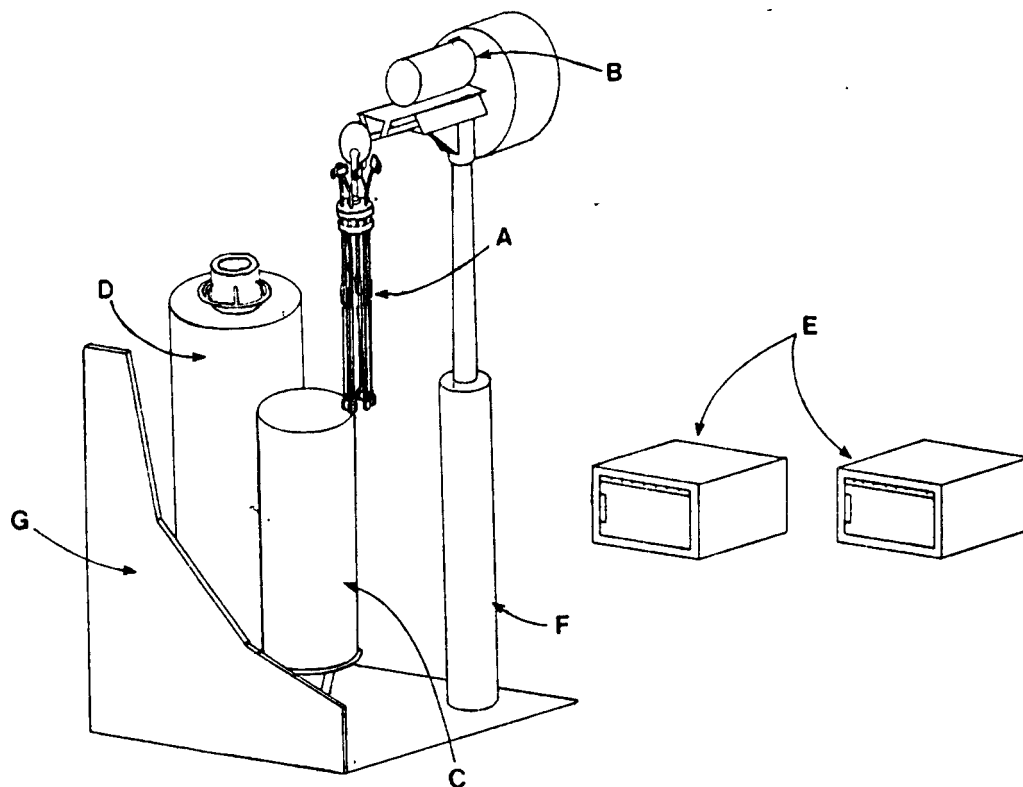
A.   TUBING BOMBS

B.   VARIABLE SPEED SHAKER

C. H_2O QUENCH BATH

D.   SAND BATH

E.   PRESSURE AND TEMPERATURE RECORDERS

F.   LIFT CYLINDER

G.   PLATFORM AND CONTROL PANEL

Figure 2: Tubing bomb assembly
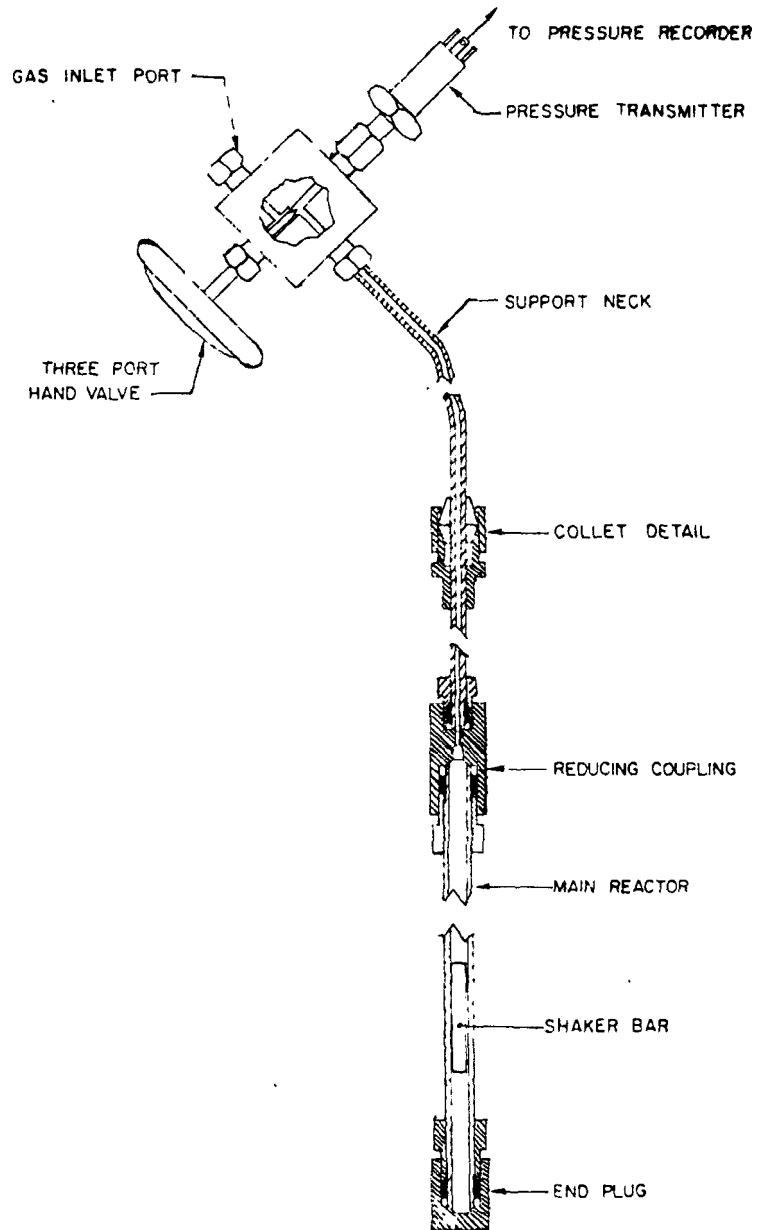
Figure 3: Tubing bomb assembly and piston and connecting
rod detail
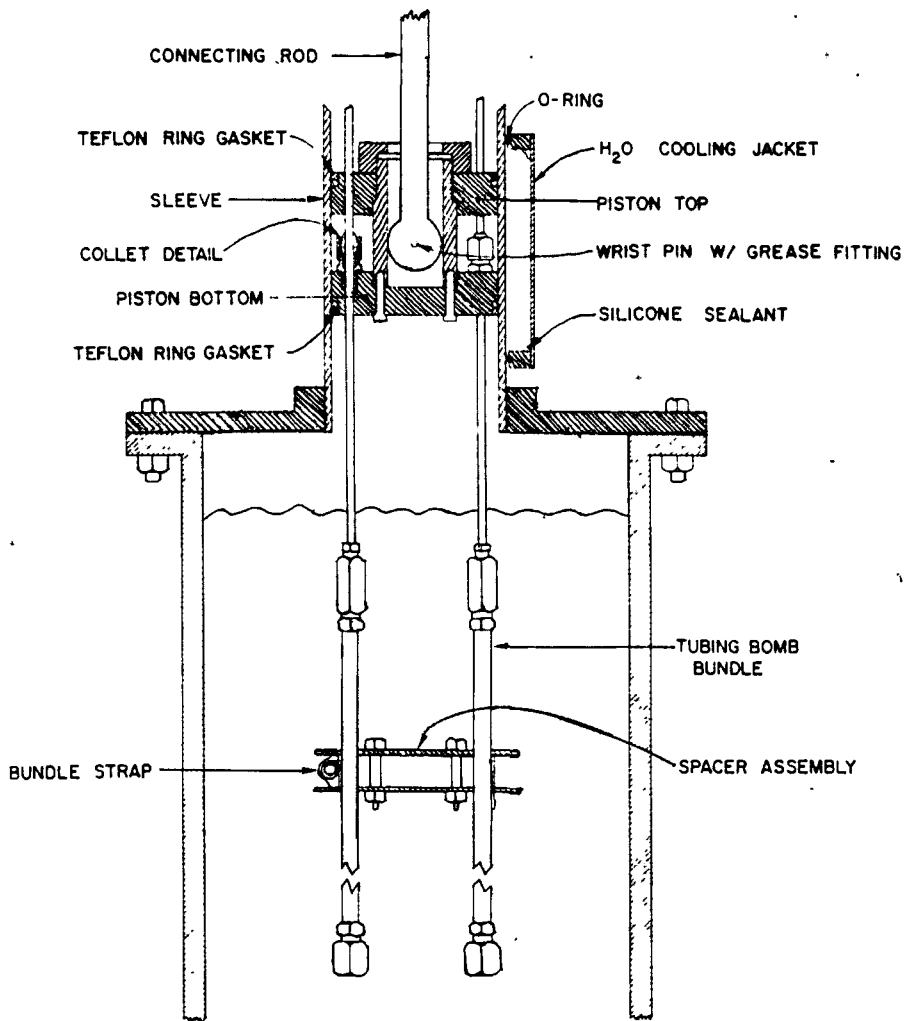
Figure 4: Tubing bomb apparatus-shaker assembly detail
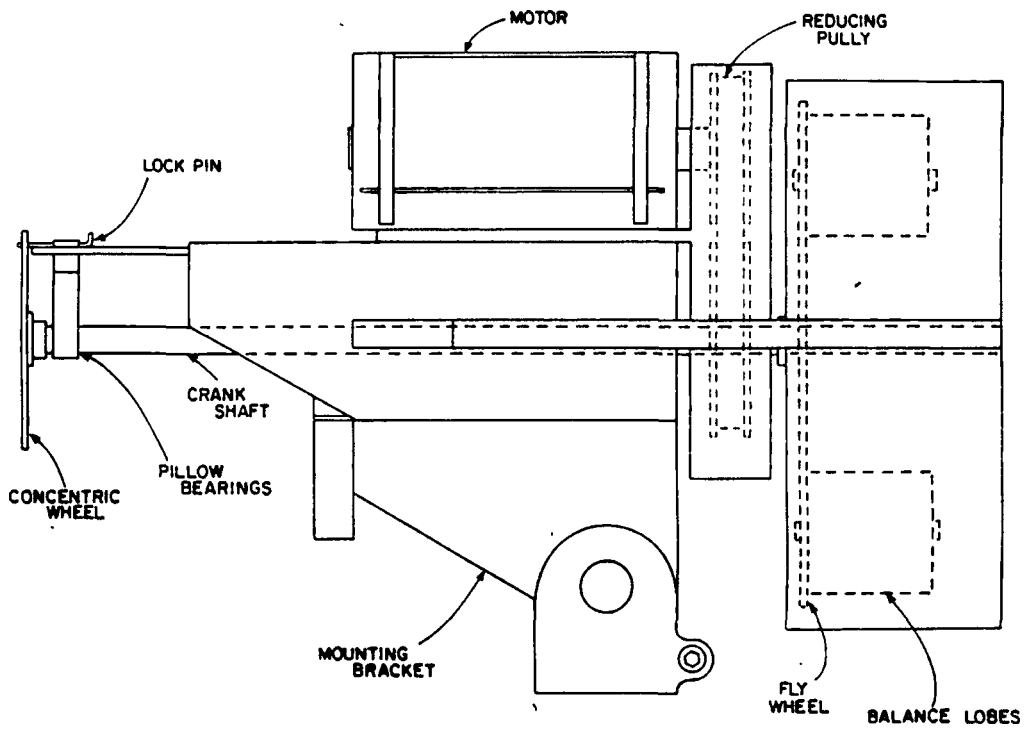
Figure 5: Tubing bomb apparatus-lift detail

AIR
VENT

TO
SHAKER
ASSEMBLY

4" by 37.5" THROW / HYDRAULIC
RAM

AIR
SUPPLY

LEVELING
SPOOLS

2 GALLON
OIL /AIR RESERVOIR

WORKING FLUID
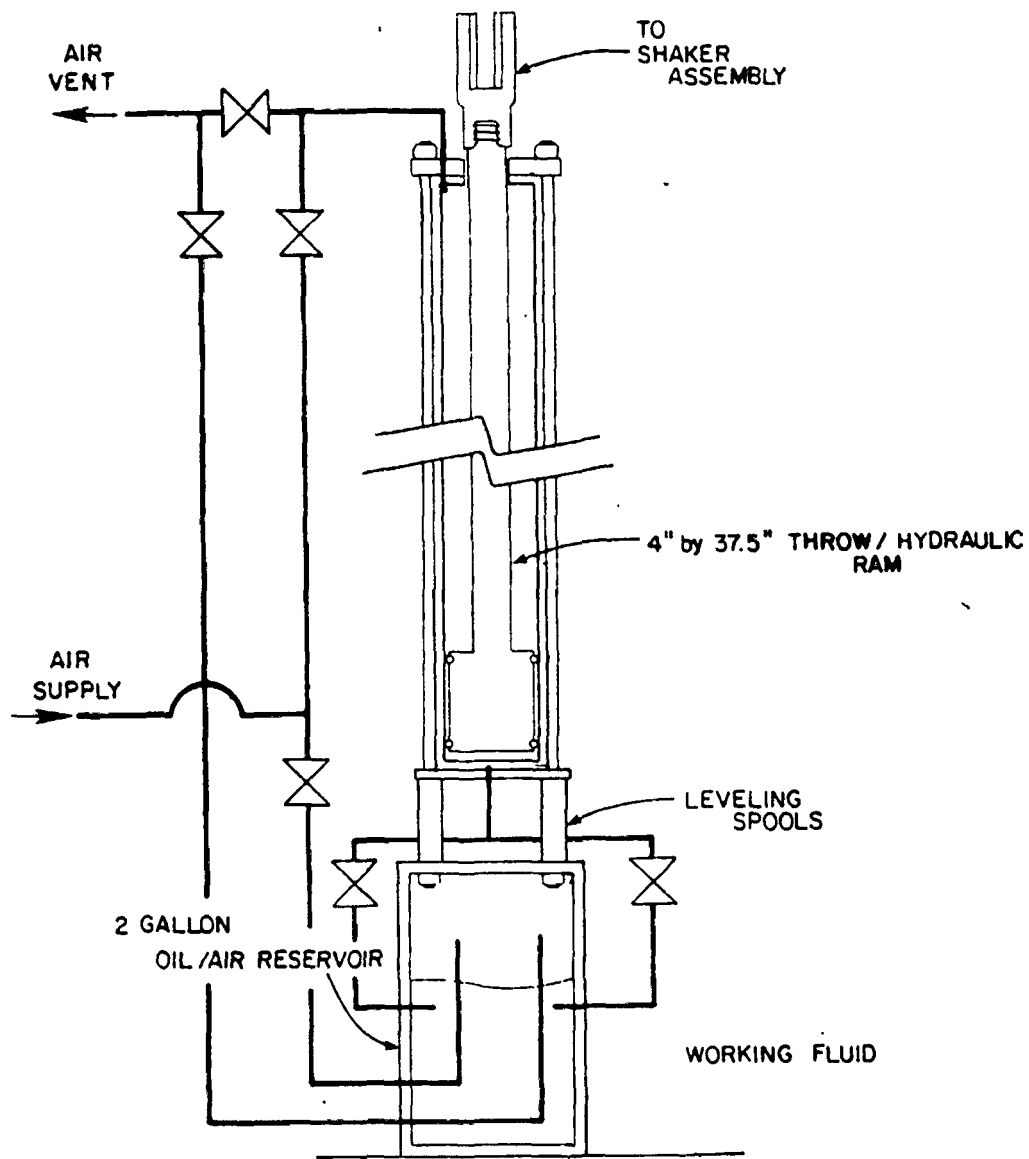
3.3 EXPERIMENTAL PROCEDURES

Operating procedures can be presented in four parts:

1. slurry preparation and reducing gas charge;

2. operation;

3. product gas and product slurry removal; and

4. analysis.

3.3.1 Slurry Preparation and Reducing Gas Charge

1. The sand bath was preheated to the desired temperature. Usually that temperature should be higher than the desired reaction temperature by about $40^{\circ}F$. A heating rate of about 4° to $12^{\circ}F$ per minute was used requiring a total heat-up time of two hours.

2. Approximately 1.5 grams of moisture-ash-free (MAF) lignite, 3 grams of anthracene oil (AO_4) and 0.08 grams of catalyst (ammonia or quinoline) were charged directly to each tubing bomb. The moisture and ash contents of Zap lignite were determined before each run by American Society for Testing Materials (ASTM) procedures D3173 and D3174, respectively.

3. The shaker bar was inserted into each tubing bomb, the tubing bombs were sealed with a reducing coupling, and the bombs were connected to the support neck.

4. Each tubing bomb was pressurized to 1000 psig (800 psig when ammonia was the catalyst) with different

reducing gases (CO, H_2 or sythesis gas). The pressure was set using a Linde SG-3840 high pressure regulator (0-1500 psi) attached directly to the reducing gas tank. After the reducing gas was charged to the tubing bomb, water was used to detect leaks in the reducing coupling and the end plug.

3.3.2 Operation

A wrist pin was used to connect the bundle of tubing bomb reactors (12 tubing bombs) to the connecting rod and a bundle strap was used as a spacer to fix the position of each tubing bomb reactor. The tubing bomb reactors were held in place using two pieces of round metal plate. The metal plates were the same size as the airtight attachment that was used as a guide for the vertical motion of the tubing bomb reactors. When the sand bath reached the desired temperature, the vertically oriented bundle of tubing bombs was plunged into the preheated sandbath and agitated vertically at a rate of 240 times per minute. The effective reaction time was considered to begin one minute after insertion. After a reaction time of twenty minutes, the reactors were quickly removed from the sand bath and plunged into an adjacent water quench tank. Cool-down in the water tank was continued until the next day.

3.3.3   Product Gas and Product Slurry Removal

A gas sample bomb (11.21 cc) was used to measure the weight of the product gas. The sample bomb was connected to the gas inlet port of the tubing bomb. The valves of the gas sample bomb and the tubing bomb were opened for several seconds to allow the system to reach equilibrium; then they were closed. An analytical balance was used to weigh the gas sample bomb. Then the product gas was removed from both the sample bomb and the tubing bomb.

The tubing bombs were opened and the slurry products were carefully removed from the reactors to previously weighed sample vials by pushing a close-fitting Nylon plunger through the tubing bombs. The product slurry remaining in tubes and valves was recovered using preweighed disposable wipes in order to determine the weight of the product slurry.

3.3.4   Analysis

Product slurry was analyzed to determine moisture content, solubility in cyclohexane and solubility in THF.

3.3.4.1 Moisture Determination

Moisture content of the product slurry was measured by the Karl Fischer method using a Photovolt Aquatest IV automatic titrator. The sample vial was well stirred to insure a uniform sample of product slurry. Approximately 15 to 20 mg of product slurry was added to an anhydrous methanol solution using a disposable pipette. This solution was then titrated with Karl Fischer reagent (KFR) until the conductance meter needle deflected to the KFR zone (water free end-point zone) and the needle remained stable in that zone.

3.3.4.2 Cyclohexane and THF Extractions

Approximately 0.25 grams of product slurry, weighed to the nearest 0.1 mg, was extracted with 100 ml of cyclohexane and filtered through a previously weighed 0.5 micro filter (Millipore, type FH) using dry nitrogen (25 psig) as the filtration gas. The filter cake was washed with additional cyclohexane until the wash liquid was clear. Then the filter cake was dried in an oven at $105^{\circ}C$ for 15 minutes and reweighed.

The residue and filter paper from the cyclohexane extraction were extracted with 100 ml of THF using the same pressure filtration procedure. The filter cake was then washed with more THF until the wash liquid was clear. The filter cake and filter paper were dried in an oven at $105^{\circ}C$ for 15 minutes and weighed.

Chapter IV

RESULTS AND DISCUSSION

Experimental conditions for all runs are summarized in
Appendix D, Tables 9 through 13. A summary of gas, distil-
late, soluble residue,and liquid product yields; overall
conversion; and material balance closures for every run is
given in Appendix E, Tables 14 through 18. It should be
noted that high distillate yield is desirable for all runs
of this study. The computer program[1] used to perform calcu-
lations and the notation used in the program are shown in
Apppendix A. Appendix B contains the data sheets and the
printouts of the results for all runs. Appendix C shows the
detailed calculations for Run-T002 (A).

4.1   REPRODUCIBILITY

Material balance closures ranged from 87.6% to 101.2%
without a catalyst, 93.5% to 100.7% with the quinoline cata-
lyst and 95.1% to 102.8% with the ammonia catalyst. Five of
these closures (T001-A,I; T002-K; T003-D,L) were out of nor-
mal range (94%-106%) for the tubing bomb reactor system.
The results obtained for these cases were disregarded as be-
ing unreliable. Results were normalized by assuming lost or

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[1] obtained from UNDERC

gained material to be proportionally distributed among all products. To check experimental techniques, all samples were duplicated in each run. A 95 percent confidence interval was calculated based on replications for overall conversion, distillate yield, soluble residue yield and gas yield for tests T001-A,E,J, T002-A,C,E,G,I,K, T003-A,C,E,G,I,K, and T004-A,C,E,G,I,K, to determine whether they gave significantly different results. Statistical calculations are given in Appendix F.

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

The distillate yield is defined as net increase in weight of distillate from microdistillation at 205°C and 1 torr expressed as the weight percent of MAF lignite charged.

The yield of soluble residue (S-Res) is the weight of residue from microdistillation that is soluble in THF (tetrahydrofuran) expressed as the weight percent of MAF lignite charged.

The yield of liquid product is defined as the distillate yield plus the soluble residue yield expressed as the weight percent of MAF lignite charged.

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

The overall conversion is defined as 100 minus the ash-free THF-insoluble material, expressed as the weight percent of MAF lignite charged.

4.2 DISCUSSION

Table 3 provides "base-line" data for comparison with results obtained in the presence of catalysts. Temperature and the type of gas were varied to study the effects on lignite liquefaction.

TABLE 3

RESULTS OF USING NO CATALYST

I.D. Number	Tempe- rature	Reducing Gas	Yields, Wt% of MAF Lignite			
			Gas	Dist	S Res	Conv
T005-A	400°C	CO	0.8	21.0	22.6	50.6
T001-A/B	420°C	CO	-14.3	43.3	10.4	45.1
T005-E	400°C	H_2	13.2	15.2	12.4	44.6
T001-E/F	420°C	H_2	14.6	13.0	-0.8	27.0
T005-I	400°C	H_2/CO	2.3	17.8	21.9	48.5
T001-I/J	420°C	H_2/CO	7.85	32.5	8.7	37.0

4.3   EFFECT OF AMMONIA CATALYST

Results from liquefaction experiments in the presence of ammonia catalyst are compared with liquefaction data obtained without a catalyst. Product yields are tabulated in Table 4.

As the temperature increased from 400° to 440^OC, there was no significant difference in conversion for either carbon monoxide or synthesis gas atmospheres. In pure hydrogen, conversion increased with increasing temperatures above 420^OC. Thus, we may say that increasing temperature does not benefit overall conversion for synthesis gas and carbon monoxide. However, in the presence of hydrogen, it is beneficial to increase the temperature. This need for higher temperatures may be due to the greater consumption of hydrogen at the higher temperatures, and also because hydrogen promotes cracking at higher temperatures.

Data in Table 4 shows that the distillate yield increased with temperature in the presence of carbon monoxide. In synthesis gas and hydrogen, the distillate yield peaked at 420^OC. For carbon monoxide, the soluble residue yield peaked at 420^OC. Synthesis gas and hydrogen showed increases in soluble residue yield with an increased temperature. Negative yields for soluble residue were obtained using hydrogen because part of the soluble residue of the feed solvent was converted to distillate.

TABLE 4

EFFECT OF AMMONIA ON YIELDS

I.D. Number	Tempe- rature	Reducing Gas	Yields, Wt% of MAF Lignite			
			Gas	Dist	S Res	Conv
T004-A/B	400°C	CO	-4.7	35.6	2.9	38.8
T002-A/B	420°C	CO	-24.9	51.9	15.5	41.9
T003-A/B	440°C	CO	-3.3	56.8	4.6	40.1
T004-E/F	400°C	H_2	1.3	44.4	-7.1	40.2
T002-E/F	420°C	H_2	1.1	47.1	-5.8	37.6
T003-E/F	440°C	H_2	4.3	27.0	1.9	46.0
T004-I/J	400°C	H_2/CO	-4.6	36.8	0.1	39.4
T002-I/J	420°C	H_2/CO	-10.0	50.1	10.1	45.2
T003-I/J	440°C	H_2/CO	-14.6	36.3	12.4	40.4

It is also seen in Table 4 that the gas yield increased
with increasing temperature for hydrogen, while gas yield
decreased when using synthesis gas. For carbon monoxide,
the gas yield was negative meaning there was a net decrease
in the mass of gas as the reaction proceeded. In every in-
stance, the gas yield in the presence of ammonia was less
than when there was no added catalyst. The relatively large
negative gas yield at $420^{\circ}C$ when using carbon monoxide as
the reductant, as shown in Table 3, is even more pronounced
when ammonia was added. It appears that the presence of
carbon monoxide, at the conditions studied, contributes to
decreased gas production as compared with that produced when
using hydrogen as reductant, and pure carbon monoxide gives
lower gas yields than does syngas. These observations are
consistent with incorporation of carbon monoxide, or carbon
dioxide product from the water-gas shift reaction (WGSR), in
the liquid reaction products. Increased distillate yields
indicate that either carbon monoxide participates in the re-
actions with products from thermal hydrocracking, or that
the hydrogen produced in the WGSR is "more reactive" than is
hydrogen that is added initially. In any case, hydrogena-
tion appears to be favored over condensation and/or polymer-
ization reactions.

Comparing distillate yields at different temperatures
with different reducing gas, it is seen that at 420° and
$440^{\circ}C$, carbon monoxide showed a higher distillate yield than

both hydrogen and synthesis gas. But, at 400°C, hydrogen gave higher distillate yield as compared to those obtained when using either carbon monoxide or synthesis gas.

Distillate yields and overall conversions with and without catalyst are tabulated in Table 5. A value 21.0±7.0 in this table, for example, signifies that the upper limit of variation is 21.0+7.0 and the lower limit is 21.0-7.0. Thus, in Table 5 we can say that for test T005-A at 400°C without a catalyst, the true value of the distillate yield is between 14-28 percent at 95 percent confidence level.

It is seen that by comparing the lower limit of overall conversion without a catalyst with the upper limit of overall conversion using ammonia as a catalyst for all tests, there is no apparent benefit in using ammonia as a catalyst. The relatively low conversion in many of the cases is caused by the low (or negative) gas yields, and overall conversion may not be a good measure of catalyst worth.

In the case of distillate yield, there is a significant difference between the lower limit of distillate yield in the presence of ammonia and the upper limit of distillate yield without catalyst. This was true at all conditions studied except when carbon monoxide was the reducing gas and the temperature was 420°C. Thus, we can say that adding ammonia is beneficial to the formation of distillate.

TABLE 5

COMPARISON BETWEEN AMMONIA AND WITHOUT CATALYST

I.D. Number	Catalyst	Temperature	Reducing Gas	Yields, Wt% of MAF Lignite	
				Distillate	Conversion
T005-A	None	400°C	CO	21.0±7.0	50.6±3.4
T004-A/B	NH$_3$	400°C	CO	35.6±5.0	38.8±2.4
T001-A/B	None	420°C	CO	43.3±7.0	45.1±3.4
T002-A/B	NH$_3$	420°C	CO	51.9±5.0	41.9±2.4
T005-E	None	400°C	H$_2$	15.2±7.0	44.6±3.4
T004-E/F	NH$_3$	400°C	H$_2$	44.4±5.0	40.2±2.4
T001-E/F	None	420°C	H$_2$	13.0±5.0	27.0±2.4
T002-E/F	NH$_3$	420°C	H$_2$	47.1±5.0	37.6±2.4
T005-I	None	400°C	H$_2$/CO	17.8±7.0	48.5±3.4
T004-I/J	NH$_3$	400°C	H$_2$/CO	36.8±5.0	39.4±2.4
T001-I/J	None	420°C	H$_2$/CO	32.5±7.0	37.0±3.4
T002-I/J	NH$_3$	420°C	H$_2$/CO	50.1±5.0	45.2±2.4

Statistical calculations in Appendix F show that gas yield, distillate yield, soluble residue yield, and overall conversion do not show significantly different results for duplicate tests at 95 percent confidence levels.

The mechanism of hydrogenation of lignite in the presence of ammmonia is not entirely known. It is believed that some groups such as alcohols, and primary and secondary amines can form hydrogen bonds with ammonia. Ethers, tertiary amines, oxygen functions, and nitrogen heterocycles can also be hydrogen-bonded by ammonia (24). As the coal is heated, the ammonia possibly hydrogen-bonds with active reaction products and prevents condensation and polymerization by physically capping active sites that have been exposed by thermal disruption of chemical bonds in the coal "molecule". Ammonia "ties up" the active sites until conditions that favor hydrogenation are reached. Because the ammonia catalyst promotes distillate yield, it indicates that condensation and/or polymerization were prevented from occuring at the reactive sites.

4.4 EFFECT OF QUINOLINE CATALYST

The reaction catalyzed by quinoline showed a variation in the extent of lignite liquefaction for the three different reducing gases used at three different temperatures. Results are shown in Table 6.

TABLE 6

EFFECT OF QUINOLINE ON YIELDS

I.D. Number	Temperature	Reducing Gas	Yields, Wt% of MAF Lignite			
			Gas	Dist	S Res	Conv
T004-C/D	400°C	CO	3.7	27.4	14.6	54.7
T002-C/D	420°C	CO	6.9	35.4	10.4	52.5
T003-C/D	440°C	CO	7.9	29.4	2.6	47.9
T004-G/H	400°C	H_2	17.1	16.7	6.9	50.3
T002-G/H	420°C	H_2	15.7	17.6	26.4	53.9
T003-G/H	440°C	H_2	17.2	22.2	16.6	61.5
T004-K/L	400°C	H_2/CO	12.0	22.7	6.8	52.1
T002-K/L	420°C	H_2/CO	8.9	36.0	3.8	47.5
T003-K/L	440°C	H_2/CO	13.2	13.2	9.8	49.0

Work done at the Mobil Research and Development Corporation (21) indicated that quinoline has been identified as a hydrogen transfer agent. Like pyrene, quinoline reacts readily with molecular hydrogen and forms an active hydrogen donor. Advanced Fuel Research, Inc. (17) showed that hydrogen bonding exists in solvents and in the coal itself when using quinoline as a solvent. Therefore, a quinoline catalyst was thought to be capable of promoting hydrogen donating ability.

When carbon monoxide was the reducing gas, increasing the temperature resulted in decreases in the overall conversion and soluble residue yield, while the gas yield increased. These results indicate that some of the THF-soluble residue is converted to THF-insoluble material with increasing temperatures. Appell, Wender, and Miller (11) ascribed the decreased conversion above 400°C to carbonization of some of the reactive compounds formed in the liquefaction when using anthracene oil as a solvent.

When using synthesis gas as the reductant the general trend of results indicates that increasing temperature causes decreases in overall conversion and in distillate yield and increases in soluble residue yield and gas yield. This may be due to the decrease in the activity of carbon monoxide (in reducing carbonyl groups) with increasing temperature (9). These results. agree with those obtained by Appell, Moroni, and Miller (10).

With hydrogen as the reducing gas the overall conversion, the distillate yield, and the gas yield increase with an increasing temperature. The soluble residue yield in this case peaked at 420°C. The experimental data indicate that improved overall conversion and distillate yield were attained at higher temperatures in the presence of quinoline. This may be due to the increase in hydrogen activity and increased rates of the hydrocracking reaction with increasing temperatures.

Carbon monoxide gave higher overall conversion at lower temperatures, while hydrogen gave higher conversion at higher temperatures. (Results tabulated in Table 6.) This agrees with the results obtained by many investigators (9, 10, 19). They report that the overall conversion and the distillate yield of lignite improved when carbon monoxide or synthesis gas was used instead of hydrogen at low temperatures.

Comparing distillate yields for three types of reducing gases at three different temperatures, Table 6 shows that at 400° and 440°C, using carbon monoxide as the reducing gas produced higher distillate yield. But at 420°C, synthesis gas showed a higher percentage of distillate. It is also seen that for all temperatures and reducing gases the maximum distillate yield was obtained at 420°C using synthesis gas as the reductant.

Table 7 presents data for the tests with quinoline as a catalyst and tests without a catalyst. Comparing the lower limit of both distillate yields and overall conversion with a catalyst with the upper limits of these quantities without a catalyst, no significant effect in using quinoline as a catalyst is recognized. Only when using hydrogen and synthesis gas at 420°C was there significant difference in overall conversion when comparing "no catalyst" with the addition of quinoline, but the conversion and yield of distillate are not particularly attractive for liquefaction.

Even when using quinoline as a catalyst statistical calculations in Appendix F show that gas yield, distillate yield, soluble residue yield, and overall conversion do not show significantly different results for duplicate tests.

It was believed that water provides a transport medium for the basic nitrogen into the coal. Water is an excellent solvent for ionic substances because it contains the hydroxyl group and, hence, can form hydrogen bonds. Hydrogen bonding between nitrogen heterocycles and water can combine with the solvent and then penetrate the coal structure. In this experiment, quinoline does not seem to perform this function with ease. This is probably due to the relatively low solubility of quinoline in water.

TABLE 7

COMPARISON BETWEEN QUINOLINE AND WITHOUT CATALYST

I.D. Number	Catalyst	Temperature	Reducing Gas	Yields, Wt% of MAF Lignite	
				Distillate	Conversion
T005-A	None	400°C	CO	21.0±7.0	50.6±3.4
T004-C/D	C_9H_7N	400°C	CO	27.4±5.0	54.7±2.4
T001-A/B	None	420°C	CO	43.3±7.0	45.1±3.4
T002-C/D	C_9H_7N	420°C	CO	35.4±5.0	52.5±2.4
T005-E	None	400°C	H_2	15.2±7.0	44.6±3.4
T004-G/H	C_9H_7N	400°C	H_2	16.7±5.0	50.3±2.4
T001-E/F	None	420°C	H_2	13.0±5.0	27.0±2.4
T002-G/H	C_9H_7N	420°C	H_2	17.6±5.0	53.9±2.4
T005-I	None	400°C	H_2/CO	17.8±7.0	48.5±3.4
T004-K/L	C_9H_7N	400°C	H_2/CO	22.7±5.0	52.4±2.4
T001-I/J	None	420°C	H_2/CO	32.5±7.0	37.0±3.4
T002-K/L	C_9H_7N	420°C	H_2/CO	36.0±7.0	47.5±3.4

4.5 COMPARISON OF THE EFFECTS OF DIFFERENT CATALYSTS

Four catalysts, cobalt molybdate ($CoMoO_4$), iron pyrite (FeS_2), hydrogen sulfide (H_2S), and tin oxide (SnO_2) were chosen to compare with ammonia and quinoline. The effects for these four catalysts on overall conversion and the liquid product yield under similar conditions were obtained from the UNDERC (University of North Dakota Energy Research Center) (27). The comparison of the catalysts is shown in Table 8.

Cobalt molybdate is a commerical catalyst obtained from Harshaw Chemical Company, Beachwood, Ohio. It is used in the H-coal, Methanol-to-Gasoline (Mobil), and Fischer-Tropsch (Sasol-I) processes.

Mukherjee and Chowdhury (28) reported an increase in conversion with increasing mineral matter and identified iron pyrite as an active catalyst. Extensive work by Guin, Tarrer, Lee, Lo, and Curtis (29) and Wright and Severson (14) on the catalytic activity of coal minerals clearly demonstrated iron pyrite as an effective hydrogenation catalyst. Hamrin and Morooka (30) and Granoff and Thomas (31) demonstrated the effect of iron pyrite on product distribution and depicted the magnitude of the observed catalytic effect on the net oil yields.

TABLE 8

COMPARISON OF THE EFFECT OF DIFFERENT CATALYSTS

Catalyst	Temperature	Reducing Gas	Yields, Wt% of MAF Lignite	
			Liquid product	Conversion
None*	$440^{\circ}C$	H_2/CO	41.5	59.5
NH_3	$440^{\circ}C$	H_2/CO	48.7	40.4
C_9H_7N	$440^{\circ}C$	H_2/CO	23.0	49.0
$CoMoO_4$*	$440^{\circ}C$	H_2/CO	47.2	67.6
FeS_2*	$440^{\circ}C$	H_2/CO	49.2	65.8
H_2S*	$440^{\circ}C$	H_2/CO	31.8	63.1
SnO_2*	$440^{\circ}C$	H_2/CO	37.6	55.2

* obtained from UNDERC

Hettinger, Keith, Gring and Tetes (32) reported that hydrogen sulfide caused a significant increase in hydrocracking. Sondreal, Willson and Stenberg (33) demonstrated the positive effect of hydrogen sulfide on lignite liquefaction using synthesis gas.

Mizumoto, Yamashita and Matsuda (34), reported that tin oxide showed the highest activity in their experiments. The overall conversion and oil yield reached 100 percent and 75 percent, respectively. They also indicated that tin oxide had higher selectivity for hydrogenation than for recombination.

Data for lignite hydrogenation in the presence of ammonia, quinoline, cobalt molybdate, iron pyrite, hydrogen sulfide, and tin oxide are shown in Table 8 along with baseline data for hydrogenation without a catalyst.

It shows that ammonia gives a liquid product yield similar to that obtained in the presence of iron pyrite and cobalt molybdate both of which have been shown to be good hydrogenation catalysts. This effect on the liquid product yield may be attributed to catalysis of hydrogen transfer. Thus, it is seen that ammonia may be a good catalyst for lignite liquefaction. Mobil Research Development Corporation (21) reported that quinoline has good hydrogen donating ability, i.e, it is a good hydrogenation catalyst. But, from this study, it is seen that quinoline does not favor

the formation of liquid products as compared with other good hydrogenation catalysts. During coal liquefaction, various chemical bonds are broken as coal is heated to reaction temperature. Once these bonds are broken, molecules become free to react at the resulting active sites. Retrogressive reactions proceed at these sites if basic nitrogen is not available to form a hydrogen bond with those active sites. Water may serve as the carrier medium for basic nitrogen in the liquefaction reaction mixture (17). Solubility of the basic nitrogen compounds in water will determine the availability of those materials to "cap" the chemically active sites on the coal and to prevent the occurance of retrogressive reactions. Once the retrogressive reactions have taken place, the final coal-derived products may be more unreactive than the original coal. Ammonia is relatively much more soluble in water than is quinoline. This may explain the differences observed between the two basic nitrogen-containing materials in the liquefaction of lignite.

The effect of catalysts on overall conversion is also seen from Table 8. The lowest conversion was obtained using ammonia as a catalyst. This is the result of low gas yields. Generally, raising the temperature to increase distillate formation causes increased gas formation leading to poorer hydrogen efficiency and increased processing costs. Results from this study show that using ammonia as a catalyst minimized the formation of gas and thus promoted hydro-

gen efficiency. Low overall conversion was also obtained when quinoline was added, and there appears to be no benefit in using relatively expensive quinoline as a catalyst.

Chapter V

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

The following conclusions can be made from this study:

1. Ammonia catalyst promotes the formation of distillate but the overall conversion is not enhanced.

2. A reaction temperature of $420^{\circ}C$ and a reducing gas of either synthesis gas or carbon monoxide are favorable for obtaining maximum distillate yield in the presence of ammonia.

3. The addition of quinoline as a catalyst has no significant effect on the yield of distillate or on the overall conversion, except when using hydrogen at temperature of $420^{\circ}C$.

5.2 RECOMMENDATIONS

1. Further studies should be made to determine the concentration effect of ammonia catalyst on lignite liquefaction.

2. Using either carbon monoxide or synthesis gas, a series of different initial pressure runs should be made at a fixed temperature in the presence of ammonia.

3. Reaction time base runs should be made at a fixed temperature ($420^{\circ}C$) to determine the effect of reaction time in the two different catalysts system.

4. A series of runs should be made using other types of coal such as subbituminous coal or Texas lignite in the presence of ammonia.

Appendix A

COMPUTER PROGRAM FOR RUN CALCULATIONS

```
10 DIM L$129,A$24(13),B$37(13),D$22(15),E$34(15),K$(13)
20 DIM M(12,9),N(12,8),O(12,8),P(12,9)
30 DIM F$(12),C$(12)
40 PRINT " "
50 PRINT "    1. Input Data for a Run"
60 PRINT "    3. Calculate Material Balance for a Run"
70 PRINT "    7. Finished"
80 INPUT A5
90 IF A5=7 THEN GOTO 2300
100 IF A5=3 THEN GOTO 1260
110 IF A5¼½1 THEN GOTO 40
120 A$(1)="        Run No."
130 A$(2)=" DATE ( __ - __ - __ )"
140 A$(3)="    BOMBS PER RUN"
150 A$(4)="   REACTION TEMP."
160 A$(5)="   RUN TIME (MIN)"
170 A$(6)="  ·            COAL"
180 A$(7)="         SOLVENT"
190 A$(8)="        AR % ASH"
200 A$(9)="        AR % H2O"
210 A$(10)="       WT. COAL"
220 A$(11)="     WT. SOLVENT"
230 A$(12)="    WT. ADDED H2O"
240 A$(13)="COMMENTS ( TO ? ½½ )"
250 D$(1)="        BOMB ID"
260 D$(2)="   WT. SLURRY IN"
270 D$(3)="   CATALYST USED"
280 D$(4)="WT. CATALYST IN"
290 D$(5)=" GAS IN, PRESS."
300 D$(6)="  GAS IN, TEMP."
310 D$(7)="    GAS IN, %H2"
320 D$(8)="    NH3, PRESS."
330 D$(9)="AMBIENT, PRESS."
340 D$(10)="     LIQUID OUT"
350 D$(11)="   % DIST. RES."
360 D$(12)="   % THF INSOL."
370 D$(13)="         % H2O"
380 D$(14)="    WT. GAS OUT"
390 D$(15)="COMMENTS (TO ? ½)"
400 PRINT " * RUN DATA *"
410 X=1
420 FOR I=1 TO 13
430 PRINT "          .       ";I;A$(I);
440 INPUT B$(I)
450 IF B$(I)="ERR" THEN GOTO 1140
460 NEXT I
470 X=2
480 FOR I=1 TO 13
490 PRINT I;A$(I);'  -   ';B$(I)
500 NEXT I
510 GOTO 1100
520 OPEN FILE 'TBDATA', ALL HOLD
530 L$=" "
540 STR(L$,91,1)="$"
```

```
550 STR(L$,129,1)="*"
560 STR(L$,1,4)=B$(1)
570 STR(L$,6,8)=B$(2)
580 STR(L$,15,2)=B$(3)
590 STR(L$,18,4)=B$(4)
600 STR(L$,23,15)=B$(6)
610 STR(L$,39,15)=B$(7)
620 STR(L$,55,5)=B$(8)
630 STR(L$,61,5)=B$(9)
640 STR(L$,67,6)=B$(10)
650 STR(L$,74,6)=B$(11)
660 STR(L$,81,5)=B$(12)
670 STR(L$,87,3)=B$(5)
680 STR(L$,92,37)=B$(13)
690 WRITE FILE 'TBDATA',L$
700 PRINT " * BOMB DATA *"
710 PRINT " "
720 B1=NUM(B$(3))
730 FOR J=1 TO B1
740 X=3
750 PRINT " "
760 PRINT " "
770 FOR I=1 TO 15
780 PRINT "              ";I;D$(I);
790 INPUT E$(I)
800 IF E$(I)="ERR" THEN GOTO 1140
810 NEXT I
820 X=4
830 FOR I=1 TO 15
840 PRINT I;D$(I);'  -  ';E$(I)
850 NEXT I
860 GOTO 1100
870 L$=" "
880 STR(L$,94,1)="$"
890 STR(L$,129,1)="*"
900 STR(L$,1,1)=E$(1)
910 STR(L$,2,3)=STR(B$(1),2,3)
920 STR(L$,6,5)=E$(2)
930 STR(L$,12,20)=E$(3)
940 STR(L$,33,5)=E$(4)
950 STR(L$,39,4)=E$(5)
960 STR(L$,44,3)=E$(6)
970 STR(L$,48,5)=E$(7)
980 STR(L$,54,3)=E$(8)
990 STR(L$,58,5)=E$(9)
1000 STR(L$,64,5)=E$(10)
1010 STR(L$,70,5)=E$(11)
1020 STR(L$,76,5)=E$(12)
1030 STR(L$,82,5)=E$(13)
1040 STR(L$,88,5)=E$(14)
1050 STR(L$,95,34)=E$(15)
1060 WRITE FILE 'TBDATA',L$
1070 NEXT J
1080 CLOSE FILE 'TBDATA'
```

```
1090 GOTO 2300
1100 PRINT "ARE THERE ANY ERRORS";
1110 INPUT A$
1120 IF A$="2" .AND. X=2 THEN GOTO 520
1130 IF A$="2" .AND. X=4 THEN GOTO 870
1140 PRINT "WHICH LINE";
1150 INPUT K
1160 PRINT "THE CORRECT INFO IS";
1170 IF X<3 THEN INPUT B$(K)
1180 IF X<2 THEN INPUT E$(K)
1190 PRINT "ANY MORE CORRECTIONS";
1200 INPUT A$
1210 IF A$="1" THEN GOTO 1140
1220 IF X=1 THEN GOTO 430
1230 IF X=2 THEN GOTO 480
1240 IF X=3 THEN GOTO 780
1250 IF X=4 THEN GOTO 830
1260 PRINT "WHICH RUN DO YOU WANT DATA FOR";
1270 INPUT R$
1280 R$=STR(R$,2,3)
1290 OPEN FILE 'TBDATA',ALL HOLD
1300 READ FILE 'TBDATA',L$,EOF 2030
1310 IF STR(L$,2,3)<>R$ THEN GOTO 1300
1320 IF STR(L$,1,1)<>'T' THEN GOTO 1470
1330 Q$=STR(L$,1,4)
1340 D$=STR(L$,6,8)
1350 B1=NUM(STR(L$,15,2))
1360 T4=NUM(STR(L$,18,4))
1370 I$=STR(L$,23,15)
1380 S$=STR(L$,39,15)
1390 A1=NUM(STR(L$,55,5))
1400 H2=NUM(STR(L$,61,5))
1410 I1=NUM(STR(L$,67,6))
1420 S1=NUM(STR(L$,74,6))
1430 H3=NUM(STR(L$,81,5))
1440 V$=STR(L$,87,3)
1450 U$=STR(L$,92,37)
1460 GOTO 1300
1470 FOR I=1 TO B1
1480 F$(I)=STR(L$,1,1)
1490 S2=NUM(STR(L$,6,5))
1500 C$(I)=STR(L$,12,20)
1510 C1=NUM(STR(L$,33,5))
1520 P1=NUM(STR(L$,39,4))
1530 T2=NUM(STR(L$,44,3))
1540 H1=NUM(STR(L$,48,5))
1550 P2=NUM(STR(L$,54,3))
1560 P3=NUM(STR(L$,58,5))
1570 L1=NUM(STR(L$,64,5))
1580 D1=NUM(STR(L$,70,5))
1590 I4=NUM(STR(L$,76,5))
1600 H5=NUM(STR(L$,82,5))
1610 G1=NUM(STR(L$,88,5))
1620 T$=STR(L$,95,34)
```

```
1630 REM   --=¼½=--    MATERIAL BALANCE CALCULATIONS
     --=¼½=--
1640 H1=H1/100
1650 D1=D1/100
1660 I4=I4/100
1670 H5=H5/100
1680 IF I¼½1 THEN GOTO 1710
1690 A1=A1/100
1700 H2=H2/100
1710
M(I,1)=(P1/(54.83*(273+T2)))*(((1-H1)*28.01)+(H1*2.016))
1720 M(I,2)=(P2*17.03)/(54.83*(273+T2))
1730 M(I,3)=((S2*I1)/(S1+I1+H3))*(1-H2-A1)
1740 M(I,4)=S2*((I1*A1)/(I1+S1+H3))
1750 M(I,5)=S2*(((H2*I1)+H3)/(S1+I1+H3))
1760 M(I,9)=(S2*S1)/(S1+I1+H3)
1770 M(I,6)=M(I,9)*.8218
1780 M(I,7)=M(I,9)*.1782
1790 M(I,8)=M(I,1)+M(I,2)+S2+C1
1800 N(I,1)=G1*3.000
1810 N(I,2)=M(I,2)
1820 N(I,3)=L1*I4-M(I,4)-C1
1830 N(I,4)=M(I,4)
1840 N(I,5)=L1*H5
1850 N(I,6)=(L1*(1-D1))-N(I,5)
1860 N(I,7)=L1*(D1-I4)
1870 N(I,8)=L1+N(I,1)+N(I,2)+C1
1880 O(I,7)=100-((M(I,8)-N(I,8))/M(I,8))*100
1890 FOR K=1 TO 8
1900 P(I,K)=N(I,K)/((O(I,7)/100))
1910 NEXT K
1920 FOR K=1 TO 8
1930 P(I,9)=P(I,9)+P(I,K)
1940 NEXT K
1950 O(I,1)=((P(I,1)+P(I,2)-M(I,1)-M(I,2))/M(I,3))*100
1960 O(I,2)=((M(I,3)-P(I,3))/M(I,3))*100
1970 O(I,3)=((P(I,6)-M(I,6))/M(I,3))*100
1980 O(I,4)=((P(I,7)-M(I,7))/M(I,3))*100
1990 O(I,5)=((P(I,6)-M(I,6))/M(I,6))*100
2000 IF I=B1 THEN GOTO 2020
2010 READ FILE 'TBDATA',L$,EOF 2030
2020 NEXT I
2030 CLOSE FILE 'TBDATA'
2040 FORM POS31,C,POS70,C,SKIP1
2050 FORM POS22,C,POS28,C,POS35,C,POS43,C,POS50,C,POS56,
     C,POS62,C,POS70,C,POS77,C,POS84,C,POS91,C,POS99,C,SKIP1
2060 FORM POS91,C,POS99,C,SKIP1
2070 PRINT,TAB(5),'            DATE    - ';D$
2080 PRINT,TAB(5),'            COAL    - ';I$
2090 PRINT,TAB(5),'          SOLVENT   - ';S$
2100 PRINT,TAB(5),'Temperature, C   - ';T4
2110 PRINT,TAB(5),' RUN TIME, MIN   - ';V$
2120 PRINT,TAB(5),'GAS COMP., %H2   - ';H1*100
```

```
2130                                                          PRINT
'                                                            ';
2140                                                          PRINT
'                                                        '
2150    PRINTUSING    2040,'GRAMS','YIELD   (WT%   OF   MAF   COAL
CHARGED)'
2160 PRINT " "
2170 PRINTUSING 2060,'CONVE','CLO-'
2180 PRINTUSING 2050,'GAS','DIST','S RES','H2O','ASH',
     'IOM','TOTAL','GAS','DIST','S RES','RSION','SURE'
2190 PRINT " "
2200 FORM C,POS20,PIC(ZZ.##),POS27,PIC(ZZ.##),
     POS34,PIC(ZZ.##),POS41,PIC(ZZ.##),POS48,PIC(ZZ.##),
     POS55,PIC(ZZ.##),POS62,PIC(ZZ.##),SKIP1
2210 FORM C,POS20,PIC(ZZ.##),POS27,PIC(ZZ.##),
     POS34,PIC(ZZ.##),POS41,PIC(ZZ.##),POS48,PIC(ZZ.##),
     POS55,PIC(ZZ.##),POS62,PIC(ZZ.##),POS69,PIC(ZZZ.##),
     POS76,PIC(ZZZ.##),POS83,PIC(ZZZ.##),POS90,PIC(ZZZ.##),
     POS97,PIC(ZZZZ.##),SKIP1
2220 FOR I=1 TO B1
2230 PRINT 'BOMB ';F$(I);'   CATALYST - ';C$(I)
2240 PRINTUSING 2220,'        GRAMS IN',
     M(I,1),M(I,6),M(I,7),M(I,5),M(I,4),M(I,3),M(I,8)
2250 PRINTUSING 2230,'        GRAMS OUT',
     N(I,1),N(I,6),N(I,7),N(I,5),N(I,4),N(I,3),
     N(I,8),O(I,1),O(I,3),O(I,4),O(I,2),O(I,7)
2260 PRINTUSING 2220,'        GRAMS NORM',
     P(I,1),P(I,6),P(I,7),P(I,5),P(I,4),P(I,3),P(I,8)
2270 NEXT I
2280 END
```

Appendix B

OUTPUT FROM COMPUTER PROGRAM

Run No   -   T001

Date   -   5-23-83

Coal   -   ZAP

Solvent   -   A04

Temperature, C   -   420

Run Time, Min   -   20

Bomb A   Catalyst - NONE

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.71	2.47	.53	.76	.20	1.51	7.18
Grams OUT	1.29	2.59	.54	.73	.20	.76	6.29
Grams NORM	1.47	3.16	.62	.83	.22	.87	7.18

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	-15.62	46.14	5.51	42.57	87.62

Bomb B   Catalyst - NONE

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.71	2.47	.53	.76	.20	1.51	7.18
Grams OUT	1.35	2.83	.63	.75	.20	.75	6.50
Grams NORM	1.49	3.12	.69	.83	.22	.83	7.18

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	-14.38	43.35	10.45	45.15	94.55

Bomb C   Catalyst - H2S

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.50	2.47	.53	.76	.20	1.51	7.04

```
Grams OUT    1.71  2.52   .57   .48   .20   .61   6.16
Grams NORM   1.95  2.87   .66   .55   .23   .70   7.04
```

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conve rsion	Clo- sure
Grams OUT	30.68	27.06	8.07	53.95	87.50

Bomb D   Catalyst - H2S

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.53	2.52	.55	.78	.20	1.55	7.18
Grams OUT	1.74	2.51	.59	.50	.20	.66	6.26
Grams NORM	1.99	2.87	.67	.57	.23	.76	7.18

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conve rsion	Clo- sure
Grams. OUT	30.89	22.77	8.31	50.79	87.24

Bomb E   Catalyst - NONE

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.47	.53	.76	.20	1.51	5.59
Grams OUT	.33	2.52	.49	.72	.20	1.04	5.29
Grams NORM	.35	2.66	.52	.76	.21	1.10	5.59

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conve rsion	Clo- sure
Grams OUT	14.94	12.95	- 1.23	27.44	94.59

Bomb F   Catalyst - NONE

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.47	.53	.76	.20	1.51	5.59
Grams OUT	.33	2.59	.51	.73	.20	1.08	5.43
Grams NORM	.34	2.66	.53	.75	.20	1.11	5.59

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conve rsion	Clo- sure
Grams OUT	14.34	13.18	- .44	26.63	97.09

Bomb G   Catalyst - H2S

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.11	2.47	.53	.76	.20	1.51	5.69
Grams OUT	.42	2.67	.83	.76	.20	.77	5.76
Grams NORM	.42	2.64	.82	.75	.19	.76	5.69

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conve rsion	Clo- sure
Grams OUT	19.97	11.64	18.84	49.69	101.20

Bomb H   Catalyst - H2S

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.11	2.47	.53	.76	.20	1.51	5.71
Grams OUT	.45	2.57	.68	.76	.20	.66	5.45
Grams NORM	.47	2.69	.71	.80	.21	.70	5.71

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conve rsion	Clo- sure
Grams OUT	24.41	15.08	11.53	54.01	95.48

Bomb I   Catalyst - NONE

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.91	2.47	.53	.76	.20	1.51	6.38
Grams OUT	.99	2.67	.65	.49	.20	.82	5.81
Grams NORM	1.09	2.93	.71	.54	.22	.90	6.38

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	11.44	30.83	11.54	40.59	91.00

Bomb J   Catalyst - NONE

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.91	2.47	.53	.76	.20	1.51	6.38
Grams OUT	.96	2.75	.62	.52	.20	.89	5.93
Grams NORM	1.03	2.96	.67	.56	.21	.95	6.38

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	7.85	32.56	8.70	37.00	94.88

Bomb K   Catalyst - H2S

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.83	2.47	.53	.76	.20	1.51	6.41
Grams OUT	.87	2.32	.68	.76	.20	.98	5.92
Grams NORM	.94	2.51	.73	.82	.21	1.06	6.41

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	8.19	3.02	13.21	29.62	92.41

Bomb L   Catalyst - H2S

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.84	2.47	.53	.76	.20	1.51	6.41
Grams OUT	.81	2.63	.65	.76	.20	.83	5.97
Grams NORM	.87	2.82	.69	.82	.21	.89	6.41

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	2.63	23.56	10.57	41.38	93.18

Run No   -   T002

Date   -   05-27-83

Coal   -   ZAP

Solvent   -   A04

Temperature, C   -   420

Run Time, Min   -   20

Bomb A   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.37	2.47	.53	.76	.20	1.51	6.96
Grams OUT	1.05	3.26	.79	.77	.20	.89	7.06
GRAMS NORM	1.03	3.21	.78	.75	.19	.88	6.96

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
GRAMS OUT	-22.62	48.99	16.33	42.12	101.51

Bomb B   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.37	2.47	.53	.76	.20	1.51	6.96
Grams OUT	.96	3.28	.76	.74	.20	.88	6.93
Grams NORM	.96	3.30	.76	.76	.20	.88	6.96

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	-27.17	54.88	14.85	41.75	99.64

Bomb C   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.73	2.43	.53	.75	.19	1.49	7.20

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Grams OUT      1.77  2.89  .64   .63   .19   .70  6.98
Grams NORM     1.83  2.98  .66   .65   .20   .72  7.20
```

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	7.58	33.79	11.82	53.17	95.95

Bomb D   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.73	2.43	.53	.75	.19	1.49	7.20
Grams OUT	1.77	2.81	.67	.63	.19	.67	6.91
Grams NORM	1.84	2.93	.70	.65	.20	.70	7.20

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	7.58	33.79	11.82	53.17	95.95

Bomb E . Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.10	2.47	.53	.76	.20	1.51	5.69
Grams OUT	.12	3.18	.49	.70	.20	.96	5.77
Grams NORM	.12	3.13	.48	.69	.19	.95	5.69

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	1.22	44.19	-	3.48	37.29	101.44

Bomb F   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.10	2.47	.53	.76	.20	1.51	5.69
Grams OUT	.12	3.31	.42	.71	.20	.96	5.84
Grams NORM	.12	3.22	.41	.69	.19	.94	5.69

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	1.03	50.03	- 8.26	38.07	102.67

Bomb G   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.43	.53	.75	.19	1.49	5.59
Grams OUT	.33	2.63	.93	.59	.19	.68	5.51
Grams NORM	.34	2.67	.94	.60	.20	.69	5.59

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	14.11	16.14	27.74	53.52	98.49

Bomb H   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.43	.53	.75	.19	1.49	5.59
Grams OUT	.36	2.55	.85	.51	.19	.64	5.26
Grams NORM	.38	2.71	.90	.54	.21	.68	5.59

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	17.32	19.13	25.19	54.37	94.02

Bomb I   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.75	2.47	.53	.76	.20	1.51	6.34
Grams OUT	.60	3.22	.67	.63	.20	.82	6.26
Grams NORM	.61	3.26	.67	.64	.20	.83	6.34

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	- 9.44	52.81	9.26	44.94	98.70

Bomb J   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.75	2.47	.53	.76	.20	1.51	6.34
Grams OUT	.60	3.23	.71	.74	.20	.84	6.43
Grams NORM	.59	3.18	.70	.73	.19	.82	6.34

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	-10.71	47.44	10.94	45.55	101.38

Bomb K   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.96	2.43	.53	.75	.19	1.49	6.43
Grams OUT	1.05	2.75	.50	.61	.19	.75	6.01
Grams NORM	1.12	2.94	.53	.65	.21	.81	6.43

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	11.12	34.16	.27	45.99	93.51

Bomb L   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.96	2.43	.53	.75	.19	1.49	6.43
Grams OUT	1.05	2.86	.56	.61	.19	.75	6.19
Grams NORM	1.09	2.97	.58	.64	.20	.78	6.43

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	8.93	36.09	3.85	47.53	96.31

Run No   –   T003

Date   –   06-02-83

Coal   –   ZAP

Solvent   –   A04

Temperature, C   –   440

Run Time, Min   –   20

Bomb A   Catalyst – Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.35	2.47	.53	.65	.19	1.50	6.81
Grams OUT	1.29	3.26	.61	.37	.19	.90	6.74
Grams NORM	1.30	3.30	.62	.38	.19	.91	6.81

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	– 3.16	55.26	5.44	39.43	98.93

Bomb B   Catalyst – Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.35	2.47	.53	.65	.19	1.50	6.81
Grams OUT	1.23	3.18	.56	.35	.19	.85	6.48
Grams NORM	1.29	3.34	.59	.37	.20	.89	6.81

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	– 3.47	58.43	3.93	40.84	95.14

Bomb C   Catalyst – Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.71	2.43	.53	.64	.18	1.48	7.05

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams OUT	1.74	2.72	.54	.63	.18	.73	6.70
Grams NORM	1.83	2.86	.57	.66	.19	.77	7.05

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	7.96	29.43	2.68	47.94	94.98

Bomb D   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.71	2.43	.53	.64	.18	1.48	7.05
Grams OUT	1.77	2.69	.54	.63	.18	.73	6.70
Grams NORM	1.88	2.86	.57	.66	.19	.77	7.05

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	11.42	28.98	2.12	49.51	93.98

Bomb E . Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.10	2.47	.53	.65	.19	1.50	5.55
Grams OUT	.18	2.87	.60	.84	.19	.80	5.59
Grams NORM	.18	2.85	.60	.83	.19	.80	5.55

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	5.35	25.52	4.08	46.86	100.77

Bomb F   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.10	2.47	.53	.65	.19	1.50	5.55
Grams OUT	.15	2.92	.53	.86	.19	.83	5.59
Grams NORM	.15	2.90	.53	.86	.19	.82	5.55

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	3.36	28.63	- .27	45.32	100.77

Bomb G   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.43	.53	.64	.18	1.48	5.46
Grams OUT	.36	2.75	.76	.70	.18	.59	5.50
Grams NORM	.36	2.73	.75	.70	.18	.58	5.46

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	15.83	20.30	15.43	60.51	100.67

Bomb H   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.43	.53	.64	.18	1.48	5.46
Grams OUT	.39	2.73	.78	.57	.18	.54	5.35
Grams NORM	.40	2.79	.79	.58	.19	.55	5.46

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	18.58	24.28	17.93	62.61	97.92

Bomb I   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.74	2.47	.53	.65	.19	1.50	6.20
Grams OUT	.54	3.02	.75	.72	.19	.90	6.27
Grams NORM	.53	3.02	.74	.71	.18	.89	6.20

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	-14.05	36.65	14.00	40.57	101.06

Bomb J   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.74	2.47	.53	.65	.19	1.50	6.20
Grams OUT	.51	2.98	.69	.77	.19	.89	6.15
Grams NORM	.51	3.01	.70	.78	.19	.90	6.20

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	-15.22	35.99	10.94	40.25	99.13

Bomb K   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.94	2.43	.53	.64	.18	1.48	6.28
Grams OUT	1.08	2.49	.64	.70	.18	.71	5.96
Grams NORM	1.14	2.62	.67	.73	.19	.75	6.28

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	13.20	13.21	9.86	49.06	94.85

Bomb L   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.94	2.43	.53	.64	.18	1.48	6.28
Grams OUT	1.02	2.48	.67	.66	.18	.71	5.88
Grams NORM	1.09	2.65	.71	.70	.20	.76	6.28

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	9.86	14.83	12.68	48.38	93.57

Run No   -   T004

Date   -   06-07-83

Coal   -   ZAP

Solvent   -   AO4

Temperature, C -   400

Run Time, Min -   20

Bomb A   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.36	2.48	.54	.58	.18	1.49	6.74
Grams OUT	1.26	2.94	.57	.64	.18	.92	6.63
Grams NORM	1.28	2.99	.58	.65	.18	.94	6.74

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	-	4.89	34.16	2.86 36.82	98.26

Bomb B   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.36	2.48	.54	.58	.18	1.49	6.74
Grams OUT	1.29	3.04	.59	.66	.18	.88	6.76
Grams NORM	1.29	3.04	.58	.66	.18	.88	6.74

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	-	4.69	37.06	3.09 40.93	100.19

Bomb C   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.72	2.45	.53	.57	.18	1.47	6.99

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams OUT	1.71	2.81	.73	.62	.18	.65	6.86
Grams NORM	1.74	2.86	.75	.63	.18	.66	6.99

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	1.62	28.56	14.87	54.67	98.16

Bomb D   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.72	2.45	.53	.57	.18	1.47	6.99
Grams OUT	1.74	2.73	.72	.58	.18	.64	6.74
Grams NORM	1.80	2.83	.74	.60	.18	.66	6.99

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	5.77	26.41	14.50	54.78	96.42

Bomb E . Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.10	2.48	.54	.58	.18	1.49	5.48
Grams OUT	.12	3.10	.40	.62	.18	.92	5.46
Grams NORM	.12	3.11	.40	.63	.18	.92	5.48

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	1.59	42.27	- 9.09	37.99	99.50

Bomb F   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.10	2.48	.54	.58	.18	1.49	5.48
Grams OUT	.12	3.26	.47	.60	.18	.88	5.64
Grams NORM	.12	3.18	.46	.59	.17	.86	5.48

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	1.08	46.53 -	5.27	42.51	102.78

Bomb G   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.45	.53	.57	.18	1.47	5.39
Grams OUT	.36	2.71	.63	.64	.18	.74	5.42
Grams NORM	.36	2.70	.62	.64	.18	.74	5.39

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	16.02	17.39	6.27	49.54	100.49

Bomb H   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.45	.53	.57	.18	1.47	5.39
Grams OUT	.39	2.67	.64	.62	.18	.71	5.37
Grams NORM	.39	2.68	.64	.62	.18	.72	5.39

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	18.29	16.13	7.60	51.05	99.56

Bomb I   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.75	2.48	.54	.58	.18	1.49	6.14
Grams OUT	.66	2.96	.55	.68	.18	.90	6.05
Grams NORM	.67	3.01	.56	.69	.18	.92	6.14

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	- 5.33	35.16	1.15	38.45	98.52

Bomb J   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.75	2.48	.54	.58	.18	1.49	6.14
Grams OUT	.69	3.05	.53	.68	.18	.89	6.13
Grams NORM	.69	3.06	.53	.58	.18	.89	6.14

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	- 3.95	38.47	- .78	40.37	99.84

Bomb K   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.95	2.45	.53	.57	.18	1.47	6.22
Grams OUT	1.05	2.61	.60	.60	.18	.68	5.88
Grams NORM	1.11	2.77	.64	.64	.19	.72	6.22

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	10.93	21.80	7.28	51.09	94.52

Bomb L   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.95	2.45	.53	.57	.18	1.47	6.22
Grams OUT	1.08	2.64	.59	.58	.18	.65	5.87
Grams NORM	1.14	2.79	.62	.61	.19	.69	6.22

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	13.23	23.73	6.41	53.11	94.36

Run No   –   T005

Date   –   10-28-83

Coal   –   ZAP

Solvent   –   AO4

Temperature, C   –   400

Run Time, Min   –   20

Bomb A   Catalyst – NONE

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.71	2.47	.53	.58	.18	1.54	7.01
Grams OUT	1.62	2.62	.83	.62	.18	.71	6.58
Grams NORM	1.73	2.79	.88	.66	.20	.76	7.01

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	.82	21.01	22.69	50.68	94.81

Bomb B   Catalyst – H2S

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.71	2.47	.53	.58	.18	1.54	7.22
Grams OUT	1.56	2.55	.82	.58	.18	.71	6.62
Grams NORM	1.70	2.79	.89	.63	.20	.78	7.22

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	.48	20.94	23.34	49.30	91.64

Bomb C   Catalyst – Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.71	2.42	.52	.57	.18	1.51	7.02

	Grams						
Grams OUT	1.29	3.11	.95	.76	.18	.75	7.15
Grams NORM	1.27	3.05	.93	.75	.18	.74	7.02

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	-29.80	42.11	26.83	50.95	101.79

Bomb D   Catalyst - Quinoline

	Grams						
	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	1.71	2.47	.53	.58	.18	1.54	7.09
Grams OUT	1.65	2.67	.88	.63	.18	.63	6.81
Grams NORM	1.72	2.79	.91	.66	.19	.66	7.09

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	.30	20.87	24.67	56.97	95.99

Bomb E   Catalyst - NONE

	Grams						
	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.47	.53	.58	.18	1.54	5.42
Grams OUT	.33	2.73	.74	.65	.18	.86	5.49
Grams NORM	.33	2.70	.73	.64	.18	.85	5.42

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	13.20	15.22	12.49	44.61	101.23

Bomb F   Catalyst - H2S

	Grams						
	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.47	.53	.58	.18	1.54	5.63
Grams OUT	.36	3.09	.74	.58	.18	.75	5.91
Grams NORM	.34	2.94	.71	.55	.17	.71	5.63

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	13.67	30.97	11.36	53.67	104.91

Bomb G   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.47	.53	.58	.18	1.54	5.53
Grams OUT	.12	3.46	.51	.74	.18	.95	6.06
Grams NORM	.11	3.16	.46	.68	.17	.86	5.53

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	- 1.52	44.93	- 4.79	43.73	109.71

Bomb H   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.12	2.47	.53	.58	.18	1.54	5.50
Grams OUT	.30	2.63	.71	.70	.18	.71	5.39
Grams NORM	.31	2.69	.72	.71	.19	.72	5.50

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	11.92	14.54	12.29	53.05	97.94

Bomb I   Catalyst - NONE

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.95	2.47	.53	.58	.18	1.54	6.25
Grams OUT	.96	2.67	.85	.66	.18	.77	6.10
Grams NORM	.98	2.82	.87	.68	.19	.79	6.25

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	2.33	17.82	21.93	48.53	97.64

Bomb J   Catalyst - H2S

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.54	2.47	.53	.58	.18	1.54	5.96
Grams OUT	1.08	2.41	.90	.58	.18	.70	5.97
Grams NORM	1.08	2.40	.90	.58	.18	.70	5.96

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	35.10-	4.02	23.73	54.72	100.19

Bomb K   Catalyst - Ammonia

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.95	2.47	.53	.58	.18	1.54	6.35
Grams OUT	.69	2.99	1.11	.74	.18	.92	6.72
Grams NORM	.65	2.82	1.04	.70	.17	.87	6.35

Yield (Wt% of maf coal charged)

	Gas	Dist	S RES	Conversion	Closure
GRAMS OUT	-19.69	23.22	33.23	43.57	105.85

Bomb L   Catalyst - Quinoline

Grams

	Gas	Dist	S Res	H2O	Ash	IOM	Total
Grams IN	.95	2.47	.53	.58	.18	1.54	6.33
GRAMS OUT	1.02	2.86	.63	.75	.18	.72	6.32
GRAMS NORM	1.02	2.86	.63	.75	.18	.72	6.33

Yield (Wt% of maf coal charged)

	Gas	Dist	S Res	Conversion	Closure
Grams OUT	4.80	25.65	6.44	53.19	99.88

Appendix C

SAMPLE YIELD CALCULATIONS (RUN T002-A)

C.1   NOTATION USED IN COMPUTER PROGRAMS

T2   Gas in Temperature

P1   Gas in pressure

P2 NH_3 in pressure

S1   Weight of solvent

S2   Weight of slurry in

I1   Weight of coal

H1   Gas in % H_2

H2   % H_2O in

H5   % H_2O out

A1   % ash

G1   Weight % gas out

C1   Weight of catalyst in

L1   Slurry out

I4   % of THF insoluble

D1   % of dist residue

M(I)   Weight of in

N(I)   Weight of out

R(I)   Weight of Normalize

O1   Gas yield

O2   Conversion

O3   Dist

O4   Soluble residue

O5   Closure

C.2   CALCULATIONS

1. Mass of reducing gas charge in tubing bomb reactor

   $M1=(P1/54.83*(273+T2))*(((1-H1)*28.01)+(H1*2.016))$

   $M1=(794/54.83*(273+22))*(((1-0)*28.01)+(0*2.016))$

   $M1=1.43$ grams

2. Mass of ammonia gas charge

   $M2=(P2*17.03)/(54.83*(273+T2))$

   $M2=(106*17.03)/(54.83*(273+22))$

   $M2=0.11$ grams

3. IOM in

   $M3=I1*(1-H2-A1)$

   $M3=2.47*(1-0.3076-0.0798)$

   $M3=1.51$ grams

4. Mass of ash in

   $M4=I1*A1$

   $M4=2.47*0.0798$

   $M4=0.20$ grams

5. Mass of H_2O in

   $M5=I1*H2$

   $M5=2.47*0.3076$

   $M5=0.76$ grams

6. Mass of distillate in

   $M6=S1*0.8218$

   $M6=3.0*0.8218$

   $M6=2.47$ grams

7. Mass of soluble residue

   $M7=S1*0.1782$

M7=3.0*0.1782

M7=0.5346 grams

8. Mass of total in

M8=M1+M2+S2+C1

M8=1.37+0.11+5.47+0

M8=6.95 grams

9. Mass of product gas

N1=G1*(V1+V2)/V2

N1=G1*(22.26+11.21)/11.21

N1=G1*3.00

N1=0.35*3.00

N1=1.05 grams

10. Mass of catalyst

N2=M2

N2=0.11 grams

11. IOM out

N3=L1*I4-M4

N3=5.90*0.1841-0.20

N3=0.89 grams

12. Ash out = Ash in

N4=M4

N4=0.20 grams

13. H_2O out = H_2O in

N5=L1*H5

N5=0.77 grams

14. Mass of distillate out

N6=L1*(1-D1)-N5

N6=5.90*(1-0.3186)-0.76

N6=3.26 grams

15. Mass of soluble residue out

N7=L1*(D1-I4)

N7=5.90*(0.3186-0.1841)

N7=0.79 gram

16. Mass of total out

N8=L1+N1+N2

N8=5.90+1.05+0.11

N8=7.06 grams

17. Percent closure

O5=100-((M8-N8)/M8*100)

O5=100-((6.96-7.06)/6.96*100)

O5=101.51

18. Normalization procedure

R(I)=N(I)/(O5/100)

R1=1.05/(101.51/100)=1.03 grams

R2=0.11/1.0151=0.11 gram

R3=0.89/1.0151=0.88 gram

R4=0.20/1.0151=0.19 gram

R5=0.76/1.0151=0.75 gram

R6=3.26/1.0151=3.22 grams

R7=0.79/1.0151=0.78 gram

R8=6.96 grams

19. Net gas yield (Weight % of MAF lignite)

O1=((R1+R2-M1-M2)/M3)*100

O1=((1.03+0.11-1.37-0.11).1.51)*100=-22.62%

20. Net distillate yield (Weight % of MAF lignite)

$O3 = ((R6-M6)/M3)*100$

$O3 = ((3.22-2.47)/1.51)*100 = 48.99\%$

21. Net soluble residue yield (Weight % of MAF lignite)

$O4 = ((R7-M7)/M3)*100$

$O4 = ((0.78-0.5346)/1.51)*100 = 16.33\%$

22. Conversion (Weight % of MAF lignite)

$O2 = ((M3-R3)/M3)*100$

$O2 = ((1.51-0.88)/1.51)*100 = 42.12\%$

Appendix D

TEST CONDITIONS OF ALL RUNS

TABLE 9

CONDITIONS OF RUN T001

Bomb Number	Catalyst	Temperature	Reducing Gas	Lignite Moisture	Lignite Ash	Initial Pressure	Catalyst Pressure
A	None	$420^{\circ}C$	CO	30.76	7.98	1000 psi	0 psi
B	None	$420^{\circ}C$	CO	30.76	7.98	1000 psi	0 psi
C	H_2S	$420^{\circ}C$	CO	30.76	7.98	878 psi	67 psi
D	H_2S	$420^{\circ}C$	CO	30.76	7.98	893 psi	59 psi
E	None	$420^{\circ}C$	H_2	30.76	7.98	999 psi	0 psi
F	None	$420^{\circ}C$	H_2	30.76	7.98	999 psi	0 psi
G	H_2S	$420^{\circ}C$	H_2	30.76	7.98	908 psi	51 psi
H	H_2S	$420^{\circ}C$	H_2	30.76	7.98	881 psi	64 psi
I	None	$420^{\circ}C$	H_2/CO	30.76	7.98	999 psi	0 psi
J	None	$420^{\circ}C$	H_2/CO	30.76	7.98	999 psi	0 psi
K	H_2S	$420^{\circ}C$	H_2/CO	30.76	7.98	903 psi	55 psi
L	H_2S	$420^{\circ}C$	H_2/CO	30.76	7.98	914 psi	49 psi

TABLE 10

CONDITIONS OF RUN T002

Bomb Number	Catalyst	Temperature	Reducing Gas	Lignite Moisture	Lignite Ash	Initial Pressure	Catalyst Pressure
A	NH_3	$420^\circ C$	CO	30.76	7.98	794 psi	106 psi
B	NH_3	$420^\circ C$	CO	30.76	7.98	794 psi	106 psi
C	C_9H_7N	$420^\circ C$	CO	30.76	7.98	1000 psi	0 psi
D	C_9H_7N	$420^\circ C$	CO	30.76	7.98	1000 psi	0 psi
E	NH_3	$420^\circ C$	H_2	30.76	7.98	788 psi	112 psi
F	NH_3	$420^\circ C$	H_2	30.76	7.98	788 psi	112 psi
G	C_9H_7N	$420^\circ C$	H_2	30.76	7.98	1000 psi	0 psi
H	C_9H_7N	$420^\circ C$	H_2	30.76	7.98	1000 psi	0 psi
I	NH_3	$420^\circ C$	H_2/CO	30.76	7.98	786 psi	114 psi
J	NH_3	$420^\circ C$	H_2/CO	30.76	7.98	786 psi	114 psi
K	C_9H_7N	$420^\circ C$	H_2/CO	30.76	7.98	1000 psi	0 psi
L	C_9H_7N	$420^\circ C$	H_2/CO	30.76	7.98	1000 psi	0 psi

TABLE 11

CONDITIONS OF RUN T003

Bomb Number	Catalyst	Temperature	Reducing Gas	Lignite Moisture	Lignite Ash	Initial Pressure	Catalyst Pressure
A	NH_3	$440^{\circ}C$	CO	27.83	7.98	789 psi	111 psi
B	NH_3	$440^{\circ}C$	CO	27.83	7.98	789 psi	111 psi
C	C_9H_7N	$440^{\circ}C$	CO	27.83	7.98	1000 psi	0 psi
D	C_9H_7N	$440^{\circ}C$	CO	27.83	7.98	1000 psi	0 psi
E	NH_3	$440^{\circ}C$	H_2	27.83	7.98	790 psi	110 psi
F	NH_3	$440^{\circ}C$	H_2	27.83	7.98	790 psi	110 psi
G	C_9H_7N	$440^{\circ}C$	H_2	27.83	7.98	1000 psi	0 psi
H	C_9H_7N	$440^{\circ}C$	H_2	27.83	7.98	1000 psi	0 psi
I	NH_3	$440^{\circ}C$	H_2/CO	27.83	7.98	788 psi	112 psi
J	NH_3	$440^{\circ}C$	H_2/CO	27.83	7.98	788 psi	112 psi
K	C_9H_7N	$440^{\circ}C$	H_2/CO	27.83	7.98	999 psi	0 psi
L	C_9H_7N	$440^{\circ}C$	H_2/CO	27.83	7.98	1000 psi	0 psi

TABLE 12

CONDITIONS OF RUN T004

Bomb Number	Catalyst	Temperature	Reducing Gas	Lignite Moisture	Lignite Ash	Initial Pressure	Catalyst Pressure
A	NH_3	400°C	CO	25.81	7.98	789 psi	112 psi
B	NH_3	400°C	CO	25.81	7.98	789 psi	112 psi
C	C_9H_7N	400°C	CO	25.81	7.98	999 psi	0 psi
D	C_9H_7N	400°C	CO	25.81	7.98	1000 psi	0 psi
E	NH_3	400°C	H_2	25.81	7.98	788 psi	100 psi
F	NH_3	400°C	H_2	25.81	7.98	788 psi	100 psi
G	C_9H_7N	400°C	H_2	25.81	7.98	998 psi	0 psi
H	C_9H_7N	400°C	H_2	25.81	7.98	1000 psi	0 psi
I	NH_3	400°C	H_2/CO	25.81	7.98	790 psi	111 psi
J	NH_3	400°C	H_2/CO	25.81	7.98	790 psi	111 psi
K	C_9H_7N	400°C	H_2/CO	25.81	7.98	1000 psi	0 psi
L	C_9H_7N	400°C	H_2/CO	25.81	7.98	1000 psi	0 psi

TABLE 13

CONDITIONS OF RUN T005

Bomb Number	Catalyst	Temperature	Reducing Gas	Lignite Moisture	Lignite Ash	Initial Pressure	Catalyst Pressure
A	None	$400^{\circ}C$	CO	25.27	7.98	1000 psi	0 psi
B	H_2S	$400^{\circ}C$	CO	25.27	7.98	1000 psi	101 psi
C	NH_3	$400^{\circ}C$	CO	25.27	7.98	1000 psi	101 psi
D	C_9H_7N	$400^{\circ}C$	CO	25.27	7.98	1000 psi	0 psi
E	None	$400^{\circ}C$	H_2	25.27	7.98	1000 psi	0 psi
F	H_2S	$400^{\circ}C$	H_2	25.27	7.98	1000 psi	101 psi
G	NH_3	$400^{\circ}C$	H_2	25.27	7.98	1001 psi	100 psi
H	C_9H_7N	$400^{\circ}C$	H_2	25.27	7.98	1000 psi	0 psi
I	None	$400^{\circ}C$	H_2/CO	25.27	7.98	1000 psi	0 psi
J	H_2S	$400^{\circ}C$	H_2/CO	25.27	7.98	1000 psi	101 psi
K	NH_3	$400^{\circ}C$	H_2/CO	25.27	7.98	1001 psi	100 psi
L	C_9H_7N	$400^{\circ}C$	H_2/CO	25.27	7.98	1000 psi	0 psi

Appendix E

SUMMARY OF RESULTS

TABLE 14

RESULTS OF RUN T001

Bomb Number	Cata- lyst	Tempe- rature	Reducing Gas	Yields, Wt% of MAF Lignite				
				Gas	Dist	S Res	Conv	Closure
A	None	$420^{\circ}C$	CO	-15.62	46.14	5.51	42.57	87.62
B	None	$420^{\circ}C$	CO	-14.38	43.35	10.45	45.15	94.55
C	H_2S	$420^{\circ}C$	CO	30.68	27.06	8.07	53.95	87.50
D	H_2S	$420^{\circ}C$	CO	30.89	22.77	8.31	50.79	87.24
E	None	$420^{\circ}C$	H_2	14.94	12.95	-1.23	27.44	94.59
F	None	$420^{\circ}C$	H_2	14.34	13.18	-0.44	26.63	97.09
G	H_2S	$420^{\circ}C$	H_2	20.00	9.93	18.83	49.69	101.21
H	H_2S	$420^{\circ}C$	H_2	24.22	15.05	11.55	53.99	95.42
I	None	$420^{\circ}C$	H_2/CO	11.44	30.83	11.54	40.59	91.00
J	None	$420^{\circ}C$	H_2/CO	7.85	32.56	8.70	37.00	94.88
K	H_2S	$420^{\circ}C$	H_2/CO	7.93	10.70	13.25	29.57	92.34
L	H_2S	$420^{\circ}C$	H_2/CO	2.42	39.23	10.60	41.34	93.13

TABLE 15

RESULTS OF RUN T002

Bomb Number	Cata- lyst	Tempe- rature	Reducing Gas	Yields, Wt% of MAF Lignite				
				Gas	Dist	S Res	Conv	Closure
A	NH_3	$420^{\circ}C$	CO	-22.62	48.99	16.33	42.12	101.51
B	NH_3	$420^{\circ}C$	CO	-27.17	54.88	14.85	41.75	99.64
C	C_9H_7N	$420^{\circ}C$	CO	6.34	37.18	9.04	51.88	96.92
D	C_9H_7N	$420^{\circ}C$	CO	7.58	33.79	11.82	53.17	95.95
E	NH_3	$420^{\circ}C$	H_2	1.22	44.19	-3.48	37.29	101.44
F	NH_3	$420^{\circ}C$	H_2	1.03	50.03	-8.26	38.07	102.67
G	C_9H_7N	$420^{\circ}C$	H_2	14.11	16.14	27.74	53.52	98.49
H	C_9H_7N	$420^{\circ}C$	H_2	17.32	19.13	25.19	54.37	94.02
I	NH_3	$420^{\circ}C$	H_2/CO	-9.44	52.81	9.26	44.94	98.70
J	NH_3	$420^{\circ}C$	H_2/CO	-10.71	47.44	10.94	45.55	101.38
K	C_9H_7N	$420^{\circ}C$	H_2/CO	11.12	34.16	0.27	45.99	93.51
L	C_9H_7N	$420^{\circ}C$	H_2/CO	8.93	36.09	3.85	47.53	96.31

TABLE 16

RESULTS OF RUN T003

Bomb Number	Cata- lyst	Tempe- rature	Reducing Gas	Yields, Wt% of MAF Lignite				
				Gas	Dist	S Res	Conv	Closure
A	NH_3	$440^\circ C$	CO	-3.16	55.26	5.44	39.43	98.93
B	NH_3	$440^\circ C$	CO	-3.47	58.43	3.93	40.84	95.14
C	C_9H_7N	$440^\circ C$	CO	7.96	29.43	2.68	47.94	94.98
D	C_9H_7N	$440^\circ C$	CO	11.42	28.98	2.12	49.51	93.98
E	NH_3	$440^\circ C$	H_2	5.35	25.52	4.08	46.86	100.77
F	NH_3	$440^\circ C$	H_2	3.36	28.63	-0.27	45.32	100.77
G	C_9H_7N	$440^\circ C$	H_2	15.83	20.30	15.43	60.51	100.67
H	C_9H_7N	$440^\circ C$	H_2	18.58	24.28	17.93	62.61	97.92
I	NH_3	$440^\circ C$	H_2/CO	-14.05	36.65	14.00	40.57	101.06
J	NH_3	$440^\circ C$	H_2/CO	-15.22	35.99	10.94	40.25	99.13
K	C_9H_7N	$440^\circ C$	H_2/CO	13.20	13.21	9.86	49.06	94.85
L	C_9H_7N	$440^\circ C$	H_2/CO	9.86	14.83	12.68	48.38	93.57

TABLE 17

RESULTS OF RUN T004

Bomb Number	Catalyst	Temperature	Reducing Gas	Yields, Wt% of MAF Lignite				
				Gas	Dist	S Res	Conv	Closure
A	NH_3	$400^\circ C$	CO	-4.89	34.16	2.86	36.82	98.26
B	NH_3	$400^\circ C$	CO	-4.69	37.06	3.09	40.93	100.19
C	C_9H_7N	$400^\circ C$	CO	1.69	28.56	14.87	54.67	98.16
D	C_9H_7N	$400^\circ C$	CO	5.77	26.41	14.50	54.78	96.42
E	NH_3	$400^\circ C$	H_2	1.59	42.27	-9.09	37.99	99.50
F	NH_3	$400^\circ C$	H_2	1.08	46.53	-5.27	42.51	102.78
G	C_9H_7N	$400^\circ C$	H_2	16.02	17.39	6.27	49.54	100.49
H	C_9H_7N	$400^\circ C$	H_2	18.29	16.13	7.60	51.05	99.56
I	NH_3	$400^\circ C$	H_2/CO	-5.33	35.16	1.15	38.45	98.52
J	NH_3	$400^\circ C$	H_2/CO	-3.95	38.47	-0.78	40.37	99.84
K	C_9H_7N	$400^\circ C$	H_2/CO	10.93	21.80	7.28	51.09	94.52
L	C_9H_7N	$400^\circ C$	H_2/CO	13.23	23.73	6.41	53.11	94.36

TABLE 18

RESULTS OF RUN T005

Bomb Number	Cata- lyst	Tempe- rature	Reducing Gas	Yields, Wt% of MAF Lignite				
				Gas	Dist	S Res	Conv	Closure
A	None	400°C	CO	0.82	21.01	22.69	50.68	94.81
B	H_2S	400°C	CO	0.48	20.94	23.34	49.30	91.64
C	NH_3	400°C	CO	-29.80	42.11	26.83	50.95	101.79
D	C_9H_7N	400°C	CO	0.30	20.87	24.67	56.97	95.99
E	None	400°C	H_2	13.20	15.22	12.49	44.61	101.23
F	H_2S	400°C	H_2	13.67	30.97	11.36	53.67	104.91
G	NH_3	400°C	H_2	-1.52	44.93	-4.79	43.73	109.71
H	C_9H_7N	400°C	H_2	11.92	14.54	12.29	53.05	97.94
I	None	400°C	H_2/CO	2.33	17.82	21.93	48.53	97.64
J	H_2S	400°C	H_2/CO	35.10	-4.02	23.73	54.72	100.19
K	NH_3	400°C	H_2/CO	-19.69	23.22	33.23	43.57	105.85
L	C_9H_7N	400°C	H_2/CO	4.80	25.65	6.44	53.19	99.88

Appendix F

STATISTICAL ANALYSIS

Statistical Analysis

The statistical analysis was performed using a 95% confidence or an alpha level of 0.05.

At the 95% confidential level, alpha/2 = 0.025. Since the degrees of freedom are 15, the t-table yields t = 2.13 (36). The defining equations are:

standard deviation (SD) = square root($(\Sigma d_i^2-(\Sigma d_i)^2/n)/(n-1)$)

confidence interval (CI) = ave+SD/square root(m) * t

where
i	= 1 to 21
n	= 16
m	= number of the duplicate sample.
d	= difference between the parameters of interest in the duplicate tests.
ave	= the average of the parameters of interest in the duplicate tests.

1. Conversion (percent MAF lignite)

Bomb Number	Conversion	m	d	average	C.I.
001-A/B	-----/45.15	1	0	45.15	45.1±3.4
001-E/F	27.44/26.63	2	0.81	27.04	27.0±2.4
001-I/J	-----/37.00	1	0	37.00	37.0±3.4
002-A/B	42.42/41.75	2	0.37	41.94	41.9±2.4
002-C/D	51.88/53.17	2	-1.29	52.53	52.5±2.4
002-E/F	37.29/38.07	2	-0.78	37.68	37.6±2.4
002-G/H	53.52/54.37	2	-0.85	53.95	53.9±2.4
002-I/J	44.94/45.55	2	-0.61	45.25	45.2±2.4
002-K/L	-----/47.53	1	0	47.53	47.5±3.4
003-A/B	39.43/40.84	2	-1.41	40.14	40.1±2.4
003-C/D	47.94/-----	1	0	47.94	47.9±3.4
003-E/F	46.86/45.32	2	1.54	46.09	46.0±2.4
003-G/H	60.51/62.61	2	-2.4	61.56	61.5±2.4
003-I/J	40.57/40.25	2	0.32	40.41	40.1±2.4
003-K/L	49.06/-----	1	0	49.06	49.0±3:4
004-A/B	36.82/40.93	2	-4.11	38.88	38.8±2.4
004-C/D	54.67/54.78	2	-0.11	54.73	54.7±2.4
004-E/F	37.99/42.51	2	-4.52	40.25	40.2±2.4
004-G/H	49.54/51.05	2	-1.51	50.30	50.3±2.4
004-I/J	38.45/40.37	2	-1.92	39.41	39.4±2.4
004-K/L	51.09/53.11`	2	-2.02	52.40	52.4±2.4

$$SD = \text{square root}((\Sigma d_i^2 - (\Sigma di)^2/n)/(n-1))$$

$$= \text{square root}((61.76 - (-18.49)^2/16)/15)$$

$$= 1.60$$

Sample calculation of Confidence Interval (001-A/B)

$$CI = ave + SD/\text{square root}(m) * t$$

$$= 45.15 + 1.6/1 * 2.13$$

$$= 45.15 + 3.41$$

2. Distillate Yield (percent MAF lignite)

Bomb Number	Distillate	d	average	C.I.
001-A/B	-----/43.35	0	43.35	43.3±7.0
001-E/F	12.95/13.18	-0.23	13.07	13.0±5.0
001-I/J	-----/32.56	0	32.56	32.5±7.0
002-A/B	48.99/54.88	-5.89	51.94	51.9±5.0
002-C/D	37.18/33.79	3.39	35.49	35.4±5.0
002-E/F	44.19/50.03	-5.84	47.11	47.1±5.0
002-G/H	16.14/19.13	-2.99	17.64	17.6±5.0
002-I/J	52.81/47.44	5.37	50.13	50.1±5.0
002-K/L	-----/36.09	0	36.09	36.0±7.0
003-A/B	55.26/58.43	-3.17	56.85	56.8±5.0
003-C/D	29.43/-----	0	29.43	29.4±7.0
003-E/F	25.52/28.63	-3.11	27.08	27.0±5.0
003-G/H	20.30/24.28	-3.98	22.29	22.2±5.0
003-I/J	36.65/35.99	0.66	36.32	36.3±5.0
003-K/L	13.21/-----	0	13.21	13.2±7.0
004-A/B	34.16/37.06	-2.9	35.61	35.6±5.0
004-C/D	28.56/26.41	2.15	27.49	27.4±5.0
004-E/F	42.27/46.58	-4.31	44.43	44.4±5.0
004-G/H	17.39/16.13	1.26	16.76	16.7±5.0
004-I/J	35.16/38.47	-3.31	36.82	36.8±5.0
004-K/L	21.80/23.73	-1.93	22.77	22.7±5.0

SD = 3.30

3. Soluble Residue Yield (percent MAF lignite)

Bomb Number	Soluble Residue	d	average	C.I.
001-A/B	-----/10.45	0	10.45	10.4±5.5
001-E/F	-1.23/-0.44	-0.79	-0.84	-0.8±3.8
001-I/J	-----/ 8.70	0	8.70	8.7±5.5
002-A/B	16.33/14.85	1.48	15.59	15.5±3.8
002-C/D	9.04/11.82	-2.78	10.43	10.4±3.8
002-E/F	-3.48/-8.26	4.78	-5.87	-5.8±3.8
002-G/H	27.74/25.19	2.55	26.47	26.4±3.8
002-I/J	9.26/10.94	-1.68	10.1	10.1±3.8
002-K/L	-----/ 3.85	0	3.85	3.8±5.5
003-A/B	5.44/ 3.93	1.51	4.69	4.6±3.8
003-C/D	2.68/-----	0	2.68	2.6±5.5
003-E/F	4.08/-0.27	4.35	1.91	1.9±3.8
003-G/H	15.43/17.93	-2.5	16.68	16.6±3.8
003-I/J	14.00/10.94	3.06	12.47	12.4±3.8
003-K/L	9.86/-----	0	9.86	9.8±5.5
004-A/B	3.86/ 3.09	-0.23	2.98	2.9±3.8
004-C/D	14.87/14.50	0.37	14.69	14.6±3.8
004-E/F	-9.09/-5.27	-3.82	-7.18	-7.1±3.8
004-G/H	6.27/ 7.60	-1.33	6.94	6.9±3.8
004-I/J	1.15/-0.78	2.53	0.19	0.1±3.8
004-K/L	7.28/ 6.41	0.87	6.85	6.8±3.8

SD = 2.57

4. Gas Yield (percent MAF lignite)

Bomb Number	Gas	d	average	C.I.
001-A/B	-----/-14.38	0	-14.38	-14.3±4.7
001-E/F	14.94/ 14.34	0.6	14.64	14.6±3.4
001-I/J	-----/ 7.85	0	7.85	7.8±4.7
002-A/B	-23.42/-27.17	4.55	-24.90	-24.9±3.4
002-C/D	6.34/ 7.58	-1.24	6.96	6.9±3.4
002-E/F	1.22/ 1.03	0.19	1.13	1.1±3.4
002-G/H	14.11/ 17.32	-3.21	15.72	15.7±3.4
002-I/J	-9.44/-10.71	1.27	-10.08	-10.0±3.4
002-K/L	-----/ 8.93	0	8.93	8.9±4.7
003-A/B	-3.16/ -3.47	0.31	-3.32	-3.3±3.4
003-C/D	7.96/ -----	0	7.96	7.9±4.7
003-E/F	5.35/ 3.36	1.99	4.36	4.3±3.4
003-G/H	15.83/ 18.58	-2.75	17.21	17.2±3.4
003-I/J	-14.05/-15.22	1.17	-14.64	-14.6±3.4
003-K/L	13.20/ -----	0	13.20	13.2±4.7
004-A/B	-4.89/ -4.69	-0.2	-4.79	-4.7±3.4
004-C/D	1.62/ 5.77	-4.15	3.70	3.7±3.4
004-E/F	1.59/ 1.08	0.51	1.34	1.3±3.4
004-G/H	16.02/ 18.29	-2.27	17.16	17.1±3.4
004-I/J	-5.33/ -3.95	-1.38	-4.64	-4.6±3.4
004-K/L	10.93/ 13.23	-2.3	12.08	12.0±3.4

SD = 2.23

Appendix G
LIST OF ABBREVIATIONS

UNDREC   University of North Dakota Energy Research Center

COSTEAM   Carbon Monoxide and Steam

ASTM   American Society of Testing and Materials

MAF   Moisture Ash Free

SRL   Solvent Refined Lignite

IOM   Insoluble Organic Matter

Dist   Distillate

S Res.   Soluble Residue

Conv.   Conversion

AO-4   Anthracene oil-Batch Number 4

THF   Tetrahydrofuron

H/C   Hydrogen to Carbon ratio(mass or molar)

^{o}C Degree Celcius

^{o}F Degree Fahrenheit

^{o}K Degree Kelvin

Vol.   Volume

I.D.   Inside Diameter

O.D.   Outside Diameter

C.P.   Chemical Pure

psi   Pounds per square inch

psig   Pounds per square inch guage

mg   milligrams

c.c.   cubic centimeter

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