

Development of Mild Gasification Process

Final Report

**C.I.C. Chu
T.M. Derting
S.W. Williams
B.L. Gillespie**

Work Performed Under Contract No.: DE-AC21-87MC23289

**For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
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**By
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July 1989

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EXECUTIVE SUMMARY

This final technical report documents the technical effort over roughly a two year period by Coal Technology Corporation (CTC, formerly UCC Research Corporation) on the research program sponsored by the U.S. Department of Energy (DOE) to optimize a mild coal gasification process.

Under a previous contract with Morgantown Energy Technology Center (METC), DOE contract No. DE-AC21-84MC21108, CTC built and tested a 1500 lb/day, fixed bed batch Mild Gasification Development Unit (MGU). Testing completed under the previous contract showed that good quality hydrocarbon liquids and char can be produced in the MGU. However, the MGU was not optimized. The primary objectives of the current project were to optimize the MGU and determine the suitability of using the char as a replacement fuel for coal or coke in three types of commercial applications: industrial/utility boiler; stoker boiler; and foundry blast furnace.

To optimize the MGU, facility modifications were made to the MGU in order to solve the major problems encountered during the previous contract and a series of parametric test runs were carried out in search of the optimum operating conditions. The major modifications include the reactor diameter size, coal feeding system, coal liquid condensing system, reactor tube support system, and the char chamber design. The operating parameters tested during the process studies to gauge their individual effect on product quality and yield were coal feedstock, final coal bed temperature, coal particle size, sweep gas, and coal additive. The operating pressure was essentially atmospheric - ~1 psig vacuum to ~2 psig pressure.

The modified MGU employs two six-inch diameter reactor tubes (~eight feet in height), each connected to an eight-inch diameter sweep gas pre-heating tube. The six-inch reactor size was a compromised selection out of considerations for a somewhat increased heating rate and not too large of a reduction in coal processing capacity. The sweep gas pre-heating feature also was installed with a view to help increase the coal heating rate and to reduce the potential for secondary vapor cracking reactions. The coal feed hoppers are now located much closer to the reactor entrance point to simplify coal feeding. The reactor tubes are now fixed (welded) at the bottom and free at the top for thermal expansion to eliminate the leaking of coal gases into the furnace box. The modified condensing system originally was to utilize a venturi-scrubber/tray tower system. However, the system did not perform as well as anticipated - primarily due to the fact that a much lower sweep gas rate was utilized in the MGU than was originally anticipated. The venturi-scrubber/tray tower system was designed for a gas flow rate of +10 cfm - however at these "high" gas flow rates, a substantial amount of the coal charge was carried out of the reactor tubes in the gas stream. At lower sweep-gas flow rates, coal liquids would condense in the piping between the reactor tubes and the condensing system. Nevertheless, a well-designed slip-stream condensing system with built-in flexibility (initially installed for material balance and individual parametric run product characterization purposes) was found to work very well as a substitute for the venturi-scrubber/tray tower system. The new char chamber design with a clamp-type lid and a reduced quenching/cooling water requirement has resulted in a more gas-tight system as well as allowing for more accurate char yield measurements.

Most of these major design changes have apparently worked as testified by substantially better product quality and quantity as well as smoother overall MGU operation.

Through the parametric test run studies, the following optimum MGU operating conditions have been established:

- The use of freshly mined, high volatile bituminous coal.
- A furnace temperature of ~1000°F and a final center coal bed temperature of ~900°F.
- The use of hot nitrogen sweep gas.
- A coal feed particle size of 1/8-inch x 0.

Using these optimum operating conditions, the modified MGU has able to run day-in and day-out with good reproducibility and produce a coal liquid superior in quality to the coal liquid produced by the COALITE plant in England (the world's only commercial mild gasification plant). While it was found that a higher operating temperature can increase the liquid yield a small degree, it will result in a substantial deterioration of the liquid quality and thermal efficiency. Nitrogen sweep gas can increase the liquid yield and to a small degree improve the liquid quality. However, steam sweep gas did not appear to render the same beneficial effects as the nitrogen did, and can add a substantial burden to the gas cooling/condensing system as well as result in increased operating costs related to oil/water separation and waste water treatment. A smaller coal particle size tended to improve the coal liquid quality and generally resulted in easier char discharge from the reactor tubes. Although a substantial amount of lime additive does have a positive effect on improving the liquid quality, it greatly increases the ash content of the main MGU product - char. Since our survey on char upgrading indicates that the most economical utilization of char is via making coke from char, the ash content limitation (~8%) for coke products makes the lime additive undesirable.

The MGU char was burned successfully in a TAS System industrial boiler. Although a support fuel was required to maintain a stable flame at the burner's low fire rate, none was needed at the high fire rate. The combustion efficiency and char flame temperature were only slightly less than that for the parent coal. However, the MGU char did not appear to have the strength nor the burning characteristics (i.e., it was too reactive and thus burned off too fast) needed for the foundry furnace application without further processing.

The MGU char burned well in the stoker boiler application, but its combustion was not as complete as that of its parent coal. However, the char burned with a nearly smokeless flame, which indicates that it should be a more environmentally acceptable fuel than coal.

In conclusion, the modified MGU has worked much better than the original MGU under the previous METC/DOE contract and has been able to generate highly reproducible and high quality coal liquids and chars. The MGU coal liquid composed primarily of light alkyl and simple ring aromatic compounds (44.3% alkanes, 34.3% mono-cyclic aromatics, 13.9% di-cyclic aromatics, and 7.6% tri-cyclic aromatics). This type of coal liquid is believed to be easily upgradable into transportation type fuels and/or advanced military jet fuel. The char, with good volatile matter remaining (~10-11%), also is a good boiler fuel. However, the best means of char utilization is through further upgrading to produce a higher market value product, such as coke, so that it can effectively enhance the overall economics of the mild gasification technology.

In light of the good results achieved in this project, the next logical step appears to be the development of a continuous mild gasification process in order that the commercialization of the mild gasification technology can take place in the U.S. in the near future.

1.0 INTRODUCTION

Oil currently accounts for over 42% of the total U.S. energy consumption and over 40% of the nations oil is imported from foreign countries. The remaining oil reserve available in this country constitutes less than 6% of the proven total U.S. recoverable fossil energy reserves while coal represents over 90% of the proven total U.S. fossil energy reserves (1)*. Total coal resources in the U.S. are estimated at more than 3.9×10^{12} tons (2). Just the demonstrated coal reserve alone, the coal reserve that is proven and can be economically mined using today's technologies and mining techniques, amounts to 488×10^9 tons. At the current annual U.S. coal production rate of about 900×10^6 tons, the demonstrated coal reserve alone will last more than 500 years. In light of this contrast in available resources, coal vs. oil, it is very desirable to make good use of our abundant coal resource in our ever more difficult pursuit of energy independence.

Most of the high-severity coal conversion processes that have been developed or are being developed are too complicated, too expensive or both, largely because of their reliance on very severe operating conditions and heavy uses of expensive hydrogen.

While conventional coal devolatilization (or "mild gasification") processes are among the oldest methods for obtaining liquid fuels from coal, they are also technically among the least complex. Mild gasification also has the advantages of higher thermal efficiencies than those of other routes to liquid synfuels from coal. Efficiencies of 85-90% can be expected from mild gasification processes, in contrast to only 50 to 70% for high-severity, indirect and direct liquefaction processes (3). Recent papers reporting various coal liquid qualities and hydrotreatment requirements also indicate that mild gasification liquids are generally superior in quality to those produced from high-severity coal liquefaction processes and require a substantially lesser degree of hydrotreating (3-8).

However, in the existing mild gasification processes, the relative quantities and properties of the co-products are not optimized to make the technology economically and environmentally viable. Many times, either the liquid yield is too low or the liquid quality is poor; and the main product, char (representing 65-75 wt.% coal feedstock), often cannot find its proper marketplace.

Under a previous contract with Morgantown Energy Technology Center (METC), Department of Energy (DOE) Contract No. DE-AC21-84MC21108, Coal Technology Corporation (CTC) (formerly UCC Research Corporation) built and tested a 1500 lb/day Mild Gasification Process Development Unit (MGU). The MGU, as tested under the previous contract, is shown in Figure 1. Testing completed under the previous contract showed that good quality hydrocarbon liquids and good quality char can be produced in the MGU. However, the MGU was not optimized. The primary objectives of the current project are to optimize the MGU and determine the suitability of char for several commercial applications. The program consists of three tasks as follows:

- Task 1 - Test Plan
- Task 2 - Optimization of the Mild Gasification Process
- Task 3 - Evaluation of Char and Char/Coal Blends as
a Boiler/Blast Furnace Fuel

* Numbers in parentheses designate references at the end of this report.

2.0 RESULTS AND DISCUSSIONS

2.1 TASK 1. TEST PLAN FOR OPTIMIZATION OF THE MILD GASIFICATION PROCESS

2.1.1 Objective

The object of Task 1 was to develop a test plan for optimizing the mild gasification process.

2.1.2 Discussion

Optimization was to be accomplished in two phases. Phase 1 involved modification of the mild gasification unit (MGU) to improve unit operation. The MGU was built and initially tested during research performed by Coal Technology Corporation (CTC) under Department of Energy Contract No. DE-AC21-84MC21108, "Management of Coal Waste by Energy Recovery: Mild Gasification (Pyrolysis) of Coal Preparation Wastes". In addition to MGU modifications, Phase 1 also included reactor tube diameter tests to determine the optimum reactor tube diameter for the MGU.

Phase 2 of the optimization process involved conducting a series of parametric tests on the modified MGU. The effects of temperature, coal type, coal particle size, sweep gas, and lime additive on the quantity and quality of the liquid, solid, and gas products were investigated in these tests. A copy of the original test plan for this project is included in Appendix A.

2.1.2.1 Phase 1 Test Program - Reactor Diameter Tests

A total of nine reactor diameter tests and two hot sweep gas tests were conducted under Phase 1, all using bituminous coal. These tests are summarized in Table 1.

Table 1.
Test Program for Reactor Tube Diameter Testing

<u>Test No.</u>	<u>Reactor Diameter (Inches)</u>	<u>Furnace Temperature (°F)</u>	<u>Final Bed Temperature (°F)</u>
1	8	1200	1100
2	8	1100	1070
3	4	1100	1094
4	4	1100	1100
5	4	1100	1102
6	4	1100	1100
7	4	1100	1104
8	6	1100	1095
9	6	1100	1105
10	6	1100	1087
11	6	1100	1080

Note: Tests 1-9 were reactor diameter optimization tests. Tests 10 and 11 were hot N₂ sweep gas tests.

All of these MGU tests were conducted using non-tapered stainless steel reactor tubes. The primary objective of the first nine (9) tests was to determine the optimum reactor tube diameter for the MGU. Reactor tube diameters of 4, 6, and 8-inches were tested.

The final two Phase 1 tests (Tests #10 & #11) were conducted to investigate the effects of hot sweep gas on the quantity and quality of the mild gasification products as well as its effect on the coal heating rate.

2.1.2.2 Phase 2 Test Program - Parametric Testing

A total of twenty-one (21) parametric tests were conducted on the MGU (see Table 2). These tests were designed to determine the effects of the following parameters on unit performance and on product yield and quality:

Table 2.
Test Program for MGU Parametric Testing

<u>Test No.</u>	<u>Coal Type</u>	<u>Temperature (°F)</u>	<u>Particle Size (Inches)</u>	<u>Sweep Gas</u>	<u>Additive</u>
#12/P1	HVB #1 ^a	900	1-1/2 x 0	-	-
#13/P2	HVB #1	1000	1-1/2 x 0	-	-
#14/P3	HVB #1	1100	1-1/2 x 0	-	-
#15/P4	HVB #1	1200	1-1/2 x 0	-	-
#17/P6	HVB #1 "Fresh"	900	1-1/2 x 0	-	-
#18/P7	HVB #1	900	1-1/2 x 0	N ₂	-
#19/P8	HVB #1	900	1/8 x 0	-	-
#20/P9	HVB #1	900	1/8 x 0	-	10% Lime
#21/P11	HVB #1	900	1/8 x 0	-	20% Lime
#22/P12	SBT ^b	900	1/2 x 0	-	-
#24/P13	Lignite	900	3/4 x 0	-	-
#25/P14	CPW ^c	900	1/16 x 0	-	-
#26/P15	HVB #2	900	1 x 0	-	-
#27/P16	HVB #1	850	1-1/2 x 0	-	-
#28/P17	HVB #1	900	1-1/2 x 0	H ₂ O	-
#29/P18 ^d	HVB #1	900	1-1/2 x 0	-	-
#30/P19	HVB #1	900	1/8 x 0	N ₂	-
#31/P20	HVB #1	900	1/4 x 0	N ₂	-
#32/P21	HVB #1	900	1/2 x 0	N ₂	-

a HVB = High Volatile Bituminous Coal

HVB #1 = H&K Bituminous Coal

HVB #2 = Wellmore #8 Bituminous Coal

b SBT = Sub-Bituminous Coal

c CPW = Coal Preparation Waste

d Insulation removed from upper part of reactor tubes inside oven.

- Temperature - 900°F to 1200°F
- Coal Type - Bituminous, Sub-Bituminous, & Lignite
- Coal Feed Particle Size - 1/16-inch x 0 to 1-1/2-inch x 0
- Sweep Gas - Nitrogen, Steam, & Non-Condensable Coal Gas
- Lime Additive - 10% & 20% by weight

One objective during the parametric testing (Phase 2) was to obtain more detailed information about what is occurring inside the MGU during the mild gasification process. Toward this end, the following process and operating data were collected during each test run:

- Coal Temperature vs. Time ([1] at the center of the bed and
[2] at the mid-point between the reactor wall and the center of the bed)
- Furnace (Oven) Temperature vs. Time
- Coal Gas Temperature vs. Time ([1] immediately after the gases exit the reactor tube, and
[2] at the vacuum pump - just before the flare)
- Coal Feedstock Weight
- Additive Weight (if used)
- Char Weight
- Liquid Product Weight
- Non-Condensable Gas Flow Rate and Total Mass Flow
- Gas Pressures ([1] at the sweep gas inlet,
[2] at the top of reactor tubes, and
[3] after the vacuum pump)
- Flow Rate of Sweep Gases

2.2 TASK 2 - OPTIMIZATION OF THE MILD GASIFICATION PROCESS

2.2.1 Objective

The objectives of this task were to (A) modify the MGU to optimize the unit operation; (B) conduct parametric tests to determine the effect of process parameters on product (gas, condensible, and char) quantity and quality; and (C) produce sufficient quantities of char and hydrocarbons in order to evaluate them in various commercial applications.

2.2.2 Facility Modification

A schematic diagram of the original MGU facility used in the earlier contract is shown in Figure 1. The first major area of modification was to the reactor tube diameter. The previous eight inch diameter reactor tubes caused extremely slow heating rates ($\sim 5^{\circ}\text{F}/\text{min}$) in the center of the coal bed. Lower heating rates generally result in lower liquid yields and better liquid quality; conversely, higher heating rates generally result in higher liquid yields and poorer liquid quality. While the quality of liquids from early MGU tests was good, liquid yields were much lower than expected. Yields were expected to be in the 12-15% range (per laboratory test results), but rather were in the 5-7% range. Therefore, in an attempt to improve the coal liquid yield, without significantly reducing the liquid quality, a series of reactor diameter optimization tests were called for.

The second area of modification to the original MGU design was the coal feed system. Previous tests were hampered by the blockage of the coal feed chutes, coal sticking in the volumetric hoppers, and with incomplete feeding of coal into the reactor tubes. Also, the flexible screw conveyor designed to carry coal from ground level to the volumetric hoppers on the fourth floor of the MGU never worked properly. The flexible screw broke during every attempt to convey coal to the volumetric hoppers. Therefore, a bucket-hoist system was used to convey coal from ground level to the volumetric hoppers. In order to correct the remaining coal feeding problems, the following modifications were made: The coal feed chutes were shortened and the volumetric hoppers were installed on the third floor directly next to the reactor tube top assembly. This would eliminate the plugging of the coal feed chutes and incomplete feeding of coal into the reactor tubes.

The third area of modification involved the coal liquid condensing system. Previously, the MGU used only an indirect cooling system to recover the condensible hydrocarbons. This indirect cooling system (which essentially consisted of two concentric barrels with ice water and/or dry ice placed in the annular space between the two barrels) was largely inefficient for condensing the aerosol-type vapors from the mild gasification process. In addition, the system was cumbersome and inconvenient to use. To improve condenser efficiency, the previous system was replaced with a two-stage, direct quenching, liquid recovery system. The modified system incorporated a venturi-scrubber followed by a tray tower (see Figures 8 & 9).

The fourth major area of modification was the reactor tube support system. Previously, the reactor tubes were supported (fixed) at the top of the furnace and were free at the bottom to allow for thermal expansion. A satisfactory seal between the char chamber and furnace was never achieved. With the slight

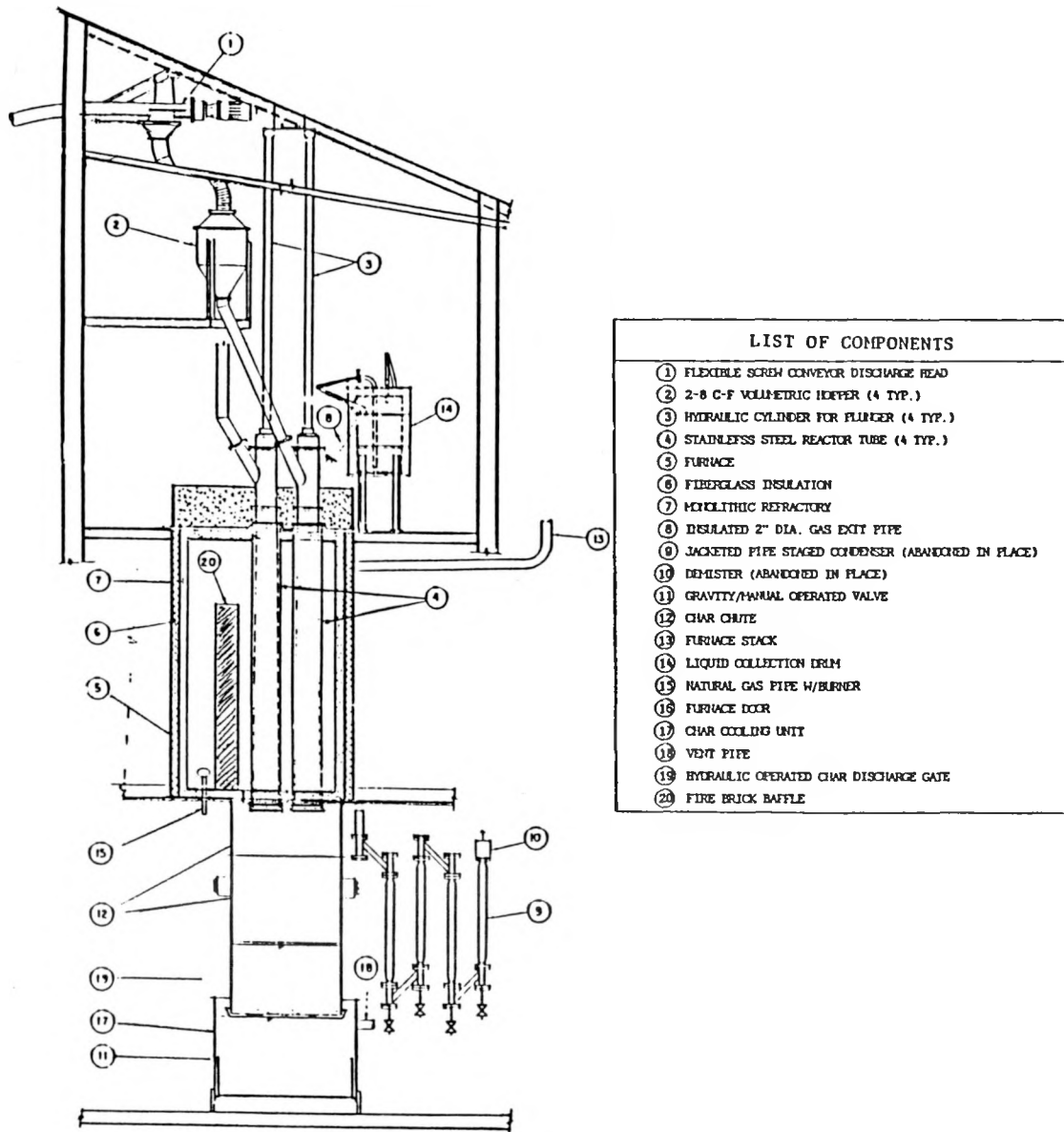


Figure 1.
Unmodified Mild Gasification Unit (MGU)

pressure conditions present at the beginning of the test run, gas escaped into the furnace chamber and was lost. With the slight vacuum conditions present during the middle and latter part of a test run, furnace gases as well as air from the char chamber were pulled into the reactor tubes causing coal oxidation and other undesired reactions in the coal bed. The leaking problem between the furnace and char chamber was especially damaging since the bottom reactor gate was not designed to form a gas-tight seal. To prevent this type of leaking problem, the support system for the reactor tubes was modified so that the tubes were fastened to the bottom of the furnace floor and were free at the top to allow the reactor tubes to float for thermal expansion.

The last major area of modification was to the char chamber. The previous char chamber design utilized a 2-inch water gate seal to prevent the coal gases from escaping from the bottom of the reactor tubes. While this design did prevent excessive pressure from building up inside the MGU, it also allowed a substantial volume of condensable gas to be "burped" out of the unit. In addition, the previous char chamber design utilized large volumes of water to quench the char, which generally resulted in difficulties in determining an accurate char yield.

The modified char chamber is substantially smaller than the previous unit and utilizes water cooled walls and water sprays to cool the char. The char chamber incorporates a clamp-type lid (similar to those on the coal hoppers) to effectively seal the unit.

2.2.2.1 Reactor Tube Diameter Optimization Tests

Reactor tube diameter optimization tests were conducted on the MGU to determine the ideal reactor diameter for the mild gasification process. As stated previously, the original 8-inch diameter cast reactor tubes utilized on the MGU caused extremely slow heating rates ($\sim 5^{\circ}\text{F}/\text{minute}$) in the center of the coal bed. This was believed to be a possible contributor to the low coal liquid yields obtained in previous testing. To determine if a different reactor tube diameter (and thus a different heating rate) would have an appreciable effect on the liquid yield and quality, tests were conducted with reactor tube diameters of 8-, 6-, and 4-inches. In order to conduct these reactor diameter optimization tests, the existing coal feed system, hydraulic char discharge rams, 8-inch cast reactor tubes, and char chute assembly were removed from the MGU. A modified coal feed and gas exit assembly (see Figure 3) was installed and utilized for these tests. Stainless steel pipe was used in contrast to the cast reactor tubes used in previous tests. The stainless steel reactor tubes were not tapered and were sealed at the bottom with a screw-on cap.

For each reactor diameter optimization test, the following test procedure was utilized:

- The furnace was preheated to the desired temperature and the system purged with nitrogen.
- The bottom portion of the reactor tube (which extended outside the bottom of the oven) was filled with sand so that all of the coal charge would be in the heated part of the reactor tube.
- A weighed amount of coal was then charged into the reactor tube.
- Coal temperature was monitored with two thermocouples. Both thermocouples were located approximately four-feet high in the coal bed. One thermocouple was placed approximately one inch from the outside wall of the reactor and the second in the center of the coal bed (one-half the diameter of the reactor tube from the outside of the tube wall).
- As the coal temperature in the center of the coal bed approached 1100°F, the furnace was shut off and nitrogen added at the bottom of the reactor tubes.
- Condensibles were collected for an additional 2 hours after the target bed temperature was reached.
- The char was allowed to cool in the reactor tube and was discharged the next morning.

During the reactor diameter tests with the 4-inch and 6-inch reactor tubes, it was observed that a char-like bridge formed at the top of the reactor inhibiting the flow of gases from the reactor tube. It is believed that this phenomenon was responsible for the low liquid yields in Tests 4-6. During the last 4-inch reactor diameter test (Test 7), a probe was periodically inserted through the top of the gas-exit manifold into the reactor in an attempt to determine when and how the char-like bridge was formed. By using this procedure, it could not be determined when or if the bridge had formed. This procedure was again utilized in tests 8 and 9 with the 6-inch reactor with no success. An inspection of the reactor after each of these tests showed that the bridge had indeed formed. However, the rodding action of the probe apparently kept the center of the reactor open. This was probably the reason for the increased yield in Test 7.

The location of the bridge material is shown in Figure 3. A sample of the bridge that formed during test 9 was obtained for analysis. The results are shown below.

<u>Proximate (dry wt.%)</u>		<u>Ash Composition (dry wt.%)</u>	
Ash	22.06	SiO ₂	84.44
Volatile Matter	11.41	Al ₂ O ₃	4.70
Fixed Carbon	64.51	TiO ₂	0.61
<u>Ultimate Analysis (dry wt.%)</u>		CaO	0.23
Carbon	70.68	K ₂ O	0.57
Hydrogen	2.77	MgO	0.21
Nitrogen	1.44	Na ₂ O	0.19
Oxygen	1.88	Fe ₂ O ₃	4.01
Sulfur	0.72	P ₂ O ₅	0.43
		SO ₃	1.42
		Undetermined	3.19

The high ash content (22%) of the bridge sample was almost 3 times that of the feed coal and 2 times that of the char product. Examination of the ash composition results reveal a possible explanation. The bridge sample ash contained 84.4% silicon dioxide. Silicon dioxide is the major component in sand and it is believed that, because the 4" (or 6") tube fit beneath the original 8" flange and upper part section, some of the sand used to fill the bottom portion of the reactor tube (that portion which extended below the furnace floor) remained around the flange area on top of the smaller reactor tube and was combined with the condensible to form the bridge. Figure 2 shows where the bridge formed and the area that sand was present.

Two hot nitrogen sweep gas tests were also conducted using the 6-inch diameter reactor tube and an 8-inch diameter pipe as the nitrogen sweep gas heater. Figure 2 shows the schematic of how the nitrogen was heated and introduced into the reactor tube. The same test procedure utilized in the previous reactor diameter optimization tests was utilized for the first hot nitrogen sweep gas test, with the following exceptions: The nitrogen flow rate was adjusted to 5 scfm while the coal (55.0 lbs.) was charged into the 6-inch reactor tube; once charging was completed, the nitrogen flow rate was increased to 10 scfm. As the coal temperature in the center of the coal bed approached 1100°F, the furnace and hot nitrogen sweep gas were turned off.

The procedure for the second hot nitrogen sweep gas test was similar, except that there was no nitrogen flow through the reactor tube during the coal charging process. The Wellmore #8 bituminous coal feedstock analyses and operating conditions for all of the reactor diameter optimization tests and hot sweep gas tests are summarized in Tables 3 and 4.

Aside from the bridge formation during the 4- and 6-inch diameter tests, the reactor diameter optimization tests were conducted without any problems. The results for the reactor diameter optimization tests as well as for the hot nitrogen sweep gas tests are summarized in Table 5. As shown in Table 5, the condensible yields during the reactor diameter optimization tests were slightly increased as reactor tube diameter was decreased (heating rate was

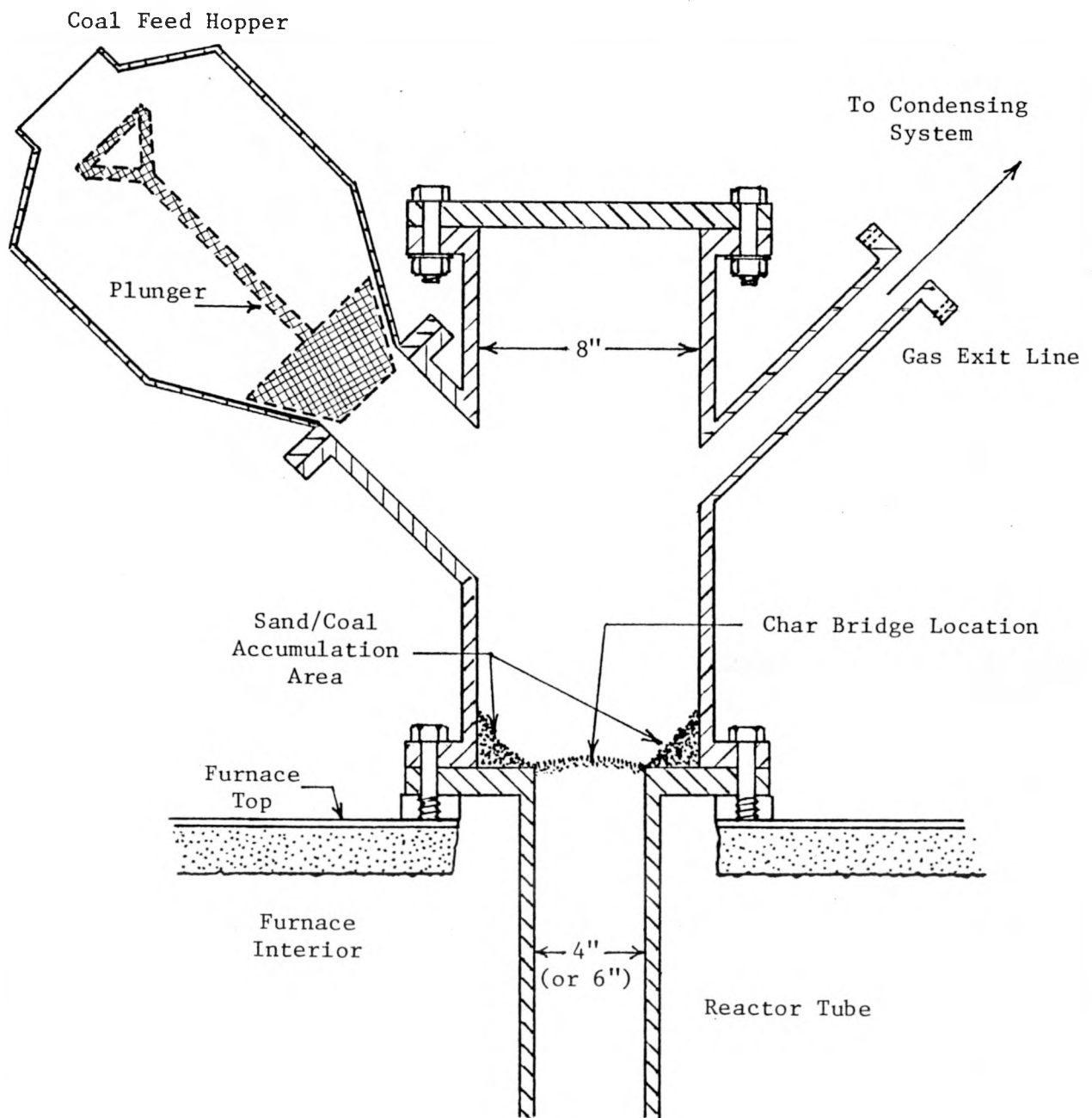


Figure 2. Location of Reactor Tube Partial Blockage Due to Bridge Formation During 4- and 6-Inch Reactor Tube Tests

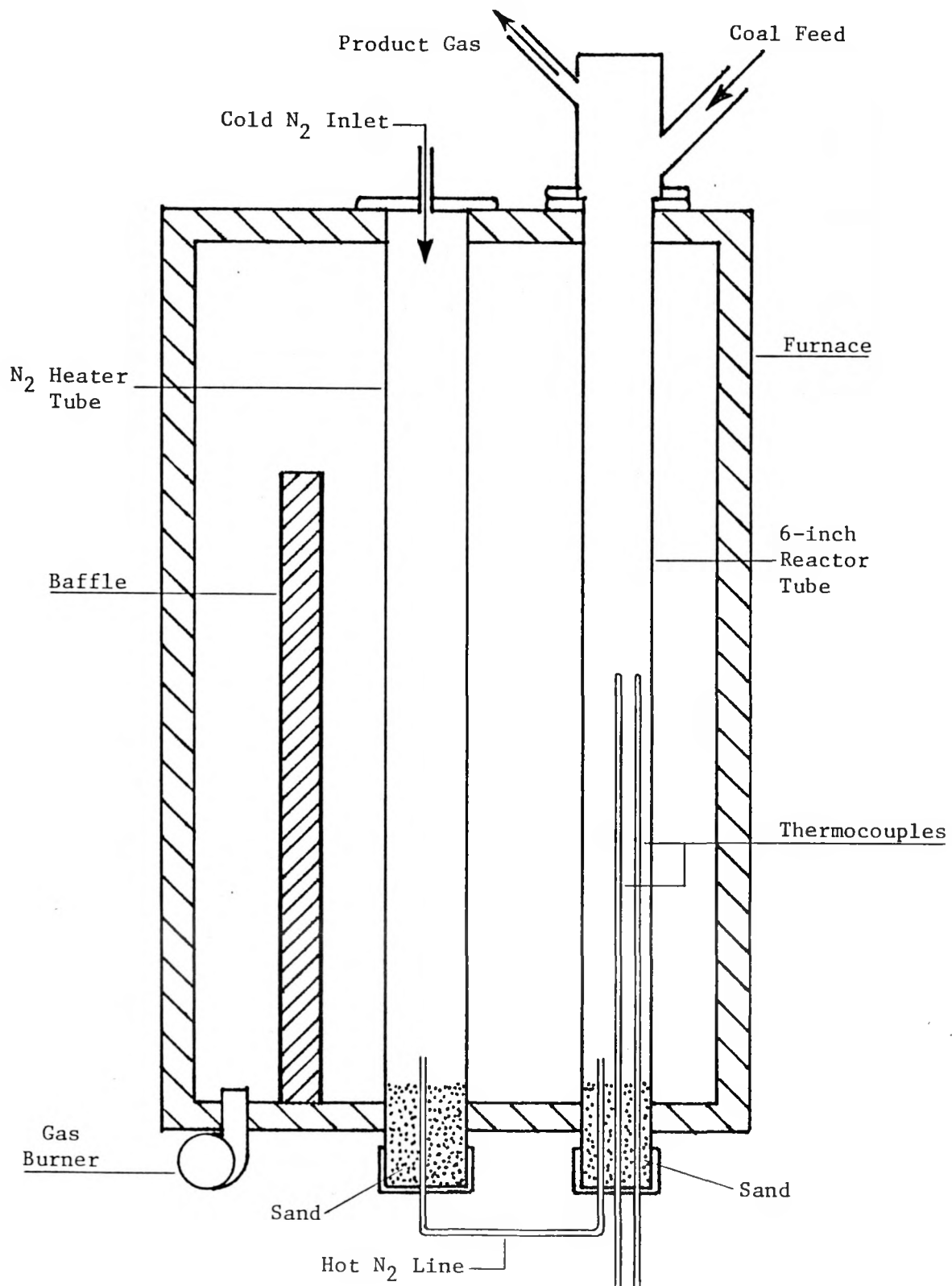


Figure 3.
Reactor Configuration for Hot N₂ Sweep Gas Tests

increased). The difference in yields can be attributed to the different reactor diameters and thus different heating rates. Figure 4 shows time vs. temperature curves for the 4-, 6-, and 8-inch reactor tubes. Char and condensible analyses are shown in Table 6.

The reactor diameter optimization tests demonstrated several points. First, straight reactor tubes could be used in place of tapered tubes in the MGU (however, the use of tapered reactor tubes is recommended for a production type unit to greatly reduce the likelihood of char discharge problems). Also, the tests showed that the increased heating rate obtained by using smaller diameter reactor tubes resulted in only a slight increase in the liquid yield. Thus, it appears that heating rate, in the range studied here (~ 2.5 - $32^{\circ}\text{F}/\text{min}$), has only a minor effect on liquid yield. Finally, the product analyses showed that different reactor diameters (4-, 6-, and 8-inches) have little or no effect on product quality. Volatile matter and ash content of the char were different from test to test, but there did not appear to be a trend dependent on reactor diameter. The average molecular weight of the condensibles for the 4-inch and 6-inch tests were somewhat lower than the average molecular weight of the condensibles for the 8-inch test, but H/C atomic ratio, nitrogen, sulfur, and oxygen contents for the condensibles were similar for all reactor diameters tested.

The tests with hot nitrogen sweep gas showed that the residence time required to reach the final bed temperature could be substantially reduced. For the first hot nitrogen sweep gas test (Test 10), only 63 minutes was required to reach the final bed temperature of 1087°F (see Figure 5). This is equivalent to an average heating rate of $16.1^{\circ}\text{F}/\text{min}$. After the reactor cooled and was opened up to remove the char, it was observed that a substantial amount of coal had been carried out of the furnace zone (see Figure 6). As the char was removed from the reactor tube, the unreacted coal was unavoidably mixed with the char. This prevented accurate determination of condensible and char yields as well as accurate char analysis. The char produced with the hot nitrogen sweep gas appeared to be more porous than char produced with no nitrogen sweep.

Table 3.
Feedstock Analysis For MGU Tests

<u>Ultimate Analysis, dry wt. %</u>	<u>W#8A¹</u>	<u>W#8B²</u>
Carbon	78.62	79.86
Hydrogen	5.09	5.05
Nitrogen	1.53	1.50
Sulfur	1.49	0.99
Chlorine	0.11	-
Oxygen	5.47	5.29
Ash	7.69	7.31
 <u>Proximate Analysis, dry wt. %*</u>		
Ash	7.69	7.31
Volatile Matter	30.64	31.67
Fixed Carbon	61.67	61.02
*As-Received Moisture	3.94	7.69

¹W#8A used during the reactor diameter optimization tests.

²W#8B used during the hot nitrogen sweep gas tests.

Table 4.
Test Conditions For Reactor Diameter Optimization
And Hot Nitrogen Sweep Gas Tests

<u>Test No.¹</u>	<u>Reactor Diameter (Inches)</u>	<u>Furnace Temp. (°F)</u>	<u>Final Bed Temp. (°F)</u>	<u>Hot Nitrogen Sweep (scfm)</u>	<u>Nitrogen Purge During Charging</u>
1	8	1200	1100	0	No
2	8	1100	1070	0	No
3	4	1100	1094	0	No
4	4	1100	1100	0	No
5	4	1100	1102	0	No
6	4	1100	1100	0	No
7	4	1100	1104	0	No
8	6	1100	1095	0	No
9	6	1100	1105	0	No
10	6	1100	1087	10	Yes
11	6	1100	1080	10	No

¹ Tests 1-9 were reactor diameter optimization tests.
Tests 10 and 11 were hot N₂ sweep gas tests.

Table 5.
Product Yields For Reactor Diameter Optimization
And Hot N₂ Sweep Gas Tests

Test No.	Reactor Diameter (Inches)	Hot Nitrogen Sweep	Product Yields Wt.% As Received Coal			
			Char	Condensibles	Water	Gas ^a
1	8	No	75.2	3.7	5.4	15.7
2	8	No	76.9	4.8	3.9	14.4
3	4	No	- ^b	5.7	5.0	- ^b
4	4	No	72.1	2.7	5.5	19.7
5	4	No	73.2	- ^c	- ^c	21.7
6	4	No	66.7	- ^c	- ^c	28.3
7	4	No	70.5	5.4	2.6	21.5
8	6	No	68.9	4.9	3.2	23.0
9	6	No	69.1	4.7	3.8	22.4
10	6	Yes	(-----	See Note d Below	-----)	
11	6	Yes	(-----	See Note d Below	-----)	

^a Gas Yields determined by difference.

^b An indeterminate error in the char collecting and weighing procedure resulting in an apparent char yield of 89.2%, which, given the condensible and water yields, does not seem probable. Because of this error, the gas (by difference) could not be determined.

^c Due to errors in handling, the exact yield of condensibles and water is not known for these two runs. The total liquid yield (condensibles + water) was 5.0% for both runs.

^d Because of discrepancies in the sweep gas flow pattern, heating rate, etc. the yield are not considered to be representative of a test run with hot nitrogen sweep gas and therefore are not presented.

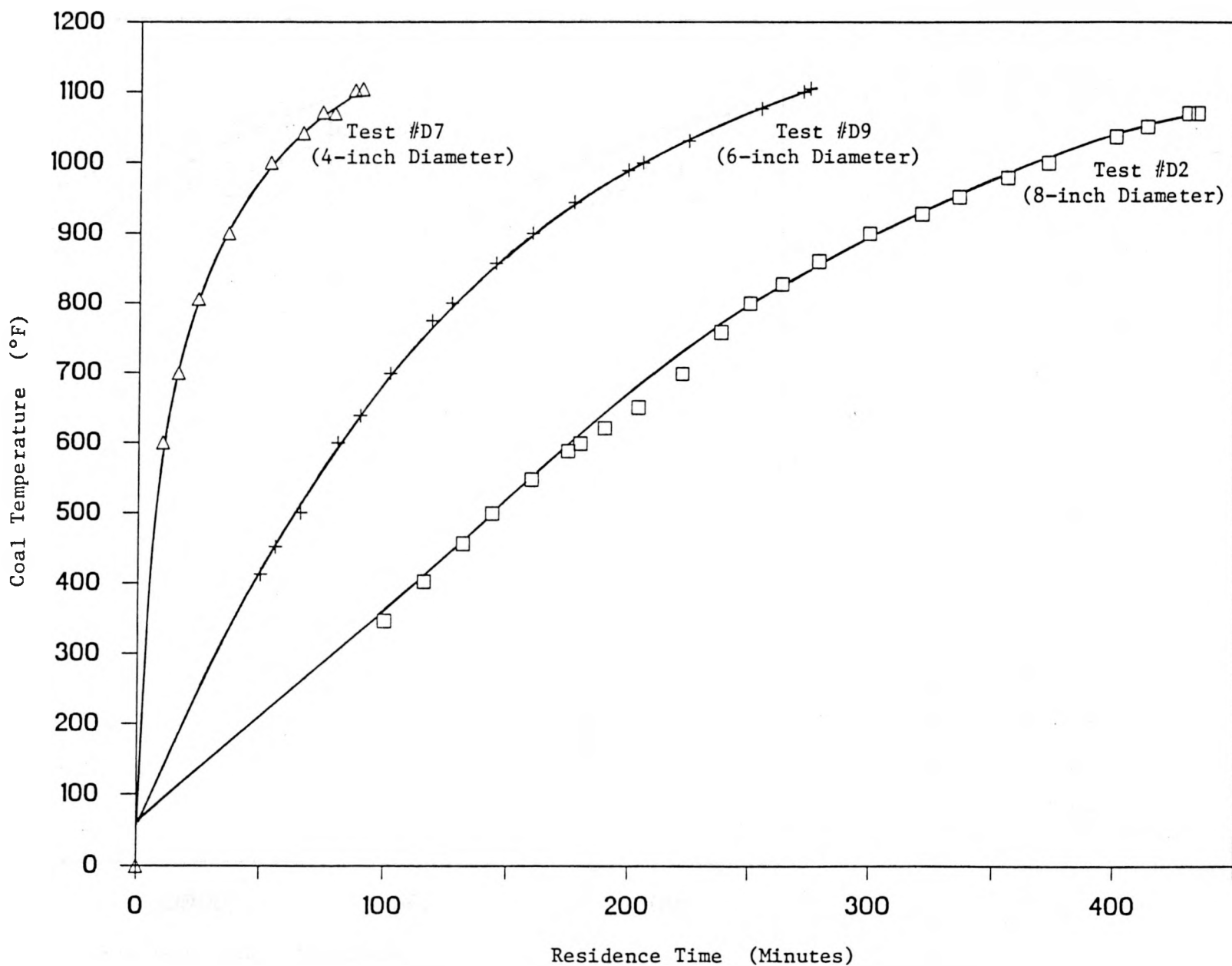


Figure 4.
Coal Heating Rates for 4, 6, and 8-inch Diameter Reactor Tube Tests
(as measured in the center of the reactor tubes)

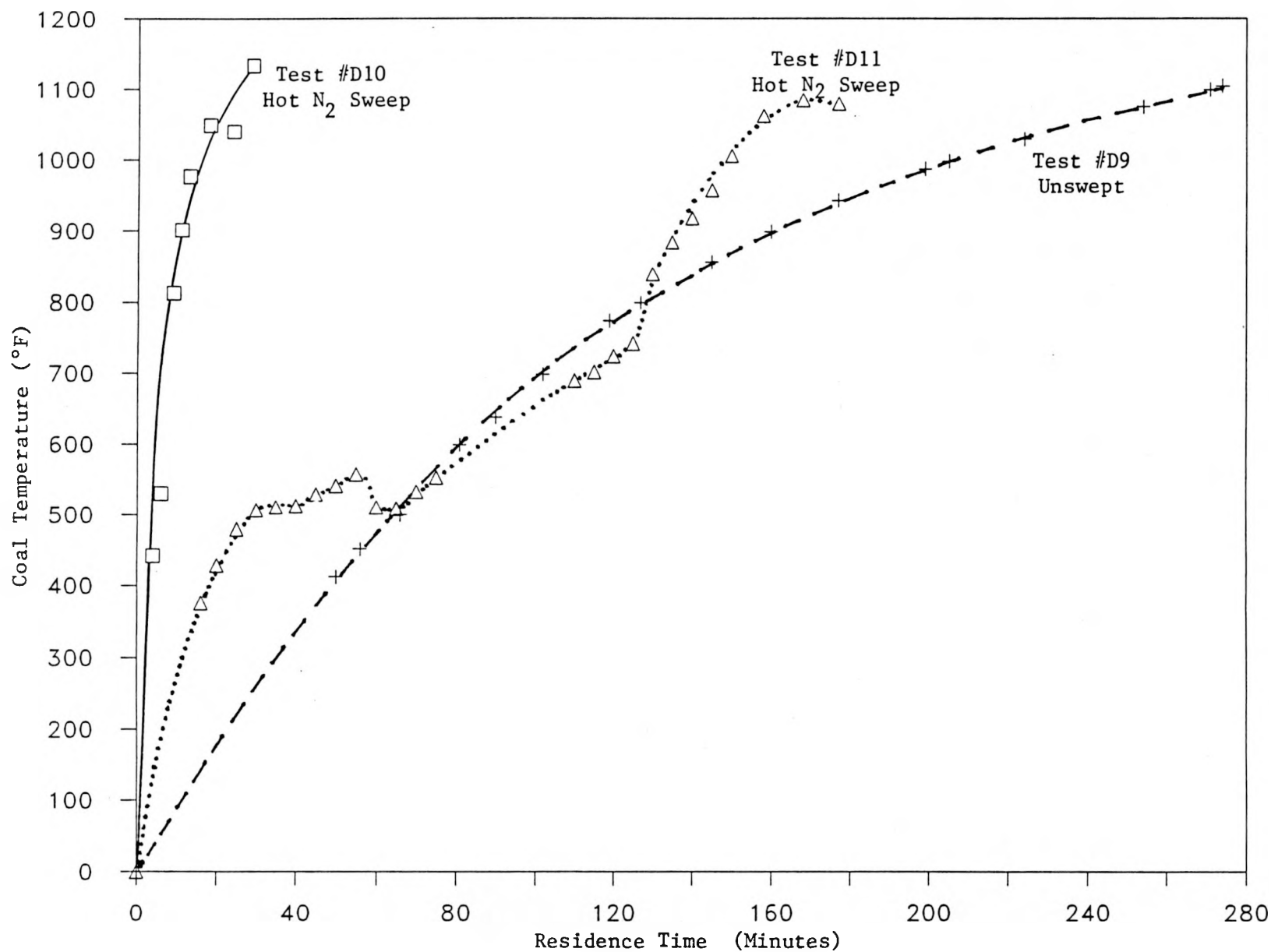


Figure 5.
Coal Heating Rates for the Hot N₂ Sweep and Unswept Tests
(as measured in the center of the 6-inch diameter reactor tube)

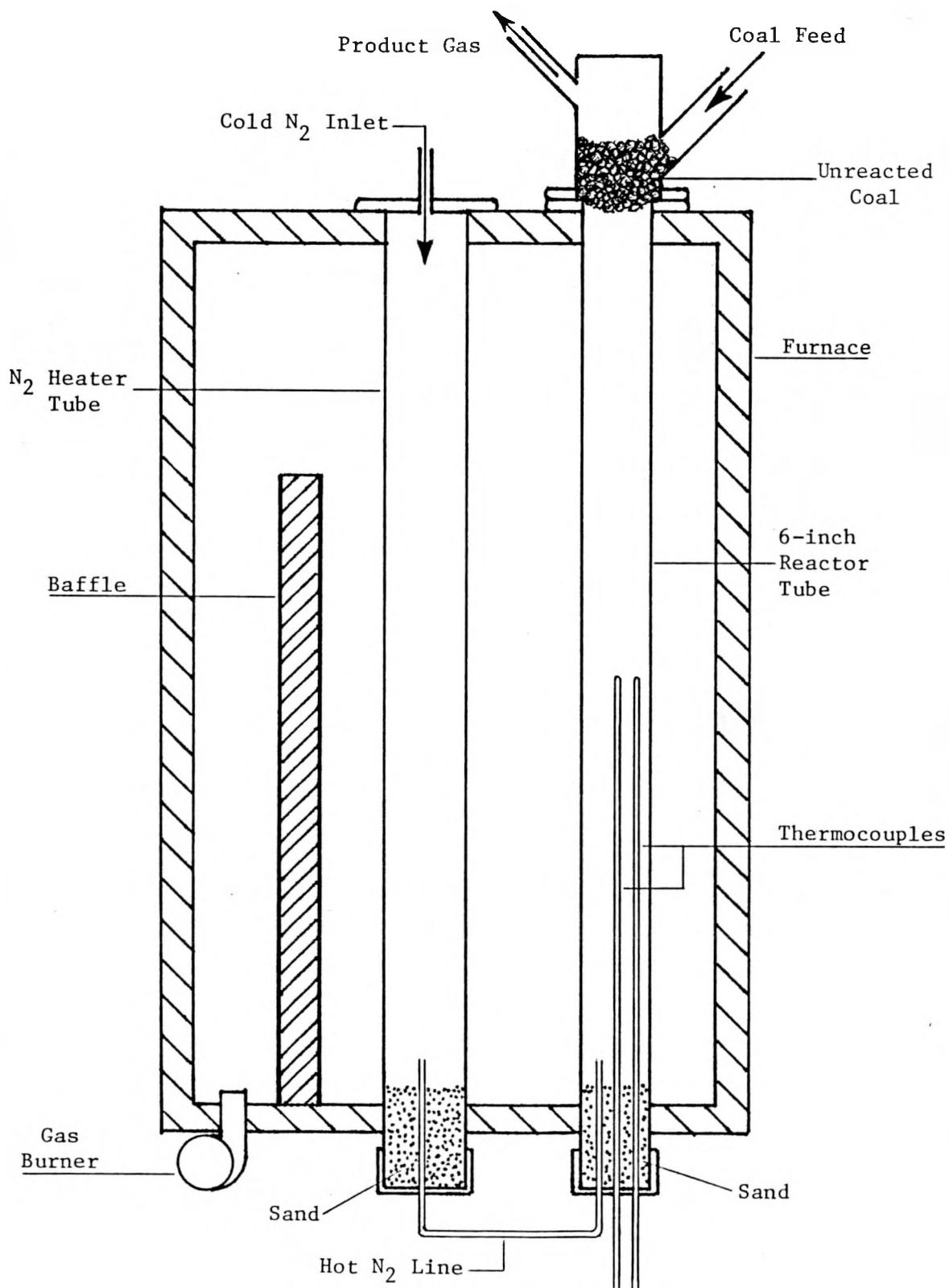


Figure 6.
Unreacted Coal Location for Test #D10

Table 6.
Product Analysis For Reactor Diameter Optimization Tests

<u>Char Analysis For Reactor Diameter Tests</u>									Heating Value (BTU/lb)
<u>Sample</u>	<u>Ash</u>	<u>Volatile Matter</u>	<u>Fixed Carbon</u>	<u>C</u>	<u>H</u>	<u>N</u>	<u>S</u>	<u>O</u>	
Test 2-8"	10.42	5.56	84.02	82.00	2.26	1.74	1.27	2.31	13,336
Test 3-4"	12.72	9.61	77.67	80.43	2.26	1.83	1.17	1.59	13,023
Test 7-4"	11.90	4.26	83.54	79.68	2.29	1.77	1.29	3.07	13,444
Test 9-6"	11.13	11.64	77.23	80.54	2.98	1.82	1.13	2.40	13,364

<u>Liquid Analysis For Reactor Diameter Tests</u>							
<u>Sample</u>	<u>C</u>	<u>H</u>	<u>N</u>	<u>S</u>	<u>O</u>	<u>H/C Atomic Ratio</u>	<u>Molecular Weight</u>
Test 2-8"	86.56	6.32	1.15	0.84	5.24	0.87	274
Test 3-4"	86.36	6.08	1.11	0.86	5.63	0.84	269
Test 7-4"	86.84	6.34	1.09	0.96	4.83	0.87	234
Test 9-6"	87.20	6.43	1.23	0.97	4.62	0.88	238

In response to the problem experienced in Test 10 with the coal being carried out of the furnace zone, no nitrogen flow was used during the coal charging stage in Test 11. After the coal was loaded into the reactor, nitrogen was added at 10 scfm. The temperature increase during Test 11 was not as quick as during Test 10. As the temperature approached ~750°F, the heating rate slowly decreased. After 2 hours, the nitrogen was turned off and from that point on, the heating rate was observed to be nearly the same as it was during tests without nitrogen sweep (see Figure 5). Examination of the char after the test showed that the nitrogen sweep gas was not uniformly distributed through the coal bed. It appeared that no nitrogen sweep passed through the outside portion of the bed (near the reactor walls) and that all of the sweep gas had traveled through the center of the bed. The outside portion of char (near the reactor wall) were very similar in appearance to char produced with no nitrogen sweep and a channel had been created in the center of the bed. It is believed that the thermocouple was in this void space and was reading the nitrogen temperature (4-feet high in the center of the bed) and that this was the reason for the very slow temperature rise above 700°F. Because of the discrepancies in the sweep gas flow pattern, heating rate, etc. the yields and product quality are not considered to be representative of a run with hot nitrogen sweep gas and therefore are not presented.

2.2.2.2 Reactor Tube Selection

Based on the results of the reactor diameter optimization tests and the hot nitrogen sweep gas tests, it was decided that the new MGU would be modified to a two (2) reactor tube system. Two 6-inch diameter, straight (not tapered) type 309 schedule 40 stainless steel pipes would be utilized as the MGU coal

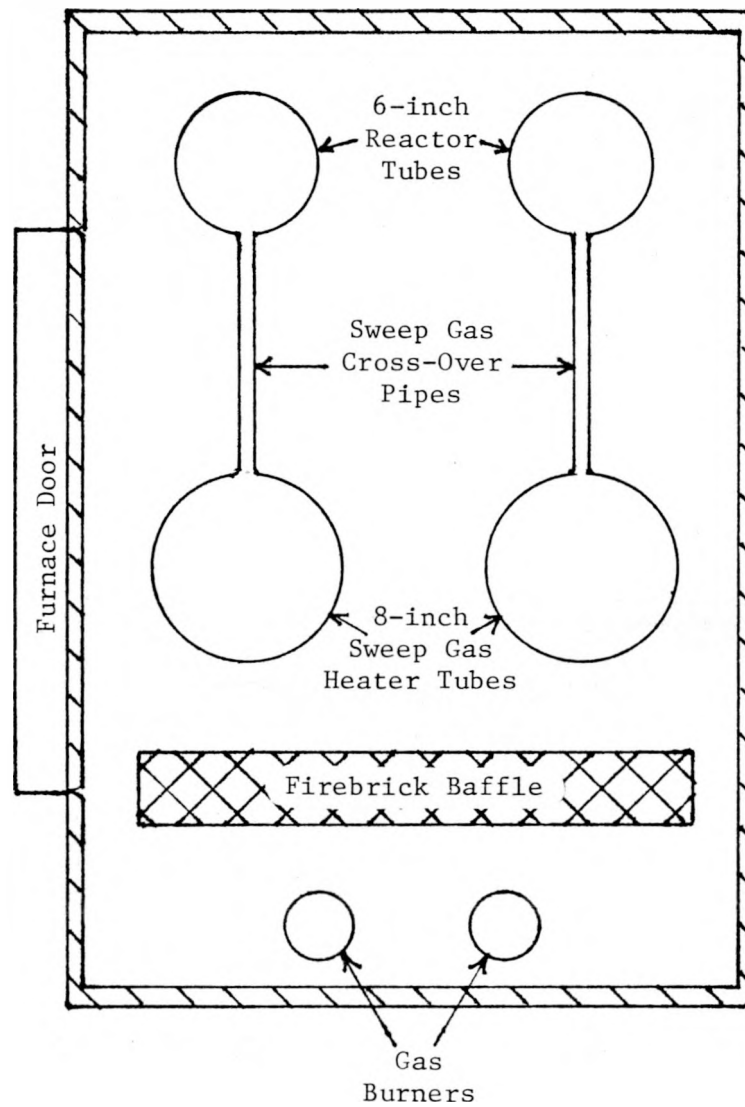


Figure 7.
Furnace Layout for Modified MGU

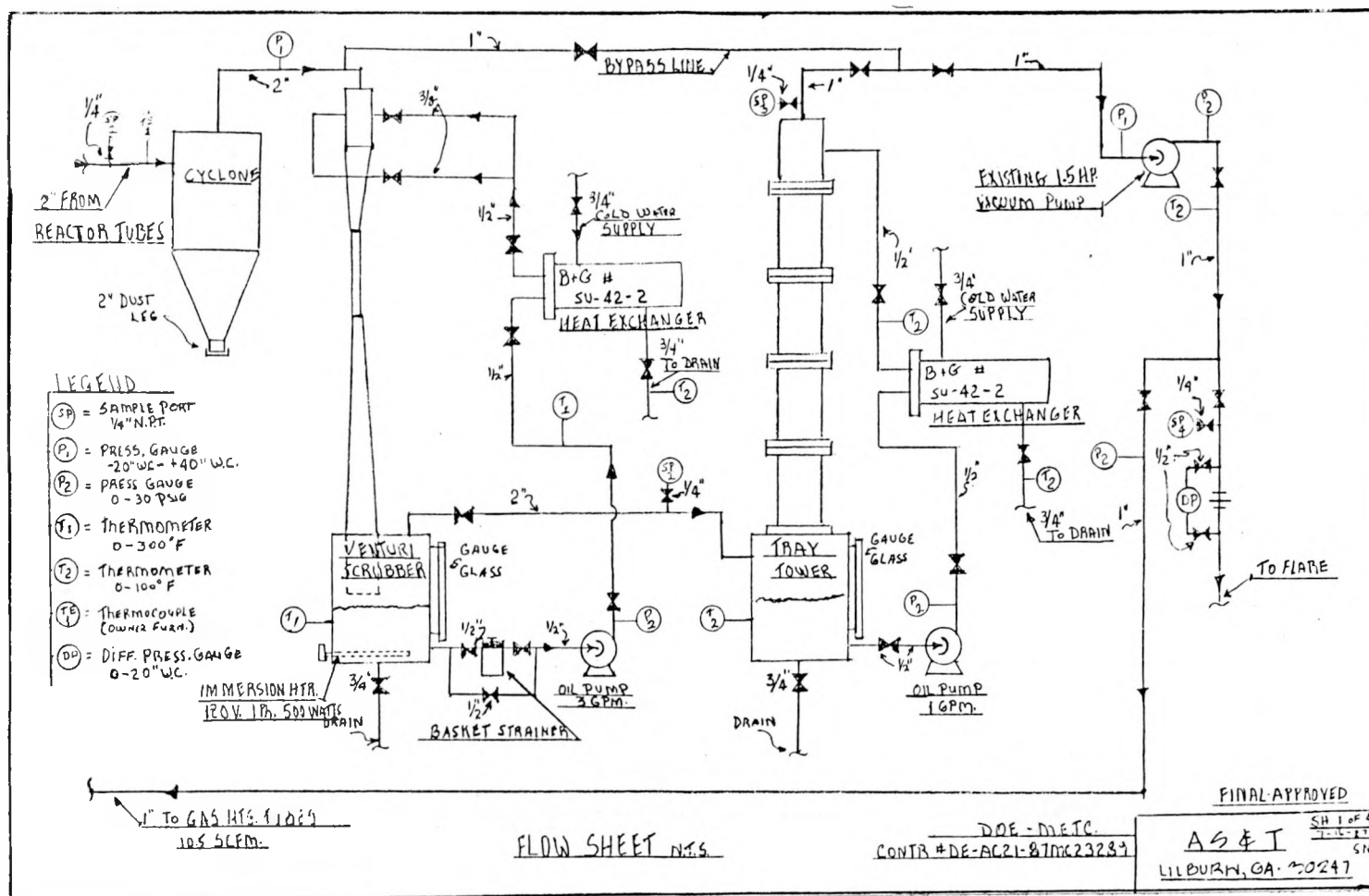


Figure 8.
Modified MGU Flowsheet

Figure 9.
Liquid Recovery System for Modified MGU

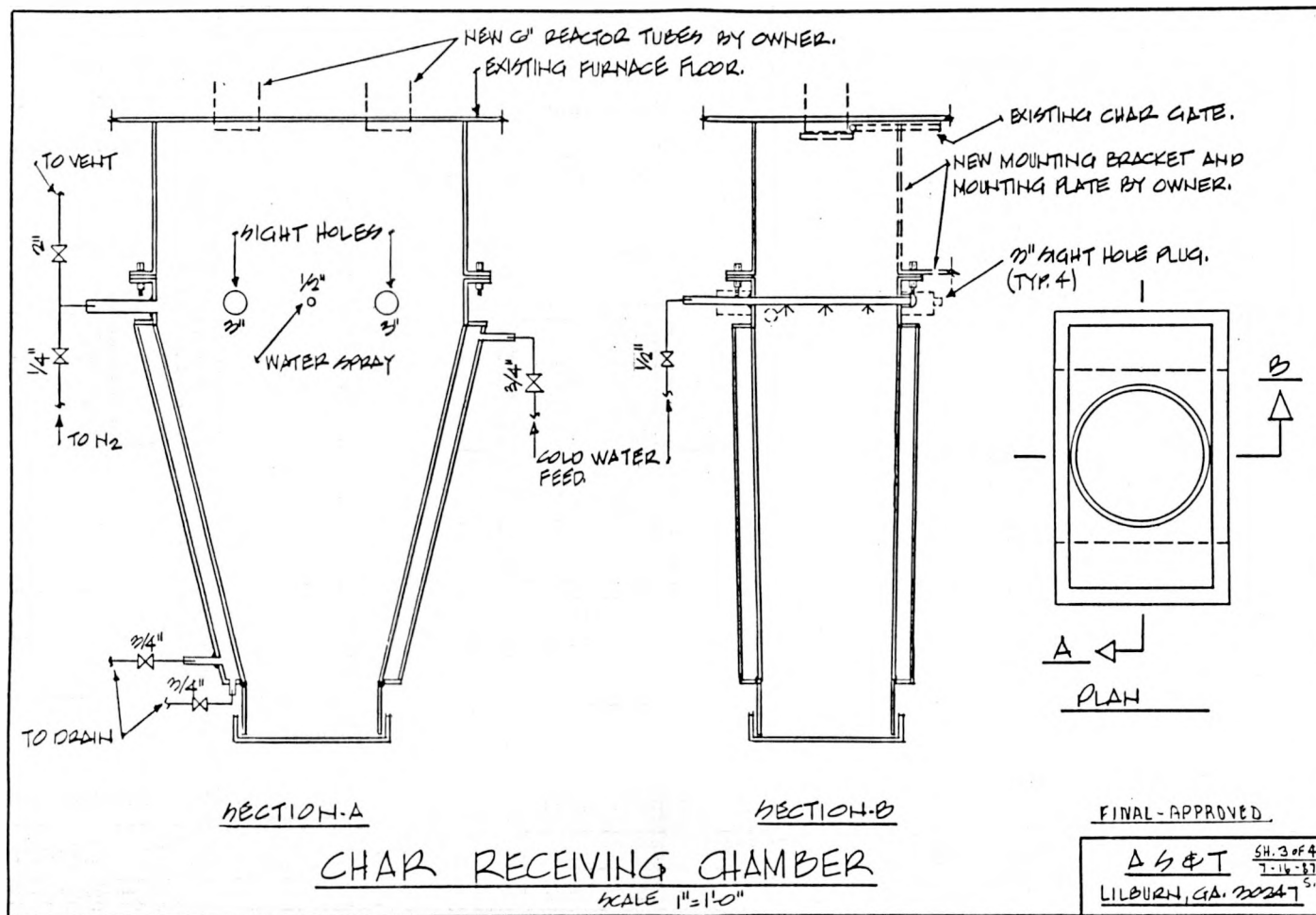


Figure 10.
Char Receiving and Quenching Chamber for Modified MGU

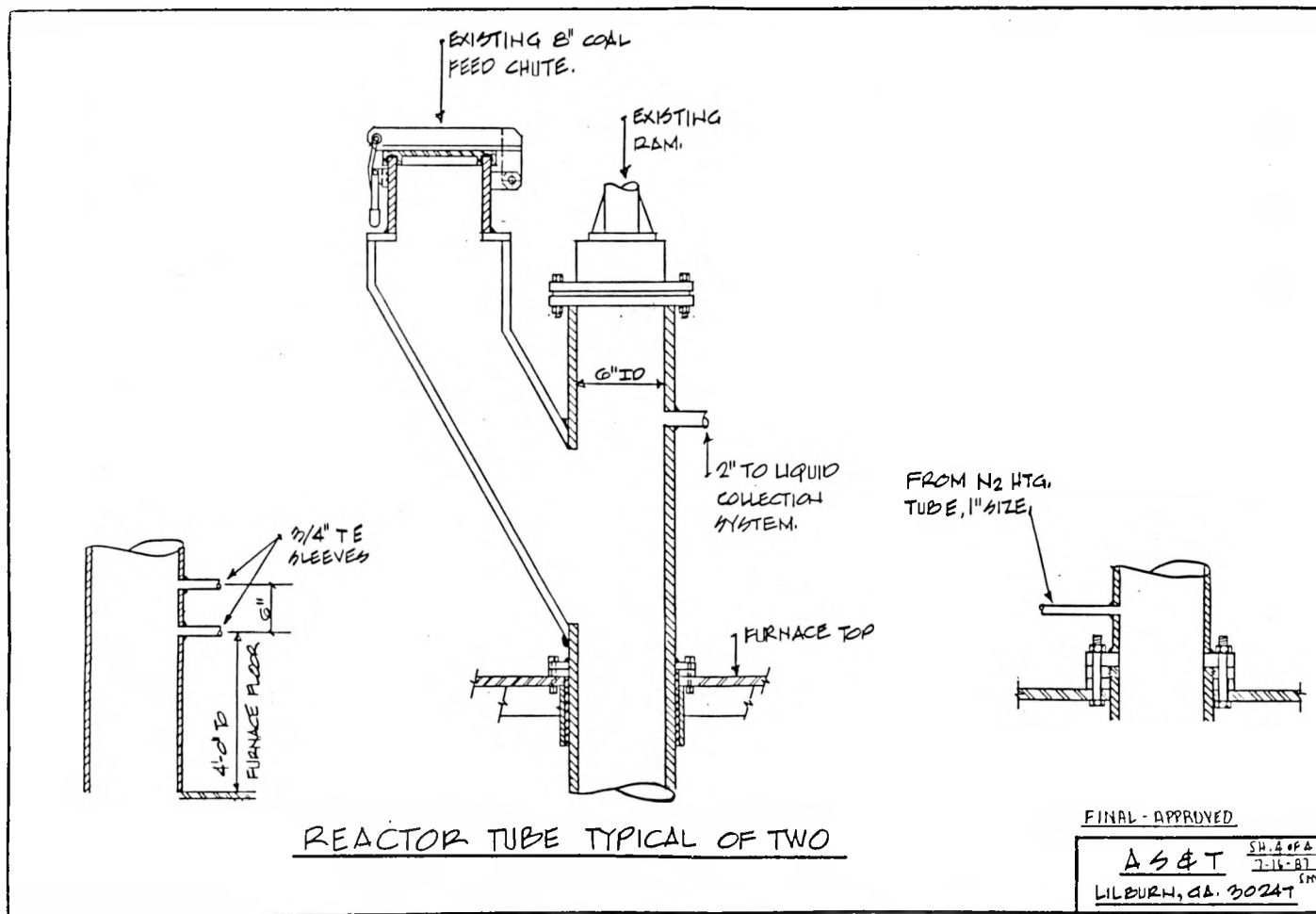


Figure 11.
Reactor Tube Details for Modified MGU

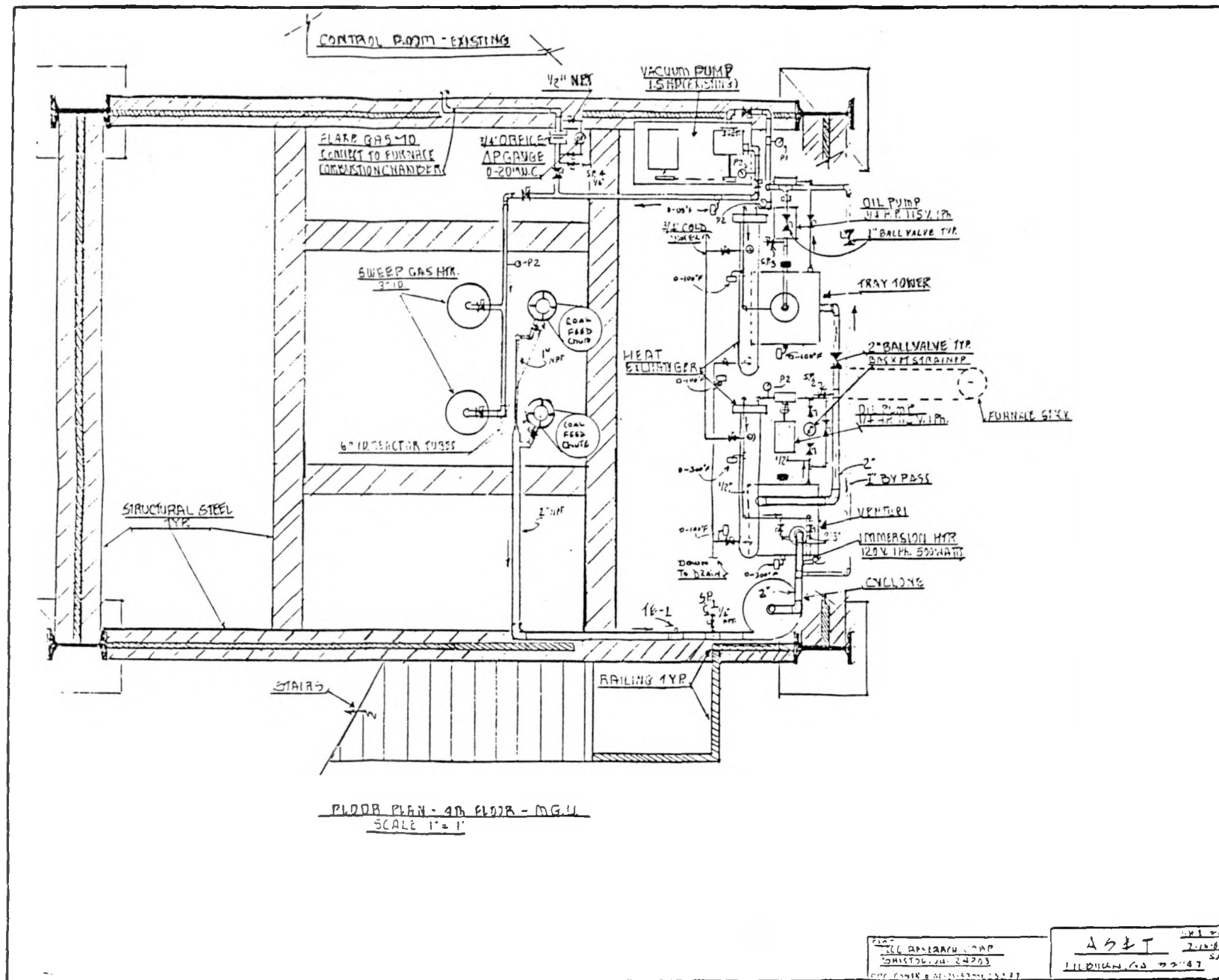


Figure 12.
Floor Plan, Third Floor of Modified MGU

Figure 13.
Equipment Layout for Third Floor of Modified MGU

reactor tubes. In addition, two 8-inch diameter stainless steel pipes (one per reactor tube) would be utilized as sweep gas heater tubes. Sweep gas will be injected at the top of the heater tubes and heated to 1000-1100°F for injection into the bottom of the 6" reactor tubes. The position of the two reactor tubes will be farthest from the oven burners while the sweep gas heater tubes will be nearest the burners (see Figure 7).

2.2.3 Mild Gasification Studies

2.2.3.1 Shake-Down Testing

The original MGU was dismantled, moved, and reassembled at CTC's new research facility in October, 1987. Work began immediately on installing the desired MGU modification features. The furnace layout, equipment layout, floor plan, flowsheet, and new components for the modified MGU are shown in Figures 2-8.

Initial shakedown testing of the modified MGU began in February, 1988. The first test was conducted using 100 lbs of Wellmore #8 bituminous coal with a furnace temperature of 1200°F and a residence time of 5 hours. The test run was terminated when the coal temperature at the center of the reactor tube reached 950°F. Approximately 20 minutes into the test, a leak developed in the new, 1-1/2 Hp vacuum pump (which served as the sweep gas recirculating pump,) causing the remainder of the test to be run without hot sweep gas. Due to the design of the vacuum pump, the leak (in the pump shaft) could not be sealed and the pump was later replaced.

The second shakedown test evaluated the operation of the hot sweep gas system and the effect of hot sweep gas on residence time. Wellmore #8 bituminous coal and a furnace temperature of 1200°F were again utilized for this test. As the leaking vacuum pump had not yet been replaced, bottled nitrogen was used as the sweeping gas. The test was terminated when the coal at the center of the reactor tube reached 950°F. The hot N₂ sweep gas reduced the residence time from the 5 hours required in Test 1 to 3 hours. After the desired maximum temperature was reached, the char was allowed to cool to ambient temperature. When char discharge was attempted, the hydraulic plungers could not push the char out of the reactor tubes. It was unclear whether this was due to the sweeping gas rate, too much coal in the reactor tubes, non-tapered reactor tubes, insufficient pressure in the hydraulic system, some other undetermined operational problem, or a combination of some or all of these. There were also problems with coal/char plugging the cross over pipes between the gas heater tubes and the reactor tubes as well as the gas line between the reactor tubes and cyclone.

Before the third shakedown test, a reconditioned 7.5 hp open drive compressor was installed to replace the leaking vacuum pump. Cold shakedown tests were conducted to insure proper operation of the compressor. Leaks around the char chamber were sealed and the latch to the char chamber door was modified in order to provide a tighter seal.

The third shakedown test was conducted to evaluate the performance of the "new" compressor. Sub-bituminous coal was used to alleviate the char discharge problem.

There continued to be problems with solids (i.e., fine coal/char and heavy tars) plugging the gas line between the reactor tubes and the cyclone. There also continued to be problems with coal/char plugging the crossover line between the gas heater tubes and the reactor tubes. It was also found that the heat distribution was uneven in the furnace.

The fourth shakedown test was conducted using lignite coal. No operational problems were observed during this tests. Although the condensible liquid yield was low (approx 4% by weight), all of the MGU components appeared to be functioning satisfactorily.

The primary objective of the fifth shakedown test was to alleviate the char sticking/discharge problems experienced in the earlier tests using Wellmore #8 bituminous coal. It was believed that this condition could be improved by reducing the rate and degree of swelling in the coal bed. This was to be accomplished by: (1) reducing the flow rate of the hot recycle/sweep gas through the coal bed and; (2) shutting off the hot recycle/sweep gas when the coal was in its plastic stage. However, during this fifth test, the hot recycle/sweep gas was inadvertently allowed to run too long and both the inner and outer regions of the bed were in the plastic stage before the sweep gas was shut off. The test was stopped and the MGU was allowed to cool. The test was resumed the following day and this time the hot sweep gas was shut off during the period that the coal was in its plastic stage. However, at the completion of the test, difficulties were still experienced with discharging the char from the reactor tubes.

During the sixth test (using Wellmore #8 bituminous coal) which was aimed at being a re-run of the fifth test, the temperature of the exit gas from the reactor tubes was observed to be unusually low (157°F). This indicated that the crossover pipes between the sweep gas heater tubes and the reactor tubes had plugged. Upon completion of the test, the hydraulic rams were able to discharge the char from one of the reactor tubes - but not the other. The char in the second tube had to be removed manually. The crossover pipes were then examined and found to be laden with char. The crossover pipes were removed, cleaned, and welded back into place.

In order to alleviate future crossover pipe plugging problems, it was decided that the bottom 12 inches of the reactor tubes (approximately 2 inches above the crossover pipe openings) would be filled with coarse gravel. This was designed to prevent the coal from migrating into the crossover tubes during the coal's plastic stage and help to more evenly disperse the hot sweep gas through the coal bed.

The objectives of the seventh test were to determine (1) if the addition of gravel would prevent the coal from entering the crossover pipes, and (2) if charging the coal while the furnace was hot would reduce the char discharging problems. The furnace was preheated to 1200°F before the coal was loaded into the reactor tubes. With a view to see if the hot sweep gas flow rate has any effect on char sticking in the reactor tubes (and to reduce particle entrainment), the rate of hot recycle/sweep gas was maintained at a much lower rate than that utilized in previous test runs (~2 vs. 10 SCFM). At the conclusion of the test, the furnace was allowed to cool over night. The following day, the hydraulic rams were not able to discharge the char from the cold reactor tubes. However, after the furnace was reheated to approximately

800°F, the char was easily discharged. It was therefore concluded that discharging the char while it was hot in all future MGU tests should greatly reduce the sticking problems experienced in the past. A liquid yield of approximately 6% by weight was obtained in the test run - approximately 2% greater than that obtained in previous test runs. The reactor off gas temperature also was found to be substantially higher than that of the past shakedown test runs, this time reaching 650°F. Filling the bottom of the reactor tubes with coarse gravel seemed to have accomplished its intended purpose, as no plugging was found in the sweep gas cross-over pipes at the completion of the test run.

This seventh test was the first test run in which the noncondensable gas stream was sampled and analyzed by the gas chromatograph. The results of the gas analysis are shown in Table 7. It can be seen from Table 7 that hydrogen and methane constitute the bulk of the noncondensable gas stream. Overall, this test run was much improved over earlier shakedown runs.

Table 7.
Gas Analysis* - MGU Shakedown Test #7

<u>SAMPLE#</u>	<u>H₂</u>	<u>CO+N₂</u>	<u>CO₂</u>	<u>H₂S</u>	<u>CH₄</u>	<u>C₂H₆</u>	<u>C₂H₄</u>	<u>C₂H₂</u>	<u>C₃H₈</u>	<u>TOTAL</u>
1	30.7	8.4	2.0	0.4	35.5	4.8	13.2	3.5	0.1	98.6
2	35.5	6.4	2.2	0.5	37.3	4.4	11.6	1.4	0.05	99.3
3	30.1	15.9	4.3	0.5	34.2	4.7	8.0	1.4	0.2	99.3
4	29.7	14.4	4.9	0.4	35.2	5.0	8.5	1.3	0.1	99.5
5	27.0	13.5	5.3	0.5	34.5	8.3	10.1	2.9	0.7	102.8

* All values are volume percentages.

<u>SAMPLE #</u>	<u>REACTION TIME</u>	<u>OUTER REACTOR TEMP.</u>	<u>INNER REACTOR TEMP.</u>
1	30 MIN	1302°F	426°F
2	69 MIN	1306°F	650°F
3	107 MIN	1299°F	840°F
4	132 MIN	1308°F	952°F
5	157 MIN	1075°F	995°F

A number of modifications were performed on the MGU during this period. An additional 6-inch flue stack was installed near the bottom of the furnace to reduce the temperature difference between the top and bottom regions within the furnace. This bottom flue stack joins the original top flue stack outside the furnace to form one single combined stack. The two flue stack dampers control the distribution of the hot flue gases between the top and bottom of the furnace and thus provide for a more uniform temperature gradient within the reactor tubes.

The gas recycle pump/compressor was dismantled and removed from the MGU and replaced with the original 3/4 horsepower vacuum pump utilized in the previous MGU contract. The frequency and severity of the seizing problems encountered with the compressor were the primary reasons for its removal. The 3/4 HP vacuum pump has a much lower capacity than the compressor, and was therefore not capable of producing a flow rate great enough to operate the MGU in "recycle" mode.

A hydraulic by-pass device was also installed in the hydraulic lines to the two reactor gates located at the bottom of the reactor tubes. In both of the two previous tests conducted on the MGU, the reactor gate supports inside the char hopper were broken by the hydraulic pressure exerted on them during the discharging of the char. The hydraulic plungers ("rams") generally require a hydraulic pressure of 800 to 1000 psi to discharge the char, which in these tests, created a force great enough to break the welds holding the reactor gate supports to the bottom of the furnace. The hydraulic by-pass allowed the rams to receive 800 to 1000 psi, while the hydraulic pressure to the reactor gates did not exceed approximately 300 psi.

A slip-stream condensing system (see Figure 14) was installed on the off-gas line downstream from the reactor tubes on the MGU. The condenser was basically a 2-stage system consisting of: (1) a 3-gallon canister which was cooled with a dry ice bath, and; (2) a 1/2-inch diameter coiled copper pipe which will be cooled by an ice and/or dry ice bath. By diverting 10 to 100% of the gas stream from the reactor bed to the slip stream condensing system, a representative coal liquid sample could be obtained from each test run.

Three gas flow meters were also installed on the MGU during this period in a move toward obtaining a material balance. One flow meter was installed in the flare gas exit line, the second in the non-condensable sweep gas recycle line, and the third in the non-condensable gas line on the slip stream condenser.

The slip-stream condensing system was designed to be capable of condensing the expected maximum liquid yield from each run. However, it was originally intended to condense only a certain percentage of the total coal gas stream, with the remaining gas going to the venturi-scrubber/tray tower. This would allow a representative liquid sample to be obtained from each run and the total coal liquid yield could then be determined by dividing the quantity of liquid collected in the slip-stream unit by the percentage of the gas stream diverted to it. However, during the first test in which the slip-stream condenser was utilized (MGU Shakedown Test #8), it was found that it could be used to condense the entire coal gas stream without upsetting the overall system pressure balance. Since this would provide the maximum liquid sample size from each test and would improve the accuracy of the liquid yield and material balance calculations, the slip-stream unit was utilized in each of remaining shakedown and parametric tests to condense the entire gas stream from the coal reactor tubes.

The material balance closure for each test was calculated in the following manner: The char yield was determined by weighing the char after discharge from the reactor tubes. The total liquid yield was determined by disconnecting and weighing the slip-stream condenser and subtracting the initial (before the test) slip-stream condenser weight.

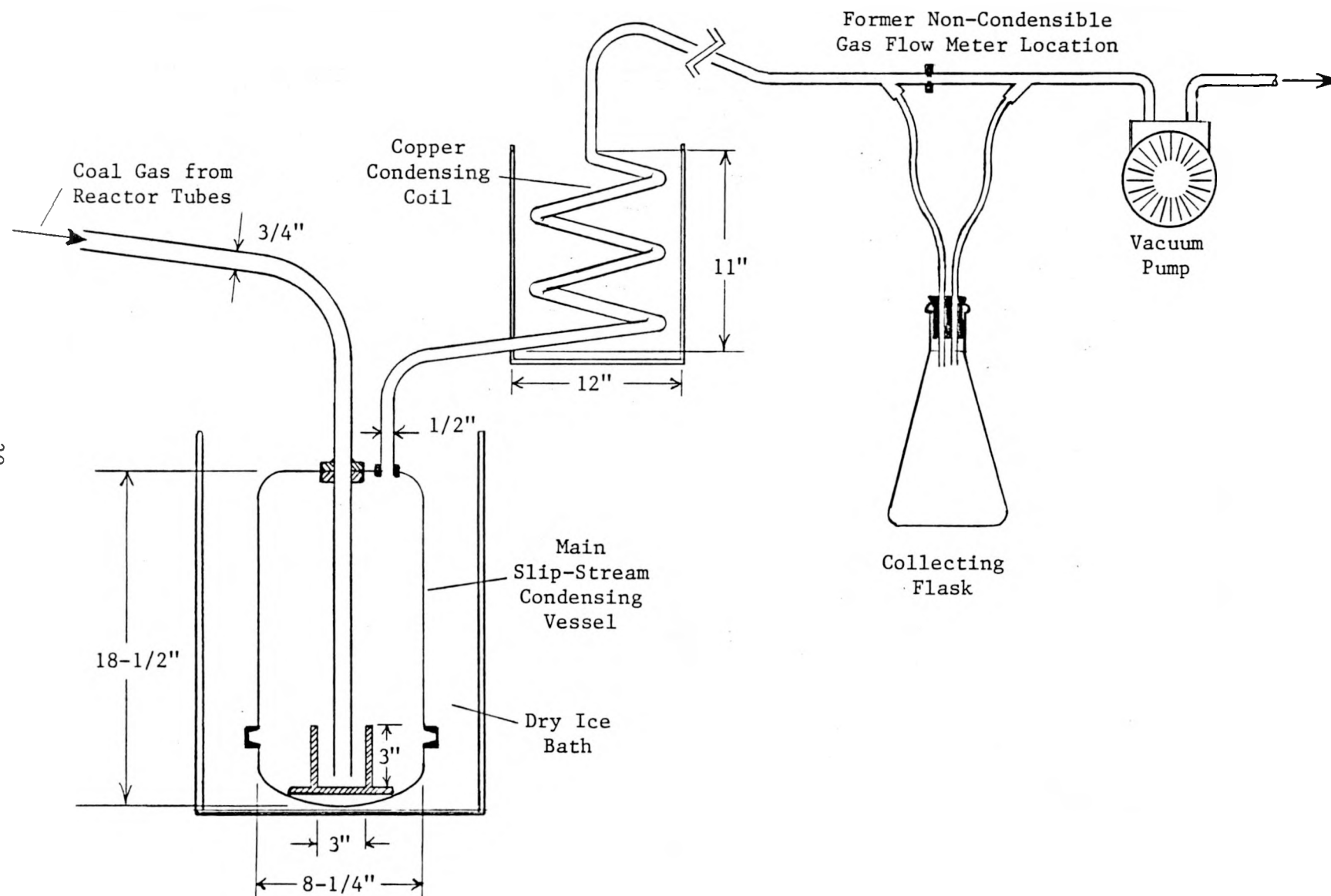


Figure 14.
Slip-Stream Condensing System

The non-condensable gas yield was determined by measuring the pressure drop across an orifice plate located in the non-condensable gas line to the flare. The temperature of the gas and the static pressure in the line were also monitored. Samples of the non-condensable gas were taken throughout each test run and analyzed by gas chromatography. The above data was used to calculate the mass flow with the following equation:

$$m = 0.61 S_o \sqrt{2 g_c (\Delta P) \rho} \quad [1]$$

where: m = Mass Flow of the Non-condensable Gas
 0.61 = Orifice Coefficient
 S_o = Surface Area of Orifice
 g_c = $32.2 \text{ lb}_m\text{-ft/lb}_f\text{-sec}^2$
 ΔP = Pressure Drop Across the Orifice
 ρ = Density of the Gas

The data from equation [1] was used to plot a curve of non-condensable gas mass flow vs. time. The area under the curve was measured to obtain the total non-condensable gas mass flow.

MGU Shakedown Test #8 was conducted using 100 pounds of 1-inch x 0 Wellmore #8 bituminous coal. The MGU was operated in vacuum mode with no sweep or recycle gas through the coal beds. After charging the coal, the oven was heated from ambient temperature to 1200°F. Very early in this run, "freeze-up" problems were encountered in the slip-stream condensing coil as the moisture was driven out of the coal bed. The dry ice bath surrounding the coil caused the water vapor to condense and freeze in the piping, thus slowly restricting and eventually preventing any flow through the condensing system. This condition was remedied by replacing the dry ice bath around the condensing coil with an ice water bath. The dry ice bath around the main condensing canister was not modified.

Test #8 was concluded after the center of the coal bed reached 900°F. The unit was allowed to cool over night and the char and liquid products were collected the following day. The total liquid yield for MGU Test #8, although improved over previous tests, was still a disappointingly low 6.5% by weight (approximately 4.0% coal liquid). The calculated material balance closure for the test was 85.6%.

The results of the gas chromatograph (GC) analysis conducted on the non-condensable gas from this test showed relatively high concentrations of hydrogen and methane (see Table 9). The concentrations of these gases and the low coal liquid yield led to the belief that significant cracking of the coal gases was occurring. It was speculated that much of the cracking was occurring in the high temperature void space above the coal bed in the reactor tubes. Methods for reducing the volume and temperature of the void space were implemented in the following test.

Table 8.
Comparison of Results From Shakedown Tests #8 - #11

Test #	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>
Oven Temp (°F)	1200	1200	1200	1200
Max Center				
Coal Temp (°F)	918	1060	1119	1084
Sweep Gas	None	N ₂	N ₂	N ₂
Char Yield (%)	65.5	74.1	71.4	71.8
Non-Condensable				
Gas Yield (%) *	13.6	13.1	10.6	14.5
Total Liquid				
Yield (%)	6.5	13.5	14.4	14.7
Oil Yield (%)	4.0	8.8	9.6	9.8
Water Yield (%)	2.5	4.7	4.7	4.9
Material Balance				
Closure (%)	85.6	100.7	96.4	101.1

* NOTE: Non-condensable gas yields were calculated by measuring the gas flow rate and composition at set intervals during each test run.

The next shakedown test (MGU Shakedown Test #9) was conducted in a manner similar to that of Shakedown Test #8, with the following exceptions:

- Approximately 1 to 2 cfm of hot nitrogen sweep gas was utilized throughout the test.
- The coal was screened to remove the minus 1/10 inch material. This was done to improve gas flow through the coal bed and reduce particulate entrainment.
- The total coal charge was increased from 100 lbs to 110 lbs to eliminate some of the void space in the upper part of the reactor tubes and hopefully reduce cracking of the coal gases.
- The upper 12 inches of the reactor tubes inside the furnace were wrapped with insulation to reduce the impact of the burner flame on the temperature of the upper portion of the reactor tubes and thereby reduce cracking potential of the coal gases.

Plugging problems were also experienced early in this test run as the moisture was being driven out of the coal bed. However this time the water froze at the point where the gases exit from the top of the dry ice cooled canister. This problem was quickly remedied by dislodging the obstruction and removing some of the dry ice from around the top of the main condensing canister.

Approximately one hour into this test, coal liquids started to appear at the orifice-type flowmeter (manometer) on the non-condensable gas line beyond the slip-stream condenser. These liquids quickly became quite significant in volume. A collection flask was connected to the manometer tubing in order to collect these liquids. A total of approximately 4 pounds of liquids were collected from the manometer location.

The test was continued until the temperature of the exit gas from the coal bed had reached its maximum (401°F) and begun to drop. The furnace was then shut off and the unit allowed to cool over night. The liquid and char products were collected the following day.

The total liquid yield for MGU Shakedown Test #9 was 14.8 pounds (13.5% by weight) - a 130% increase over the best liquid yield obtained in the eight previous shakedown tests. Although the liquid did contain some heavy tars, it appeared to be of an overall better quality than those obtained in any of the MGU tests conducted previously. The results of the elemental analyses conducted on the coal liquid and char samples are given in Tables 10 and 11. The total coal liquid yield (after water decantation) for this test was 8.8% (9.7 lbs). The material balance closure (liquid + char + non-condensable gas) was 100.7%.

It was quickly decided to conduct another MGU test to try to duplicate and substantiate the results obtained in Shakedown Test #9. Shakedown Test #10 was conducted using the same coal and procedures that were utilized in Test #9. The total liquid yield obtained in MGU Test #10 was slightly higher than that obtained in Test #9 - 15.8 pounds (14.4% by weight). The quality of the coal liquids appeared to be nearly identical to that from Test #9; however there did appear to be more heavy tars in the liquid and condenser piping. The results of the elemental analyses conducted on the coal liquid and char samples are given in Tables 10 and 11. The total coal liquid yield was 9.6% (10.6 lbs). The material balance closure was 96.4%.

In an effort to further increase the coal liquid yield, the coal feedstock was changed from Wellmore #8 to H&K Williamson #2 Seam. This coal was believed to be slightly higher in volatile matter content than Wellmore #8. The same test procedures followed in the two previous tests were utilized in Shakedown Test #11. The slip-stream condenser "freeze-up" was avoided in this test by initially placing only a very small amount of dry ice in the condenser bath while the moisture was being driven out of the coal bed. Once the temperature of the gases entering the condenser had increased sufficiently, the remaining dry ice was added to the bath. All other test procedures were carried out as closely as possible to those followed in the previous two tests.

The total liquid yield obtained in Shakedown Test #11 was 16.2 pounds (14.7% by weight). The liquids appeared to be of roughly the same quality as those obtained in the two previous tests. The results of the elemental analyses conducted on the coal liquid and char samples are given in Tables 10 and 11. The total coal liquid yield was 9.8% (10.8 lbs). The material balance closure obtained was 101.1%.

Table 9.
Gas Analysis - MGU Shakedown Tests

SAMPLE#	ELAPSED TIME	INNER REACTOR	OUTER REACTOR	VOLUME PERCENTAGE						
		TEMP °F	TEMP °F	H ₂	CO [*]	CO ₂	H ₂ S	CH ₄	C ₂ H ₆	C ₂ H ₄ + C ₂ H ₂
TEST #8 (No sweep gas used.)										
1	245 min	835°	1190°	30.2	10.1	2.4	1.2	33.1	8.4	13.2
TEST #9 (Nitrogen sweep gas subtracted.)										
1	39 min	245°	950°	8.2	3.5	17.6	-	57.3	14.0	-
2	77	458	1139	21.4	0.8	5.3	-	47.9	11.6	7.2
3	157	670	1150	23.2	-	4.9	-	47.3	11.4	8.7
4	185	695	1151	25.9	0.6	4.5	-	44.7	10.8	9.2
5	239	854	1160	27.1	0.6	4.0	-	43.3	10.3	9.3
6	278	884	1160	30.7	0.6	4.3	-	44.0	9.5	9.0
7	324	977	1171	45.8	1.0	-	-	53.2	-	-
TEST #10 (Nitrogen sweep gas subtracted.)										
1	82 min	565°	1153°	22.7	0.5	4.3	-	45.3	11.0	8.6
2	127	700	1162	25.0	0.4	4.4	-	45.0	10.5	9.6
3	162	801	1164	26.1	0.6	3.8	-	45.1	9.6	9.8
4	215	888	1169	27.7	0.5	5.8	-	42.2	9.4	8.9
5	262	985	1181	43.2	4.7	-	-	52.1	-	-
6	307	1057	1178	52.4	0.9	4.7	-	40.7	1.3	-
7	340	1112	1173	60.2	0.8	5.5	-	33.6	-	-
TEST #11 (Nitrogen sweep gas subtracted.)										
1	48 min	299°	1067°	15.5	0.6	7.4	6.7	53.8	14.0	2.1
2	80	589	1143	23.1	0.6	5.5	6.1	48.5	11.4	4.9
3	111	613	1151	25.6	-	5.1	4.4	45.9	10.9	8.1
4	165	806	1153	29.5	0.5	4.2	-	46.6	10.2	9.0
5	190	835	1158	28.7	0.5	3.3	5.4	44.1	9.8	8.2
6	236	924	1159	33.9	0.6	3.9	6.8	43.1	6.8	4.9
7	283	1012	1164	47.8	0.6	4.8	-	41.3	3.8	1.8
8	316	1077	1166	59.5	-	5.1	-	35.4	-	-

* NOTE: Values shown for CO concentration also include N₂.

Table 10.

Results of Analytical Tests Conducted on MGU Coal Liquid Samples
MGU Shakedown Tests #8 - #11

	Test <u>8</u>	Test <u>9</u>	Test <u>10</u>	Test <u>11</u>
MOLECULAR				
WEIGHT *	256	237	241	256
% MOISTURE	0.95	1.44	0.79	2.12
% CARBON	80.59	81.59	84.82	82.20
% HYDROGEN	6.94	7.03	6.69	7.14
% NITROGEN	1.32	0.86	0.90	1.05
% SULFUR	0.03	0.89	0.90	0.71
% OXYGEN	5.82	10.69	7.30	9.77
BTU/lb	15,285	15,246	13,771	15,450
H/C ATOMIC				
RATIO	1.03	1.03	0.94	1.04

* NOTE: All values except molecular weight and % moisture are reported on a dry (moisture free) basis.
All analyses were conducted by Galbraith Laboratories, Inc of Knoxville, Tennessee.

Table 11.

Results of Analytical Tests Conducted on MGU Char and Coal Samples
MGU Shakedown Tests #9 - #11

	Wellmore <u>#8 Coal</u>	Test 9 <u>Char</u>	Test 10 <u>Char</u>	H&K Coal	Test 11 <u>Char</u>
% MOISTURE	1.32	2.51	2.43	1.88	0.90
% ASH	8.71	10.26	9.73	5.00	4.93
% VOLATILE	32.73	7.15	6.34	33.80	6.37
% FIXED CARBON	57.24	80.08	81.50	59.40	87.80
% CARBON	79.58	79.67	83.27	78.18	87.69
% HYDROGEN	5.44	2.60	2.52	5.24	2.32
% NITROGEN	1.19	1.57	1.72	1.46	1.40
% SULFUR	1.54	1.07	1.13	0.98	0.76
% CHLORINE	0.12	0.04	0.05	0.13	0.05
% OXYGEN *	3.42	4.79	1.58	9.01	2.85
Btu/lb	14,059	N/A	14,051	14,876	13,941

* NOTE: Oxygen values are calculated "by difference".

2.2.3.2 Parametric Testing

The parametric testing program was initiated immediately following MGU Test #11. The objective of the first four parametric tests was to investigate the effect of temperature on the quantity and quality of coal liquids produced by the MGU. Coal temperatures ranging from 900° to 1200°F were utilized for these tests. In each case, the oven was set at a temperature 100°F higher than the maximum desired center coal bed temperature. The MGU was operated in vacuum mode with no sweep or recycle gas through the bed. Each of these tests was conducted using 110 pounds of 1-1/2 inch x 0 H&K (Williamson #2 Seam) bituminous coal. A summary of the results obtained in these first four parametric tests is given in Table 12.

As can be seen in Table 7, the total liquid yield obtained in MGU Test #12/P1 was 12.5% (8.4% oil + 4.1% water). Although this liquid yield was slightly less than that obtained in the three previous MGU tests (#9-11), the coal liquid obtained from this test was lighter in color and lower in density than any coal liquids produced previously in the MGU. The quality of this coal liquid was in fact better than that of the raw coal liquid obtained from COALITE's commercial mild gasification plant in England (see Table 14). The MGU 12/P1 liquid not only has a higher H/C atomic ratio (1.36 vs. 1.22) and lower average molecular weight (lighter) than the COALITE liquid, but it also has substantially lower hetero-atom (S, N, and O) contents. The distillation curves (see Figure 15) show that the CTC MGU liquid yields approximately 14% naphtha (includes minor amount of water), 80% diesel material, and 6% heavy ends, by volume. The corresponding values for the COALITE liquid are 5%, 55%, and 40% respectively. These data indicate that the CTC MGU 12/P1 liquid is superior to the COALITE liquid in quality and will yield substantially more light and useful transportation type fuels.

In addition to the improved liquid quality obtained in MGU Test #12/P1, the char (78.2% by weight) also contained more volatile matter than the chars obtained in previous tests. The increased volatile matter and relatively low ash content should improve the combustion characteristics of the char as well (see Table 15).

The 100°F temperature increase in the second parametric test (#13/P2) did increase the liquid yield (14.1%) over that obtained in the first parametric test. However, the quality of the coal liquid was somewhat reduced as evidenced by the blacker color and higher viscosity and density (Test #13/P2 liquids were heavier than water whereas those from Test #12/P1 were lighter than water).

The coal liquids obtained in Tests #14/P3 and #15/P4 were progressively heavier and thicker than those from Test #13/P2. The data shows that as the reaction temperature increases, more and heavier tars are volatilized and condensed, as evidenced by the increase in molecular weight and decrease in the H/C atomic ratio (see Table 14). The volatile content of the char product also decreases with increasing temperature, which will hinder its initial combustibility (see Table 15).

Table 12.
Comparison of Results from Parametric Tests P1 - P12

TEST #	12/P1	13/P2	14/P3	15/P4	16/P5	17/P6	18/P7	19/P8	20/P9	21/P10	22/P11	23/P12
COAL TYPE	H&K Bit.	H&K Bit.	H&K Bit.	H&K Bit.	H&K Bit.	H&K Fresh	H&K Bit.	H&K Bit.	H&K Bit.	H&K Bit.	Sub- Bit.	Lignite
PARTICLE SIZE (inches)	1- $\frac{1}{2}$ x 0	1- $\frac{1}{2}$ x 0	1- $\frac{1}{2}$ x 0	1- $\frac{1}{2}$ x 0	1- $\frac{1}{2}$ x 0	1- $\frac{1}{2}$ x 0	1- $\frac{1}{2}$ x 0	$\frac{1}{8}$ x 0	$\frac{1}{8}$ x 0	$\frac{1}{8}$ x 0	$\frac{1}{2}$ x 0	$\frac{3}{4}$ x 0
ADDITIVE	None	None	None	None	None	None	None	None	10% Lime	20% Lime	None	None
OVEN TEMP (°F)	1000	1100	1200	1300	1000	1000	1000	1000	1000	1000	1000	1000
MAX CENTER COAL TEMP (°F)	893	1000	1113	1228	903	906	901	898	901	899	903	645*
SWEEP GAS	None	None	None	None	None	None	N ₂	None	None	None	None	None
CHAR YIELD (%)	78.2	71.8	74.0	70.4	77.3	79.2	74.6	79.1	78.9	81.0	58.6	43.6
NON-CONDENSIBLE GAS YIELD (%) *	7.8	10.6	13.5	15.2	8.6	6.3	N/A	6.7	7.2	6.5	12.1	10.5
TOTAL LIQUID YIELD (%)	12.5	14.1	13.1	13.3	13.9	15.0	16.1	13.3	14.4	11.4	32.2	36.0
OIL YIELD (%)	8.4	9.1	8.2	7.8	9.1	10.0	10.7	9.6	10.8	6.6	3.3	4.8
WATER YIELD (%)	4.1	5.0	4.9	5.5	4.8	4.9	5.5	3.7	3.7	4.8	28.9	31.2
MATERIAL BALANCE CLOSURE (%)	98.5	96.5	100.6	98.9	99.8	100.5	N/A	99.1	100.5	98.9	102.9	90.1

* NOTE: Non-condensable gas yields were calculated by measuring the gas flow rate and composition at set intervals during each test run.
In MGU Test #23/P12, the center thermocouple did not reach the desired 900° F temperature.
It is believed that a malfunction in the thermocouple wiring was responsible for this problem.

It should be noted that the duration of the first four parametric tests were progressively shorter as the temperature increased (see Figure 16). A temperature increase of 100°F resulted in approximately a 40 minute reduction in the time required to reach the desired temperature (i.e., 420 min - 900°F, 380 min - 1000°F, 340 min - 1100°F, and 290 min - 1200°F). The improved liquid quality experienced at the lower temperatures may be attributable in part to the increased time that the coal spends in its plastic stage. Also, the condensible and non-condensable gas flow rates increased nearly proportionally with the increase in temperature.

Table 8 contains the results of the gas chromatograph (GC) analyses conducted on the non-condensable gas samples taken during each of the first eleven parametric tests. As can be seen from the results, the hydrogen, acetylene, and ethylene contents of the gas increase with increasing temperature. The ethane and hydrogen sulfide contents appear to decrease with increasing temperature. As shown in Table 7, the total non-condensable gas yield also increases with increasing temperature.

The fifth parametric test (#16/P5) was conducted following the same procedures utilized in Test #12/P1 in an attempt to check the reproducibility of the earlier results. The total liquid and oil yields were somewhat greater than those obtained in Test #12/P1 (13.9% vs. 12.5% and 9.1% vs. 8.1%, respectively). The quality of the 16/P5 liquids appeared to be very similar to that of the liquids from Test #12/P1, as the color was only slightly darker and the H/C ratio and Btu/lb values were slightly lower (see Table 14). There was a suspicion of an air leak around the char hopper door as the GC analyses of the non-condensable gas indicated higher than normal levels of air in the samples. Also, no liquids condensed inside the char hopper - a small amount of liquid has condensed in the char hopper in every MGU test except those which incorporated nitrogen sweep gas.

MGU Test #17/P6 was also conducted using a 1000°F furnace temperature and 900°F center coal bed temperature, however freshly mined (approx 2 days prior) H&K Williamson #2 coal was utilized. The coal liquid yield was higher than that obtained in any of the previous parametric tests (10.1% oil). On a comparable basis, this yield also was, to some degree, higher than that of either Test #12/P1 or #16/P5 (10.1% vs. 8.4% for #12/P1 & 9.1% for #16/P5), which were carried out under the same operating conditions, but with H&K coal of longer storage time (~6 months). The lower oxygen content of the "fresh" coal (4.99% vs. 9.01% - see Table 16) seems to reflect the lower weathering effect and thus may be responsible for this higher liquid yield. The liquids had a green tint - very similar to that of the liquids from Test #12/P1 (also see Table 14).

All of the following parametric tests were conducted using a 1000°F furnace temperature and 900°F center coal bed temperature. Although the data collected from the previous parametric tests has shown that this temperature does not produce the greatest quantity of liquids, it does produce the lightest and best quality coal liquids.

Test #18/P7 was conducted using nitrogen sweep gas. The sweep gas rate was adjusted throughout the test to prevent excessive pressure build-up in the lower part of the reactor tubes and char hopper area. Even though the sweep gas rates utilized during this test were relatively low (0.5 to 1.5 cfm), once the center bed temperature reached approximately 750°F, virtually none of the sweep gas was passing through the coal bed, and the nitrogen sweep was shut off.

The total liquid yield obtained in Test #18/P7 was 16.1% (10.7% oil). These liquids also had a green tint and appeared to be somewhat lighter than those from Test #17/P6 (see Table 14). The nitrogen sweep gas did prevent the coal gases from entering and condensing inside the char hopper, and appears to have somewhat improved the quantity and quality of the coal liquids collected (see Table 12).

Table 13.
Gas Analysis - MGU Parametric Tests

SAMPLE #	ELAPSED TIME	INNER REACTOR TEMP °F	OUTER REACTOR TEMP °F	VOLUME PERCENTAGE						
				H ₂	CO [*]	CO ₂	H ₂ S	CH ₄	C ₂ H ₆	C ₂ H ₄ + C ₂ H ₂
TEST #12/P1										
1	63 min	365°	919°	14.2	3.2	10.7	1.5	50.1	17.9	2.4
2	95	462	937	15.7	2.5	8.0	1.9	51.7	17.6	2.7
3	158	615	944	19.0	5.8	6.0	2.2	54.3	17.2	3.0
4	220	721	951	22.1	3.4	4.0	2.1	57.6	17.3	2.8
5	283	791	954	23.8	2.3	4.0	3.4	60.7	17.6	2.0
6	364	869	952	27.6	2.0	4.1	1.8	60.8	15.0	1.6
7	398	889	951	29.7	1.7	4.4	2.2	60.4	12.9	1.5
TEST #13/P2										
1	73 min	332°	1050°	18.7	2.8	6.4	1.6	50.4	15.3	4.9
2	139	568	1060	21.3	2.5	4.8	1.5	50.8	14.8	6.9
3	199	748	1064	24.4	2.4	3.9	2.0	53.2	14.4	6.1
4	262	870	1070	25.9	2.8	4.0	1.5	52.0	13.0	5.4
5	323	950	1071	30.1	2.8	4.8	0.9	53.7	10.6	4.2
6	382	999	893	31.2	3.6	5.9	0.5	56.7	6.5	1.0
TEST #14/P3										
1	52 min	340°	-	19.9	3.5	6.2	1.3	53.0	12.4	4.6
2	112	620	-	27.4	3.1	5.5	1.3	51.1	12.1	7.6
3	172	807	-	33.5	3.2	5.7	1.2	50.9	11.5	7.7
4	232	908	1155°	25.4	2.5	4.0	1.0	44.8	10.9	7.4
5	292	1025	1173	36.4	3.2	5.9	0.7	43.3	5.3	3.0
6	343	1111	973	42.3	4.3	5.4	0.3	46.2	2.5	0.5
TEST #15/P4										
1	47 min	387°	1092°	23.2	3.5	5.8	1.4	57.0	15.1	4.5
2	105	663	1249	33.4	3.6	3.6	0.5	50.0	9.0	10.5
3	179	879	1258	35.8	3.7	3.5	0.9	50.3	8.5	10.5
4	229	1005	1261	41.3	3.6	3.3	0.6	46.1	5.6	6.0
5	274	1198	1264	57.3	3.5	3.8	0.2	31.9	2.5	2.1
6	303	1208	989	55.3	4.6	7.3	-	37.4	1.3	0.4
TEST #16/P5										
1	46 min	247°	910°	11.1	-	23.1	1.7	61.3	24.1	4.0
2	76	363	930	14.5	4.6	15.0	2.3	58.1	20.8	3.0
3	121	503	944	17.0	3.8	11.4	3.7	56.1	19.0	3.3
4	181	606	951	19.8	3.6	8.9	2.4	57.0	18.6	3.1
5	241	761	960	22.6	3.5	5.1	3.1	61.1	22.2	2.7
6	301	789	964	24.6	3.2	3.4	2.3	62.7	18.1	2.2
7	361	830	966	28.1	3.4	3.2	1.8	61.4	18.8	1.8
8	425	903	848	28.9	3.9	3.2	1.0	64.0	13.7	0.9
TEST #17/P6										
1	35 min	222°	846°	1.6	-	24.9	-	8.2	3.3	0.4
2	68	330	914	12.8	5.4	11.1	2.2	51.4	18.6	2.3
3	128	527	943	18.2	4.7	5.9	2.5	55.5	18.2	3.1
4	251	788	945	22.6	3.8	3.1	2.7	61.6	18.6	2.5
5	308	825	943	22.8	3.9	2.8	2.4	59.4	19.9	2.0

* NOTE: Value shown for CO concentration also includes N₂.

Table 13. (continued)
Gas Analysis - MGU Parametric Tests

SAMPLE #	ELAPSED TIME	INNER REACTOR TEMP °F	OUTER REACTOR TEMP °F	VOLUME PERCENTAGE						
				H ₂	CO [*]	CO ₂	H ₂ S	CH ₄	C ₂ H ₆	C ₂ H ₄ + C ₂ H ₂
TEST #18/P7										
1	60 min	272°	922°	-	-	14.3	-	69.5	16.1	-
2	135	583	943	14.5	1.6	6.3	-	47.1	29.9	-
3	196	697	948	18.8	1.6	7.4	-	54.9	13.2	2.0
4	285	793	952	20.6	1.0	3.2	7.7	53.0	12.8	1.1
TEST #19/P8										
1	55 min	186°	296°	16.4	7.9	11.5	2.0	60.2	20.4	2.3
2	115	451	948	21.3	7.1	9.7	2.1	60.6	18.5	2.0
3	190	610	963	27.8	-	7.5	2.6	68.5	20.6	1.9
4	250	727	957	26.2	5.0	6.4	1.9	61.0	16.5	1.9
5	310	800	959	27.4	4.6	4.5	2.3	63.1	20.7	2.0
6	385	867	959	31.6	5.1	3.3	1.6	62.5	13.8	1.5
TEST #20/P9										
1	60 min	357°	932°	18.3	7.3	0.2	0.0	59.4	20.3	2.7
2	104	489	942	22.4	6.3	0.3	0.0	57.2	17.9	2.2
3	149	578	946	16.8	3.7	0.2	0.0	44.3	13.8	1.5
4	194	655	950	26.2	5.1	0.3	0.0	58.4	16.8	1.8
5	239	726	952	27.9	3.8	0.4	0.0	60.2	16.7	1.7
6	284	782	955	27.7	3.5	0.4	2.1	58.2	15.6	1.6
7	329	826	957	30.5	3.1	0.4	0.0	61.1	14.7	1.3
8	375	864	959	33.9	2.9	0.4	0.0	61.0	12.3	1.1
9	418	896	959	36.1	2.9	0.4	0.0	58.9	10.5	0.9
10	435	901	840	33.6	4.0	0.2	0.0	61.5	11.5	0.7
TEST #21/P10										
1	115 min	403°	887°	19.1	7.2	0.0	0.0	58.6	21.2	2.6
2A	158	521	901	23.0	1.5	0.0	0.0	63.0	21.3	2.3
2B	158	521	901	23.0	1.4	0.0	0.0	63.7	21.5	2.3
3	235	668	906	24.9	4.2	0.0	0.0	58.8	18.6	1.7
4	310	762	909	26.9	3.2	0.0	0.0	59.8	18.0	1.5
5	386	821	917	29.7	2.5	0.0	0.0	61.4	16.1	1.2
6	430	856	959	34.9	1.9	0.0	0.0	59.8	12.7	1.0
7	475	885	960	38.1	1.7	0.0	0.0	59.8	11.3	0.8
TEST #22/P11										
1	30 min	270°	796°	0.0	14.4	90.9	0.0	3.9	1.1	0.6
2	60	244	911	4.7	10.8	61.3	0.4	21.1	7.0	2.0
3	90	266	926	8.5	10.6	54.0	0.4	24.5	7.3	2.0
4	122	292	933	11.1	9.4	50.7	0.5	25.2	7.3	2.1
5	150	303	938	12.6	9.4	49.6	0.3	26.3	7.5	2.7
6	212	471	949	15.0	8.4	45.6	0.4	28.2	7.9	2.4
7	270	571	952	16.8	7.7	43.5	0.4	30.0	8.3	2.7
8	345	743	959	17.5	6.7	40.1	0.4	31.3	8.7	3.0
9	391	843	957	16.0	6.9	33.3	0.4	30.5	8.7	3.0
10	450	901	898	19.7	6.4	33.0	0.4	40.2	10.3	1.5

* NOTE: Value shown for CO concentration also includes N₂.

Table 14.
Results of Analytical Tests Conducted on MGU Coal Liquid Samples

	<u>COALITE</u>	<u>12/P1</u>	<u>13/P2</u>	<u>14/P3</u>	<u>15/P4</u>	<u>16/P5</u>	<u>17/P6</u>	<u>18/P7</u>	<u>19/P8</u>	<u>20/P9</u>	<u>21/P10</u>	<u>22/P11</u>
MOLECULAR												
WEIGHT *	218	215	291	341	334	242	230	201	198	206	259	234
% MOISTURE	1.57	1.10	7.66	11.92	2.44	0.64	0.56	0.76	0.79	0.62	0.45	0.77
% CARBON	84.49	84.87	87.77	91.67	85.61	85.17	84.87	84.71	85.03	85.60	86.30	85.59
% HYDROGEN	8.66	9.69	8.31	7.21	6.67	9.09	9.08	8.93	9.71	9.44	9.95	8.69
% NITROGEN	1.03	0.43	1.16	1.14	1.17	0.70	0.59	0.40	0.68	0.65	0.34	0.55
% SULFUR	0.97	0.49	0.63	0.70	0.68	0.55	0.54	0.57	0.55	0.50	0.42	0.41
% OXYGEN	6.25	5.38	10.56	13.59	8.96	5.52	5.53	5.29	5.17	3.47	3.26	5.13
BTU/lb	N/A	16,924	16,647	16,618	15,843	16,479	17,115	16,973	17,120	17,398	17,749	17,063
H/C ATOMIC												
RATIO	1.22	1.36	1.13	0.94	0.93	1.27	1.27	1.26	1.36	1.31	1.37	1.21

* NOTE: All values except molecular weight and % moisture are reported on a dry (moisture free) basis.
Analytical data on coal liquids from MGU test #23/P12 are not available at this writing.
All analyses were conducted by Galbraith Laboratories, Inc of Knoxville, Tennessee.

Table 15.
Results of Analytical Tests Conducted on MGU Char Samples

	<u>12/P1</u>	<u>13/P2</u>	<u>14/P3</u>	<u>15/P4</u>	<u>16/P5</u>	<u>17/P6</u>	<u>18/P7</u>	<u>19/P8</u>	<u>20/P9</u>	<u>21/P10</u>	<u>22/P11</u>
% MOISTURE	1.26	0.92	2.22	0.72	0.80	0.84	1.32	0.99	0.52	3.72	5.59
% ASH	6.11	8.73	6.99	6.51	5.61	6.20	5.40	5.55	14.25	21.05	4.96
% VOLATILE	11.17	8.80	7.30	4.56	11.17	10.49	10.90	11.86	13.08	15.90	21.19
% FIXED CARBON	81.46	81.55	83.49	88.21	82.42	82.47	82.38	81.68	72.15	59.33	68.26
% CARBON	85.27	83.30	83.31	88.00	84.68	85.96	85.11	85.98	77.87	63.64	76.70
% HYDROGEN	3.37	2.72	2.62	2.02	3.59	3.35	3.16	3.32	2.73	3.18	3.73
% NITROGEN	1.29	1.60	1.66	2.06	1.69	1.44	1.67	1.58	1.68	1.20	0.92
% SULFUR	0.90	0.79	0.97	0.75	0.80	0.90	0.08	0.66	0.89	0.01	0.25
% CHLORINE	0.10	0.07	0.06	0.05	0.08	0.06	0.69	0.10	0.10	0.001	0.23
% OXYGEN *	2.96	2.79	4.39	0.61	3.55	2.09	3.89	2.81	2.48	10.91	13.23
BTU/lb	13,204	14,114	14,045	13,974	14,302	13,854	14,043	14,000	12,569	11,636	13,152

* NOTE: Oxygen values are calculated "by difference".

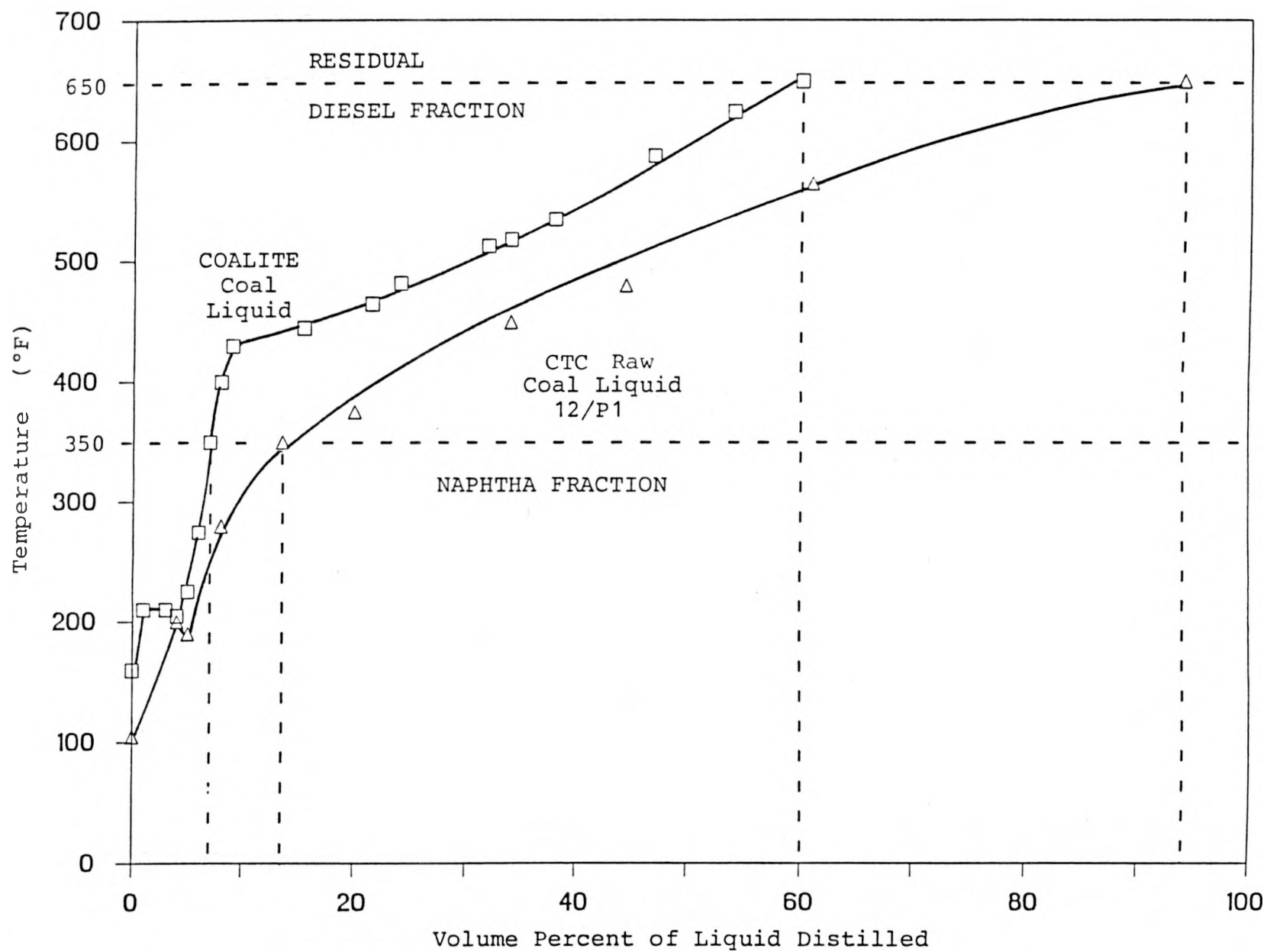


Figure 15.
Distillation Curve Comparison of
CTC MGU 12/P1 Raw Coal Liquid and Coalite Raw Coal Liquid

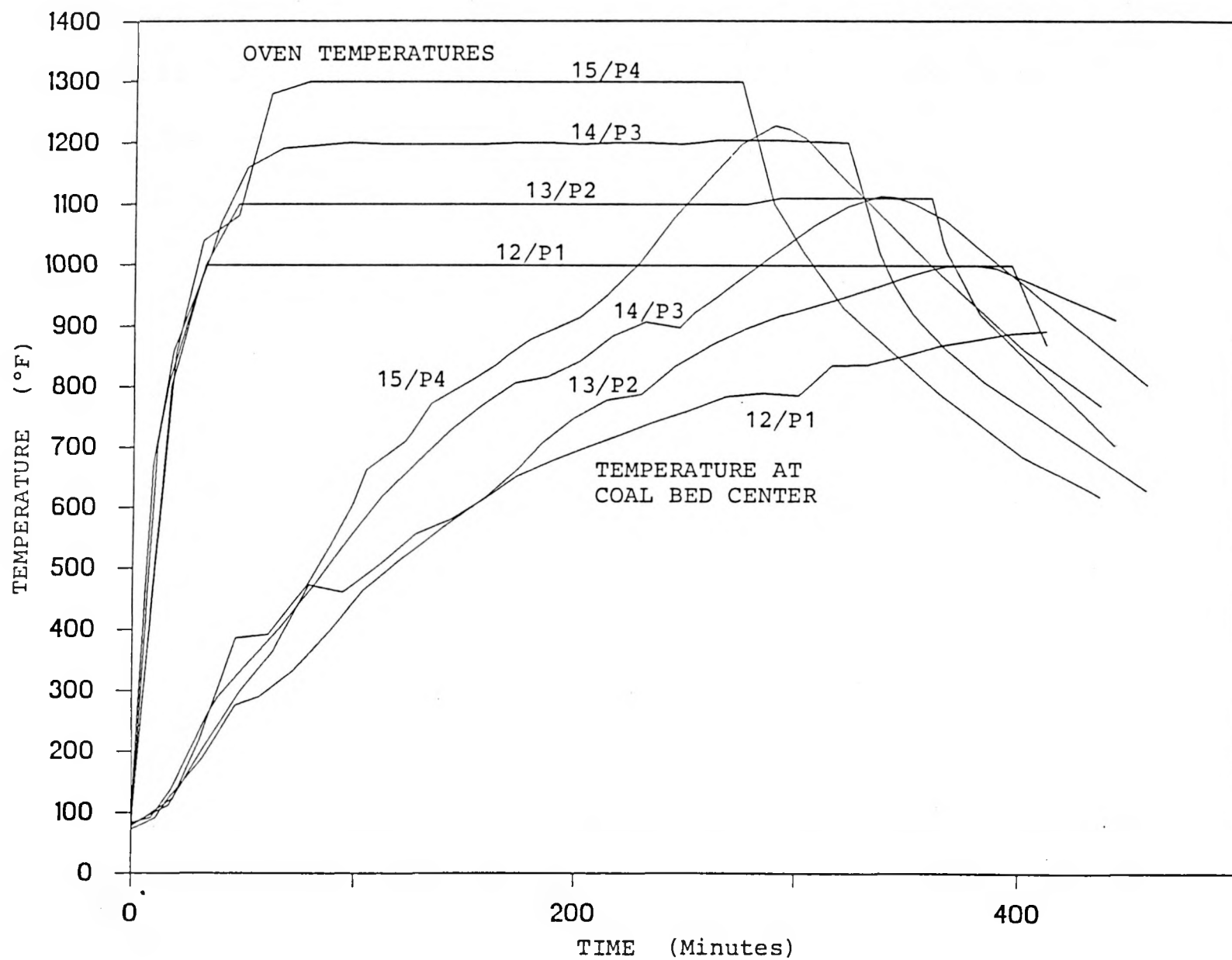


Figure 16.
Comparison of Furnace and Coal Bed Center Temperatures

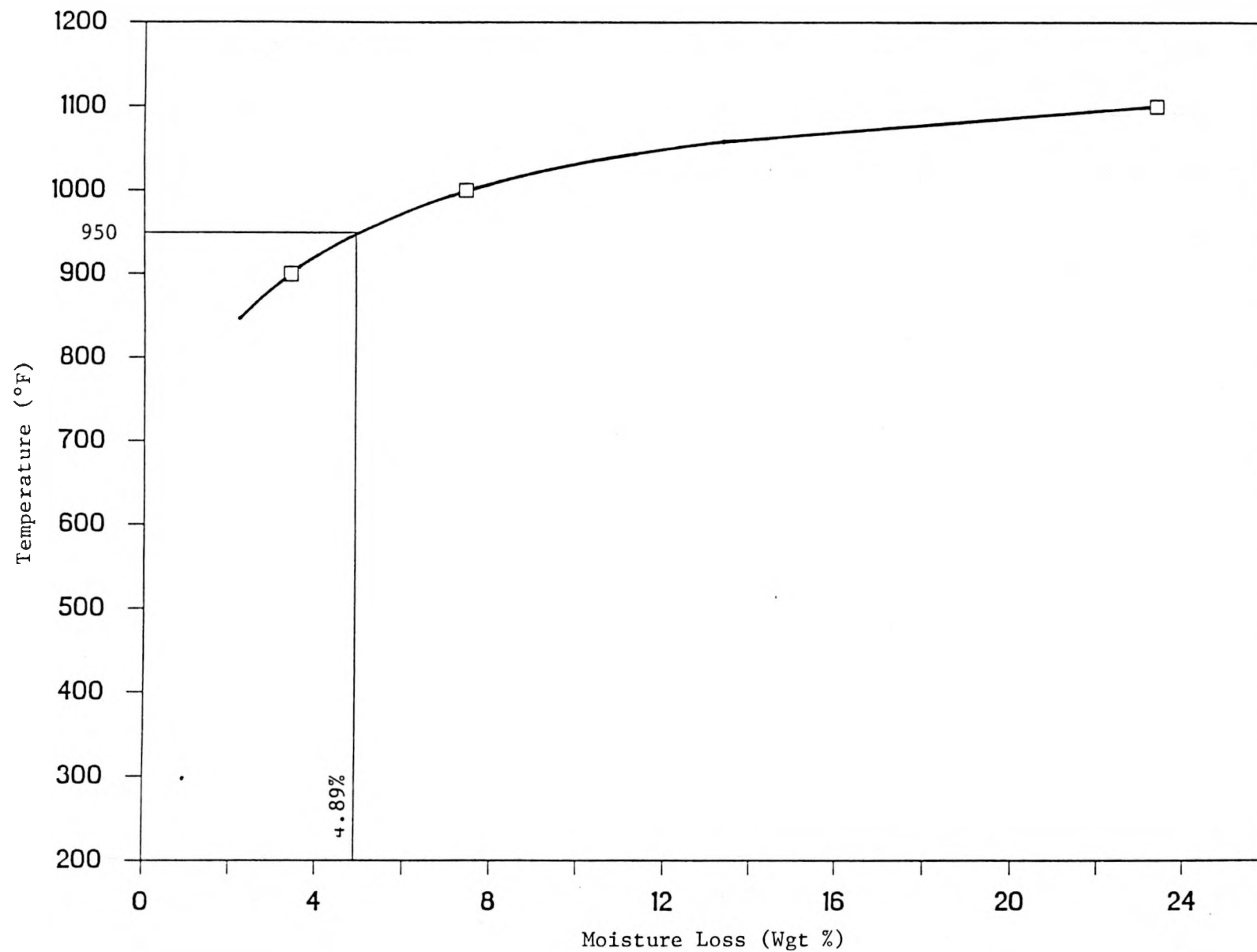


Figure 17.
Moisture Loss of Hydrated Lime

The next parametric test (MGU Test #19/P8) was designed to investigate the effect of coal particle size on the quantity and quality of the coal liquids produced. The raw H&K coal feedstock was ground from a 1-1/2" top size to 1/8" x 0. The MGU was operated in vacuum mode with no sweep or recycle gas through the coal bed. The results of this test are given in Table 12.

As shown in Table 12, the total liquid yield obtained in MGU Test #19/P8 was 13.3% (9.6% oil + 3.7% water). The liquids obtained in this test appeared to be of very good quality with a density slightly lower than that of the liquids from previous parametric tests. The results of the analytical tests conducted on these liquids are shown in Table 14.

In addition to the somewhat better coal liquid quality, the char product from Test #19/P8 contained a slightly greater amount of volatile matter than the chars from previous MGU tests (11.86%). The increase in volatile matter should make the char easier to ignite and generally improve its combustion characteristics.

The reduction in coal particle size also appeared to reduce the degree of difficulty normally associated with dislodging the char from the reactor tubes. As a result, the char product was generally larger in lump size (3"-6"), which would improve its potential as a coke substitute.

MGU Tests #20/P9 and #21/P10 were conducted to investigate the effects of lime on the mild gasification process. Lime is known to be effective in reducing the sulfur content and enhancing the quality of coal liquids. The H&K coal feedstock was crushed to 1/8" X 0 in order to blend the lime evenly throughout the coal. The lime utilized for these tests was a hydrated powder of approximately 325 mesh x 0 in size and contained 72.2% CaO with a total of 96.7% total $\text{Ca}(\text{OH})_2$. According to the *Handbook of Chemistry and Physics* (9), hydrated lime decomposes to $\text{CaO} + \text{H}_2\text{O}$ at approximately 580°C (1076°F), which is very close to the reaction temperatures utilized in the MGU testing. Laboratory moisture tests were conducted on the hydrated lime at temperatures of 900°, 1000°, and 1100°F, to construct a temperature vs. moisture loss profile for the hydrated lime (see Figure 17). The moisture loss at 950°F, as determined from Figure 17, was utilized to calculate the moisture weight loss of the lime - and the corresponding increase in total water collected in the condensing system. The highest average lime temperature in these two tests was determined to be approximately 950°F.

The coal feedstock utilized for MGU Test #20/P9 contained 10% hydrated lime by weight. Although the powdered lime and 1/8" x 0 coal feed were very dusty in nature, there was very little entrainment of fine particles in the off gas lines or in the coal liquids. A major factor which helped to curb particle entrainment was the fact that the coal was charged into the reactor tubes at ambient temperature (in contrast, the feedstocks were charged into pre-heated reactor tubes in the previous MGU contract).

The coal liquid produced in this test appeared to be quite similar to that from the previous MGU test (#19/P8). According to the analyses performed on the liquid (see Table 14), the sulfur content was only marginally reduced from that of the liquid produced in Test #19/P8 using no lime. However, as shown in Table 14, the content of one of the hetero-atoms, oxygen, is reduced substantially, from 5.17% to 3.47%. The char produced in this test was easily

discharged from the reactor tubes and contained some rather large sized lumps (6"-12"), however the lime content made the char very friable and dusty. The lime addition also significantly increased the ash content of the char.

MGU Test #21/P10 was conducted using 20% hydrated lime additive. All other operating parameters were identical to those utilized previously in Test #20/P9. The liquids appeared to have a much lower viscosity than any of the liquids produced in previous tests. There did, however, appear to be a higher concentration of suspended water in the coal liquids. As shown in Table 14, all hetero-atom (N, S, O) contents were substantially lower than that of Test #19/P8, which used no lime additive.

As in the previous test with 10% lime, the char was easily discharged from the reactor tubes. The char discharged in large lump form, but was extremely friable - much more so than in the previous test. Due to the higher concentration of lime used in this test, the char ash content was proportionally higher.

Parametric Tests #22/P11 and #23/P12 were conducted to evaluate different types of coal as MGU feedstocks. Sub-bituminous and lignite coals were utilized for these tests. The MGU again operated in vacuum mode with no sweep gas or recycle gas through the coal bed, with a furnace temperature of 1000°F and a final center coal bed temperature of 900°F.

Test #22/P11 was carried out using Andersen-Dietz Sub-bituminous coal (see Table 16). There were no major irregularities in this test due to the change in coal feed, however, the higher moisture content of the sub-bituminous coal did affect the performance of the slip-stream condensing system. When the coal bed center and reactor gas exit temperatures reached approximately 200°F, the moisture in the bed was quickly converted into steam. This steam increased both the volume and velocity of the exit gases and reduced the efficiency of the condensing system. The increased velocity of the gases through the condenser prevented a large portion of the condensed liquids from draining back into the main condenser canister. These liquids (primarily water) were swept down stream from the main condenser and collected in a flask installed at the slip-stream manometer location. A much greater quantity of ice was consumed in this test as the main condensing canister and copper coil on the slip-stream condenser remained hot throughout most of the 6 hour test.

The total liquid yield for Test #22/P11 was 32.3% - however the coal liquid yield was only 3.3%. The coal liquids obtained from this test were lighter than water and dark brown/black in color. The substantial volume of water (28.9%) appeared to be clearer than the water fractions from previous bituminous coal tests and did not display the ammonia odor usually associated with "bituminous water". Tables 14 and 15 contain the results of the analyses conducted on the coal liquid and char products.

Test #23/P12 utilized Mississippi Lignite as the coal feedstock. The high moisture content of the lignite taxed the efficiency of the slip-stream condensing system, much the same as it did in the previous test with sub-bituminous coal. The maximum temperature indicated by the center coal bed thermocouple was only 645°F, 500 minutes after initiating the test. The information obtained from all other monitoring devices (other thermocouples, GC analyses, etc.) indicated that the test run should be near its completion.

Table 16.
Results of Proximate & Ultimate Analyses for
MGU Coal Feed Samples Used in Parametric Tests P1 - P21

	<u>Wellmore</u> <u>#8</u>	<u>Wellmore</u> <u>#8 Refuse</u>	<u>H&K</u>	<u>"FRESH"</u> <u>H&K</u>	<u>Andersen</u> <u>-Dietz</u> <u>Sub-Bit</u>	<u>Miss.</u> <u>Lignite</u>
% MOISTURE	1.32	1.56	1.88	1.88	36.10	33.20
% ASH	8.83	61.30	5.10	5.03	4.55	12.87
% VOLATILE	33.17	16.42	34.45	34.38	45.11	37.43
% FIXED CARBON	58.00	22.28	60.54	60.59	50.34	29.70
% CARBON	78.53	29.66	79.68	83.71	71.93	65.54
% HYDROGEN	5.36	2.00	5.13	5.42	4.74	5.34
% NITROGEN	1.17	0.70	1.49	1.06	0.93	0.90
% SULFUR	1.52	3.56	1.00	1.27	0.34	1.50
% CHLORINE	0.12	0.04	0.13	0.11	0.03	0.02
% OXYGEN	4.47	2.73	7.47	3.40	17.48	13.83
Btu/lb	14,247	5,054	15,161	14,630	12,085	10,918

* NOTE: Oxygen values are calculated "by difference".
 All values, except moisture, are reported on "dry" basis.
 All analyses were conducted by Galbraith Laboratories, Inc. of
 Knoxville, Tennessee, except Btu/lb for Wellmore #8 and H&K Coals,
 which were analyzed by United Coal Co. Central Laboratory.

It was speculated that the coal/char bed was shrinking in volume, thus lowering the top of the coal/char bed to where it was at or near the center thermocouple location. The lower temperature of the evolving gases at the top of the coal/char bed would likely have resulted in lowering the temperature recorded by the center thermocouple. It was therefore decided to terminate this test after 500 minutes. It was later discovered that the low bed center temperature readings may have been caused by a short in the thermocouple wiring. Because of these anomalies, product analyses for Test #23/P12 were not carried out and reported. This test was repeated in Test #24/P13.

The total liquid yield obtained in Test #23/P12 was 35.98%, with a total oil yield of 4.8%. The coal liquid obtained was lighter than water and dark brown in color - although somewhat lighter in color than the sub-bituminous liquids. The oil appeared to contain a substantial amount of waxy material that would dissolve in the oil when the temperature was raised to approximately 100°F or higher.

Test #24/P13 (conducted with Mississippi Lignite coal) was a repeat of Test #23/P12 in which some irregularities were encountered with the center coal bed thermocouple. In order to insure that the thermocouples would remain covered in the coal/char bed throughout the run, the gravel charge was increased from 40 lbs. per reactor tube to 60 lbs. Also a new thermocouple was installed in the center coal/char bed location prior to the initiation of this test. A summary of the results from Tests (#24/P13 - #28/P17) are given in Table 17.

The liquids collected from Test #24/P13 were very similar to those collected in the previous lignite test (23/P12). The coal oil fraction was lighter than water and brown in color. This oil contained a substantial amount of waxy material, which would dissolve in the oil when it was heated to approximately 100°F. The results of the analytical tests conducted on these liquids are shown in Table 18.

A substantial quantity (14.2 lbs.) of liquids condensed in the char hopper during this test. The majority of this liquid was water and had a stronger odor than those that condensed in the slip-stream condenser. The oil portion of these liquids appeared to contain a high concentration of a waxy tar material. It was also apparent that this "oil" fraction had dripped from the bottom of the reactor tubes as it formed "stalagmites" in a circular pattern on the char hopper lid. It was not possible to effectively decant this oil fraction as it was very thick and contained a large concentration of fine char particles.

Test #25/P14 was conducted using 1/16" x 0 coal preparation plant refuse. Due to the greater density of this material the charge was increased to 140 pounds, which was roughly equivalent (by volume) to that of 110 pounds of bituminous coal. The duration of this test was approximately 75 to 100 minutes less than that needed to complete a test under the same conditions using a bituminous coal feedstock. Due to the higher ash content of this feedstock (56.5%), there was little gas pressure build-up inside the reactor tubes, the condensing system remained under a vacuum condition throughout the entire test period, and the char chute area was under little or no positive pressure during the latter half of the run. The char product did agglomerate to form some rather large sized lumps, however it was very friable and easily reduced to "powder".

The coal liquid produced in this test were lighter than water and dark brown/black in color. An ammonia odor was readily apparent particularly associated with the water portion of the liquids. Although there was a noticeable odor, it was milder than the ammonia odor associated with the liquids derived from previous bituminous coal tests. As shown in Table 17, the total liquid yield for this test was 9.28 pounds (6.63% by weight). This lower yield was due to the lower percentage of volatile matter content of the refuse feedstock (see Table 16). However the quality of the coal liquids was quite good - in fact, very similar to that of the liquids produced in other MGU tests with raw bituminous coals (see Table 18).

Test #26/P15 was conducted using Wellmore #8 Bituminous Coal. The purpose of this test was to examine the effect of the slightly lower volatile content of the Wellmore #8 coal (32.7% volatile) on the MGU product yields and qualities.

Table 17.
Comparison of Results from Parametric Tests P13 - P17

TEST #	<u>#24/P13</u>	<u>#25/P14</u>	<u>#26/P15</u>	<u>#27/P16</u>	<u>#28/P17</u>
OVEN TEMP (°F)	1000	1000	1000	950	1000
COAL TYPE	Lignite	Refuse	Wellmore #8	H&K	H&K
PARTICLE SIZE	$\frac{3}{4}$ x 0	$\frac{1}{16}$ x 0	1" x 0	$1-\frac{1}{2}$ x 0	$1-\frac{1}{2}$ x 0
MAX CENTER COAL TEMP	901°F	901°F	901°F	851°F	902°F
SWEEP GAS	-	-	-	-	steam
NON-CONDENSIBLE GAS YIELD (%) *	21.0	2.8	10.4	4.2	11.6
CHAR YIELD (%)	45.2	90.5	76.6	82.5	76.4
TOTAL LIQUID YIELD (%)	33.8	6.6	13.0	13.3	12.1
OIL YIELD (%)	5.8	2.5	8.2	8.6	10.0
WATER YIELD (%)	28.0	4.1	4.8	4.7	2.1

* NOTE: The non-condensable gas yield is calculated by difference.

Table 18.
Results of Analytical Tests Conducted on MGU Coal Liquid Samples
from Parametric Tests P13 - P17

TEST #	<u>24/P13</u>	<u>25/P14</u>	<u>26/P15</u>	<u>27/P16</u>	<u>28/P17</u>
% MOISTURE *	0.89	0.45	0.71	0.78	0.65
% CARBON	85.08	85.83	84.74	84.32	85.31
% HYDROGEN	10.78	8.87	8.45	9.32	8.94
% NITROGEN	0.57	0.48	0.91	0.71	0.82
% SULFUR	0.36	0.68	0.63	0.51	0.42
% OXYGEN	4.09	4.31	5.47	4.26	5.25
MOLECULAR WEIGHT *	252	235	229	229	251
BTU/LB.	17,881	17,055	16,837	17,042	16,967
H/C RATIO	1.50	1.23	1.19	1.32	1.21

* NOTE: All values except molecular weight and percent moisture are reported on dry basis.

The liquid product obtained from this test were very similar in appearance to those obtained using the H&K #2 bituminous coal. The decanted oil and water yields were 8.16% and 4.83% respectively which are close to those obtained in previous MGU tests utilizing H&K #2.

MGU Test #27/P16 was conducted to investigate the effect of reduced temperature on the quantity and quality of the MGU coal liquids. For this test the oven temperature was reduced from 1000°F to 950°F and the maximum desired coal/char bed center temperature was reduced from 900°F to 850°F.

The quantity and quality of the coal liquid produced in this test appeared to be nearly identical to those of liquids produced with a 1000°F furnace temperature.

The char product did have a somewhat higher volatile content than the chars from previous tests conducted at the higher 1000°F furnace temperature. The results of the analytical tests conducted on the char are given in Table 19.

Test #28/P17 utilized steam as a sweep gas. The steam for this test was produced by a 17 kw electric steam generator. The steam was introduced into the tops of the two 8" gas heater tubes, where it was heated to super heated steam, and then entered the coal bed through the 1" crossover pipes located at the bottoms of the reactor tubes. Due to the low injection rate (3.2 lbs./hr) and long distance that the steam had to travel before entering the gas heater tubes, the steam cooled and entered the gas heater tubes primarily in the form of hot water. The pressure surges produced as the "steam" was injected into the gas heater tubes during the test substantiate this theory.

Approximately 11 lbs. of the 19.2 lbs. of steam (water) utilized in this test condensed in the char hopper. The coal liquid generated in the test is lighter than water and appears to be quite similar in overall quality to that of the liquids produced in previous tests using a 1000°F oven temperature. The non-condensable gas analysis data for parametric test runs P13 - P17 are shown in Table 20.

Table 19.
Results of Analytical Tests Conducted on MGU Char Samples
from Parametric Tests P13 - P17

TEST#	<u>24/P13</u>	<u>25/P14</u>	<u>26/P15</u>	<u>27/P16</u>	<u>28/P17</u>
<u>PROXIMATE, %</u>					
MOISTURE	0.77	0.38	1.46	0.67	1.18
ASH	20.07	63.65	9.88	6.43	5.12
VOLATILE	24.68	9.05	10.69	11.28	10.30
FIXED CARBON	55.25	27.60	79.43	82.29	84.58
<u>ULTIMATE, %</u>					
CARBON	67.16	31.13	82.66	84.73	86.81
HYDROGEN	2.93	1.71	3.03	2.86	3.15
NITROGEN	1.19	0.70	2.60	1.83	0.82
SULFUR	1.66	2.86	0.72	0.82	0.85
CHLORINE	0.020	0.022	0.066	0.11	0.074
OXYGEN (by diff)	6.97	0.23	2.70	3.90	3.18
BTU/LB.	11,362	7681	13,452	13,879	14,345

NOTE : The ULTIMATE and PROXIMATE (except moisture) are reported on DRY BASIS.

The purpose of MGU Test #29/P18 was to investigate the consequences of removing the insulation wrapped around the upper 15 inches of the reactor tubes inside the oven. This insulation was originally placed around the reactor tubes in an attempt to reduce the degree of coal gas cracking by reducing the impact of the oven burner flame on the temperature of the upper part of the tubes. Test #29/P18 was conducted using 110 pounds of 1-1/2 inch x 0 H&K (Williamson #2 Seam) bituminous coal. The MGU was operated in vacuum mode with no sweep or recycle gas through the bed.

As can be seen in Table 21, the total liquid yield obtained in MGU Test #29/P18 was 14.3% (9.0% oil + 5.3% water). The quantity and quality of the coal liquid produced appear to be similar to those of liquids produced in MGU tests which did have insulation wrapped around the upper part of the reactor tubes (also see Table 22). This indicates that the insulation has little to no effect on reducing the degree of coal gas cracking when operating the MGU with a 1000°F oven temperature. However, the insulation may indeed reduce the degree of coal gas cracking when the MGU is operated at higher oven temperatures - such as the 1300°F temperature utilized in Test #15/P4.

The remaining three parametric tests (MGU Tests #30/P19, #31/P20, & #32/P21) were conducted to investigate the effects of using different feed coal particle sizes in combination with nitrogen sweep gas on the performance of the MGU. For MGU Test #30/P19, the 1-1/2" x 0 H&K bituminous feed coal was crushed to 1/8" x 0. A very low nitrogen flow rate was utilized (0.5 to 1.0 cfm) in this test, however, the low rate allowed the nitrogen to be utilized throughout the test without excessive pressure build-up in the gas heater pipes.

Table 20.
Non-Condensable Gas Analysis
MGU Parametric Tests P13 - P17

SAMPLE#	ELAPSED TIME	INNER REACTOR TEMP °F	OUTER REACTOR TEMP °F	VOLUME PERCENTAGE						
				H ₂	CO [*]	CO ₂	CH ₄	C ₂ H ₆	$\frac{C_2H_4}{+C_2H_2}$	H ₂ S
TEST #24/P13										
1	57 min	289°	920°	8.2	8.1	69.1	9.5	2.9	1.5	0.7
2	117	325	948	19.7	4.8	55.4	14.0	4.0	1.5	0.6
3	177	421	959	25.1	3.3	49.8	15.6	4.4	1.4	0.4
4	237	657	963	25.4	3.9	44.8	17.9	5.3	2.4	0.5
5	297	817	967	27.4	3.6	36.1	21.9	7.2	3.4	0.4
TEST #25/P14										
2A	133 min	568°	947°	16.8	5.2	12.7	41.0	13.4	3.7	7.1
2B	133	568	947	16.2	5.7	12.7	40.7	13.4	3.7	7.6
3	193	737	956	16.9	5.0	10.6	41.2	13.1	3.2	10.0
TEST #26/P15										
4	138 min	565°	962°	16.8	6.1	5.3	47.6	16.3	3.4	4.4
5A	168	634	964	19.1	1.5	4.2	50.6	16.9	3.7	4.1
5	168	634	964	16.6	4.9	4.5	44.9	21.7	3.3	4.1
6	228	736	958	19.8	5.1	3.0	50.1	15.5	3.3	3.2
7	288	785	962	19.7	3.5	3.4	50.4	16.4	3.1	3.6
8A	348	839	965	22.0	1.6	2.8	52.5	14.9	2.9	3.3
8B	348	839	965	22.0	3.7	3.0	51.1	14.4	2.8	3.0
9	408	883	971	24.3	3.8	2.8	51.4	12.4	2.4	2.9
10	448	900	825	22.5	6.4	3.0	50.6	12.1	0.9	4.5
TEST #27/P16										
1	68 min	330°	878°	10.8	4.1	15.1	48.1	18.4	2.2	1.4
2	115	481	894	12.0	5.8	9.4	50.4	18.1	2.0	2.4
3	175	617	897	15.6	8.1	7.1	48.8	16.7	1.6	2.0
4	237	714	901	18.2	5.7	5.0	50.6	16.8	1.6	2.0
5	295	779	905	18.0	5.6	4.0	51.9	16.9	1.5	2.1
TEST #28/P17										
1	64 min	372°	938°	0.0	5.2	13.6	57.3	20.1	2.4	1.4
2	108	453	946	16.7	3.5	8.0	50.6	16.7	2.5	2.0
3	168	587	949	18.9	3.2	6.1	50.6	16.3	2.8	2.2
4	228	716	953	19.8	4.8	4.1	50.9	15.5	2.5	2.5
5	288	763	956	21.5	3.2	3.2	52.5	15.1	2.2	2.4
6	348	828	959	24.1	4.4	3.0	51.6	13.2	2.1	1.6
7	408	879	960	27.8	4.4	2.7	47.8	14.4	1.6	1.3

* NOTE: Value shown for CO concentration also includes N₂.

Table 21.
Comparison of Results from Parametric Tests P18 - P21

Test #	<u>29/P18</u>	<u>30/P19</u>	<u>31/P20</u>	<u>32/P21</u>
Oven Temp (°F)	1000	1000	1000	1000
Coal Type & Size	H&K 1- $\frac{1}{2}$ " x 0	H&K $\frac{1}{8}$ " x 0	H&K $\frac{1}{4}$ " x 0	H&K $\frac{1}{2}$ " x 0
Max Center Coal Temp (°F)	903	900	907	896
Sweep Gas	None	N ₂	N ₂	N ₂
Char Yield (%)	77.3	78.9	76.6	76.1
Non-Condensable Gas Yield (%) *	N/A	N/A	N/A	11.3
Total Liquid Yield (%)	14.3	13.8	15.7	15.4
Oil Yield (%)	9.0	8.5	10.3	10.0
Water Yield (%)	5.3	5.3	5.4	5.4
Material Balance Closure (%)	N/A	N/A	N/A	102.8

* NOTE: Non-condensable gas yields were calculated by measuring the gas flow rate and composition at set intervals during each test run. Gas yields for Tests 29-31 are not given due to erroneous results obtained in gas composition analyses conducted on some samples during these tests.

Table 22.
Results of Analytical Tests Conducted on MGU Coal Liquid Samples
from Parametric Tests P18 - P21

	<u>29/P18</u>	<u>30/P19</u>	<u>31/P20</u>	<u>32/P21</u>
Molecular Weight *	251	240	251	243
% Moisture	1.14	0.87	0.49	0.84
% Carbon	85.60	84.60	85.16	85.45
% Hydrogen	8.93	9.49	9.13	9.10
% Nitrogen	0.85	0.54	0.82	0.82
% Sulfur	0.43	0.41	0.57	0.65
% Oxygen	5.73	5.98	4.75	4.77
Btu/lb	17,060	17,742	17,209	17,208
H/C Atomic Ratio	1.24	1.34	1.28	1.27

* NOTE: All values except molecular weight and % moisture are reported on a dry (moisture free) basis.
All analyses were conducted by Galbraith Laboratories, Inc. of Knoxville, Tennessee.

Table 23.
Results of Analytical Tests Conducted on MGU Char Samples
from Parametric Tests P18 - P21

	<u>29/P18</u>	<u>30/P19</u>	<u>31/P20</u>	<u>32/P21</u>
% Moisture	0.46	1.14	0.69	0.58
% Ash	5.02	4.40	5.84	6.91
% Volatile	10.69	11.41	10.22	10.48
% Fixed Carbon	84.29	84.19	83.94	82.61
% Carbon	85.72	86.75	86.47	84.74
% Hydrogen	3.38	3.67	3.16	3.23
% Nitrogen	1.84	1.44	1.95	1.64
% Sulfur	0.76	0.74	0.94	1.44
% Chlorine	0.072	0.067	<0.05	0.048
% Oxygen *	3.21	2.93	1.59	1.99
Btu/lb	14,436	14,403	14,230	13,973

* NOTE: Oxygen values are calculated "by difference".

No liquids collected in the char chamber during Test #30/P19 - as has been true in every test that utilized N₂ sweep. The char produced in the lower part of the reactor tubes appeared to contain a high concentration of volatile material (i.e., some of the char appeared to be almost "wet").

The liquid yield in Test #30/P19 was 13.8% (8.5% oil and 5.3% water). Although the coal oil yield was slightly lower than "normal", the coal liquid collected appeared to be very light and "thin" (low in viscosity). The oils did not display the greenish tint that the lightest MGU liquids have - these coal liquids were more of an amber (reddish brown) color. The results of the analytical testing conducted on the oil fraction (see Table 22) indicate that the liquid is indeed of good quality as evidenced by the relatively low molecular weight (240) and hydrogen/carbon atomic ratio of 1.34.

MGU Test #31/P20 utilized a 1/4" x 0 raw H&K bituminous coal feed size. As in the preceding MGU test, a very low nitrogen flow rate was utilized (0 to 1.2 cfm - 0.64 avg), however in this test, the nitrogen had to be shut off at certain periods during the run to prevent an excessive pressure build-up in the gas heater pipes and char hopper area. As in Test #30/P19, no liquids collected in the char chamber during this run.

The total liquid yield obtained in Test #31/P20 was 15.7% (10.3% oil and 5.4% water). The coal liquid collected appeared to be very light and "thin" (low in viscosity) and was very similar in appearance to the oil fraction from MGU Test #30/P19. However, the increase in oil quantity appears to have been accomplished at the expense of the oil quality as indicated by the higher molecular weight (251 vs. 240) and lower H/C atomic ratio (1.28 vs. 1.34).

MGU Test #32/P21 utilized a 1/2" x 0 raw H&K bituminous coal feed size. A low nitrogen flow rate was again utilized in this test (0 to 1.35 cfm - 0.54 avg). As was true in Test #31/P20, the nitrogen had to be shut off at certain periods during the run to prevent an excessive pressure build-up in the gas heater pipes.

As in the two previous tests, no liquids collected in the char chamber during this run. The coal liquid collected appeared to be very light and "thin" (low in viscosity) and was very similar in appearance to the oils from MGU Tests #30/P19 and #31/P20. This liquid did appear to contain a somewhat greater amount of fine char than that from the preceding MGU tests. As can be seen in Table 22, the molecular weight of the coal liquid was nearly the same as that from Test #30/P19 (243 vs. 240), but the lower H/C ratio more closely resembled the coal liquid from Test #31/P20 (1.27 vs. 1.28).

In addition to the ultimate analyses presented above, several carefully selected coal liquid samples were also sent to outside laboratories for NMR analysis (Virginia Polytechnic Institute & State University and East Tennessee State University). This was done to better understand the effect of the various parametric operating conditions on coal liquid quality in terms of its chemical composition. Table 24 shows a proton (¹H) distribution comparison for coal liquids produced under various operating conditions from the same principal H&K bituminous coal feedstock.

Table 24.
Proton (¹H) Distribution in H&K Bituminous Coal
Parametric Test Run Liquids

<u>Test Run No.</u>	<u>Operating* Conditions</u>	<u>Alkyl Proton, %</u>	<u>Mono- Cyclic</u>	<u>Di- Cyclic</u>	<u>Tri- Cyclic</u>
#12/P1	900°F, 1-1/2"x 0 No Sweep Gas No Additive	72	17	9	2
#13/P2	1000°F, 1-1/2"x 0 No Sweep Gas No Additive	55	22	20	3
#14/P3	1100°F, 1-1/2"x 0 No Sweep Gas No Additive	45	24	28	3
#15/P4	1200°F, 1-1/2"x 0 No Sweep Gas No Additive	35	25	36	4
#18/P7	900°F, 1-1/2"x 0 N ₂ Sweep Gas No Additive	73	17	9	1
#19/P8	900°F, 1/8"x 0 No Sweep Gas No Additive	76	16	7	1
#21/P10	900°F, 1-1/2"x 0 No Sweep Gas 20% Lime Additive	78	18	3	1
#28/P17	900°F, 1-1/2"x 0 Steam Sweep Gas No Additive	74	21	3	2

NOTE: * The first two data given in the "Operating Conditions" column indicate the final coal bed center temperature and the feed coal particle size, respectively.

Table 24 clearly shows that as the final coal bed center temperature is increased from 900°F to 1200°F, the chemical components are increasingly shifting from alkyl type to the generally heavier aromatic type compounds. Nitrogen and steam sweep gas have some marginal effect in reducing the aromatic content in favor of the alkyl compounds. Smaller coal feed particle size and lime additive appear to have a more pronounced effect than sweep gas in boosting the alkyl compound content. Table 24 also indicates that most of the aromatic compounds are in the lighter and smaller mono-cyclic and di-cyclic aromatics, not the heavy, multiple ring type aromatic material.

2.2.3.3 Determination of the Optimum MGU Operating Conditions

Based on the results obtained from the shakedown and parametric testing program, the "optimum" MGU operating conditions were determined. These are as follows:

- Temperature: The best quality coal liquids have been produced when utilizing a **1000°F furnace temperature** with a final **center coal bed temperature of 900°F**. Although somewhat greater liquid yields are obtained at higher temperatures, the additional yield is composed primarily of heavy tar which mars the combustion characteristics of the liquid. The chars produced under higher temperatures also were of poorer quality (in terms of volatile matter content). It seems apparent that the small gain in liquid yield under higher operating temperatures is more than offset by the loss in liquid and char quality as well as thermal efficiency.
- Sweep Gas: A relatively low **nitrogen sweep** gas rate appears to improve the quality as well as the quantity of the coal liquid collected. The sweep gas helps to reduce the residence time of the coal gases inside the reactor tubes and prevents them from entering and condensing in the char hopper area. It should be noted that the use of sweep gas does increase, to a small degree, the entrainment of fine coal/char particles in the coal gas stream. It also can decrease the condensing system efficiency by lowering the gas stream dew point due to the sweep gas diluting effect on reaction off gas. However, as long as the sweep gas flow rate is small, these problems are not serious. Both nitrogen and non-condensable recycle gas can be used as sweep gas and will have similar effects on MGU product quality and yield. On the other hand, using steam as sweep gas does not seem to render the same beneficial effects as nitrogen or non-condensable gas, under the general mild gasification operating conditions. In fact, it adds substantial burden to the gas cooling/condensing system causing over-all plant thermal efficiency reduction and operating cost increase related to oil/water separation and waste water treatment.
- Coal Feed & Particle Size: By far the best quality and quantity of coal liquids have been obtained from **bituminous** coals. Freshly mined bituminous coal feedstocks produced liquids of better quality and greater yield than those which have been in storage for an extended period of time (i.e., oxidized coals). It appears that using freshly mined and smaller size (**1/8" x 0**) feed coal, in combination with nitrogen sweep gas and a low operating temperature, produces the best quality liquids. Also, surprisingly, it appears that the sweep gas has less difficulty continuously penetrating a coal bed of minus 1/8-inch particles than it does beds with larger coal sizes. Furthermore, char produced from coal feeds with a smaller particle size seemed to be easier to discharge from the reactor tubes.

In addition, although lime additive does have a positive effect on improving coal liquid quality, (especially its ability of reducing heteroatom contents, S, N, O.), it nonetheless substantially increases the ash content of the main product, char. Since our current economic study indicates that the most economic utilization of mild gasification char is via producing coke from the char, the ash content limitation (~8%) of the coke product makes this lime treating option undesirable.

2.2.3.4 Effectiveness of Venturi Scrubber & Tray Tower Condensing System

Four tests were conducted on the MGU to evaluate the efficiency of the venturi scrubber & tray tower condensing system - which was originally installed as part of the MGU modification effort. In all of the previous parametric tests, the slip-stream condensing system was utilized to condense the coal gas stream due to the need to conserve liquid product individuality (as well as for material balance determination purposes) and due to the established reliability of the slip-stream condenser.

The operating conditions utilized in these four tests were those determined to be the optimum MGU operating conditions from the parametric testing. The vacuum pump (located in the non-condensable gas line to the flare) was used in these tests to improve the flow of the gases through the system. No cooling water was supplied to the coal liquid heat exchangers due to the low outdoor temperatures experienced during these tests. The first "venturi" test (#V1) was conducted using 2.5 cfm of nitrogen sweep gas. Both the venturi scrubber and the tray tower liquid circulating pumps were operated at their maximum capacities (3 gpm and 1 gpm, respectively). The quantity and location of the liquids collected during the test, as well as operating conditions experienced are shown in Table 25. After the test was completed, it was discovered that there was actually less condensible liquid in the tray tower reservoir than there was before the test started. The total liquid loss was determined to be approximately 300 ml (~0.66 lbs).

Table 25.
Quantity, Location of Condensed Liquids,
and Operating Conditions for Venturi-Scrubber/Tray Tower Tests

	<u>Test #V1</u>	<u>Test #V2</u>	<u>Test #V3</u>	<u>Test #V4</u>
Liquid Yield (%)				
- Cyclone	6.0	8.3	7.3	9.2
- Venturi	5.0	0.0	7.7	0.0
- Tray Tower	0.6	0.2	0.0	0.0
- Total Liquid	11.0	8.3	15.0	9.2
Char Yield	74.2	83.3	77.1	75.0
Non-Condensable Gas	14.8	8.3	8.0	15.8
Ambient Temperature (°F)	50°	45°	64°	42°
<u>Circulating Liquid Temperature (°F)</u>				
Venturi Scrubber	68°	54°	95°	61°
Tray Tower	60°	48°	72°	52°

Note: Non-condensable gas yields calculated by difference.

In venturi test #V2, the nitrogen sweep gas rate was decreased to 1.5 cfm. This was done to observe the effect of the sweep gas (if any) on the liquid loss in the tray tower. After completion of the test, the liquid level in the tray tower had again decreased, however only by ~100 ml in this test. As in the previous test, both the venturi scrubber and tray tower recirculation pumps were operated at their maximum flow rates (~3 and 1 gpm respectively). The only liquids that condensed in this test (8.33%) were found in the char cyclone (before the venturi scrubber). It is believed that effects of the reduced nitrogen flow rate in conjunction with the lower outdoor temperature (~40°F) on the day of the test allowed the liquids to condense before reaching the condensing system.

The liquid yield in Test #V3 (15.0%) was much higher than that obtained in the two previous venturi tests. Although the nitrogen sweep gas rate was lowered to ~1.0 cfm, the outdoor temperature (64°F) on the day of the test was much higher than in the previous tests. This higher outdoor temperature allowed the hot off gases from the reactor to increase the temperature of the condensing system piping & components to 90~95°F. This in turn caused residual condensed liquids/tars to soften and flow to areas where they could be collected in addition to the condensible liquids produced during this run.

In an effort to minimize the liquid loss from the tray tower, the recirculation pump was throttled back almost completely. The reasoning behind this action was that in the two previous tests the recirculation pump was flooding the trays with condensed liquid, which was then overflowing into the piping to the vacuum pump and out the flare gas line. This action appeared to correct this problem as there were no liquids observed at the flare gas exit and there was no change in the height of the liquid level in the tray tower reservoir.

Venturi Test #V4 incorporated all of the above findings to optimize the efficiency of the condensing system. In addition, non-condensable gas samples were collected after the tray tower and analyzed with a gas chromatograph (GC). These analyses (see Table 26) indicate that no significant quantities of condensible hydrocarbons are left in the non-condensable gas stream - even though the liquid recovery is lower than those obtained with the slip-stream condensing system. This indicates that the liquids were condensing and coating the rather lengthy span of insulated piping (~20 feet) between the reactors and the condensing system.

Table 26.
Non-Condensable Gas Analysis for Venturi Test #V4

Sample Time		Volume %												ISO-		
		H ₂	CO	CO ₂	H ₂ S	CH ₄	C ₂ H ₆	C ₂ H ₄	C ₃ H ₈	C ₃ H ₆	n-C ₄ H ₁₀	1-C ₄ H ₈	BUTANE	C ₄ &C ₅	TOTAL	
#	(Min)															
1	60	23.22	9.93	3.07	0.28	34.91	14.94	2.64	4.68	2.79	1.05	0.91	0.21	1.38	100.00	
2	120	25.19	2.64	2.54	0.99	32.19	17.18	3.72	6.26	4.02	1.44	1.38	0.29	2.16	100.00	
3	240	37.19	1.13	2.31	1.19	41.41	8.89	1.29	2.51	1.51	0.64	0.60	0.12	1.12	100.00	

2.2.3.5 Conclusions from Venturi Testing

The conclusions drawn from the venturi scrubber/tray tower testing can be summarized as follows:

- Due to the concern for excessive entrainment of particulate material from the coal/char bed, it was not possible to operate the venturi scrubber - tray tower condensing system at its designed, higher gas flow rate (~10 cfm). This experience also means that the venturi condensing system should be carefully designed only after the incoming gas flow conditions can be ascertained.
- Heat tracing of the piping is needed to control the proper condensing/separation of the condensible vapor such that the heavier liquids will be collected in the venturi scrubber (rather than in the char cyclone) and the lighter ends in the tray tower. Also heat tracing would prevent the condensible vapor from condensing in the piping before reaching the designed components for condensing. The lack of heat tracing also cause the ambient temperature of an outdoor facility to assert a significant impact on the condensing efficiency of the system.

2.2.4 Production of Char and Condensible Hydrocarbons

With the parametric testing complete and the "optimum" conditions at which to conduct the production runs established, work began on the production of approximately 1200 lbs. of char for combustion tests under Task 3. This entailed a total of 21 runs to produce sufficient quantities of char for the char combustion tests.

With a view to ascertain the composition of the fresh H&K coal selected for use in these production runs, a sample of this coal analyzed by Galbraith Laboratories in Knoxville, TN to obtain the composition of the coal feedstock. The results of these analyses are shown in Table 27 below.

Table 27.
Analyses of Production Run H&K Fresh Coal Feedstock

Moisture	1.91	Carbon	79.14
Ash	6.85	Hydrogen	4.91
Volatile	33.64	Nitrogen	1.26
Fixed Carbon	59.51	Sulfur	0.98
		Chlorine	0.15
		Oxygen (by diff)	6.71
		Btu/lb	14,723

Note: All values except moisture are reported on a dry basis.

Two production runs per day were conducted using two eight hour shifts. Less extensive sampling and cleaning procedures were needed in the production runs than in the previous parametric testing. This helped to greatly reduce the turn-around time required between runs.

The liquids from the production runs appear to be of good quality. Table 28 shows the analyses for two production run liquids and a total production run liquid blend sample. The analyses shown in Table 28 indicate that the molecular weight varies about 14% (201 for Test #33/PRO-1 vs. 228 for Test #46/PRO-14). However after discussion with Galbraith personnel, this falls within the error range of their molecular weight measurement technique for coal liquids as they have encountered in the past. There is a small quantity of "waxy" material evident in the liquids, but when heated to 100°F this material dissolves in with the oil. When the oil sample was allowed to settle, very small particles of this "waxy" material seemed to precipitate on to the sides of the glass bottle. The total yield for the liquids throughout the production runs were generally from 15% to 16%. In Table 29 the liquid yield is shown to be somewhat less for Test #33/PRO-1 than that shown for Test #46/PRO-14. We attribute this in part to the nitrogen sweep gas only slightly penetrated the coal/char bed during the test. An additional complication was that due to delivery mix up we were unable to utilize nitrogen for the complete duration on the test run.

The liquids produced during these production runs have been sent to DOE/METC designated testing organization, CORE Laboratory in Houston, Texas, for physical property data base testing.

The chars collected from the 21 production runs are of good quality, with volatile matter contents exceeding 10% and are consistent with the results from the parametric test runs. The char was discharged hot and cooled by quenching it with water to enable the second run to be initiated quickly. As shown in Table 30, this procedure did not have any adverse effects on the quality of the char. The char is friable and appears to be well cooked. Table 30 shows the analytical results for two production run chars and an over-all production run char blend sample. The 2" x 0 particle size of the char will be used to conduct Stoker boiler burning tests at CTC, and the +3" lumps will be used for burning test at the Tenetex foundry.

Table 31 contains the results of the gas chromatograph (GC) analyses conducted on the non-condensable gas samples taken during Test #33/PRO-1 and Test #46/PRO-14 of the production runs.

Table 28.
Analytical Results for Liquids from Two Production Runs
and Over-All Production Liquid Blend

	Test #33/PRO-1	Test #46/PRO-14	Production Blend
Molecular Weight	201	228	220
Moisture (%)	0.87	1.18	0.94
Carbon (%)	84.88	85.87	85.01
Hydrogen (%)	9.18	8.82	9.16
Nitrogen (%)	0.59	0.73	0.67
Sulfur (%)	0.71	0.55	0.49
Oxygen (%)	3.84	4.64	4.68
BTU/Pound	17,331	17,093	16,895
H/C Atomic Ratio	1.29	1.23	1.29

NOTE: All values except molecular weight and % moisture are reported on dry basis.

Table 29.
Comparison of Operating Conditions & Yields from Two Production Runs

	Test #33/PRO-1	Test #46/PRO-14
Oven Temp. (°F)	1000	1000
Coal Type	Fresh H&K	Fresh H&K
Coal Particle Size	1/8" x 0	1/8" x 0
Max Center Coal		
Bed Temp (°F)	893	904
Sweep Gas	N ₂	N ₂
Char Yield (%)	77.3	76.4
Non-Condensable Gas		
(by difference)(%)	7.3	7.8
Total Liquid Yield (%)	13.9	15.8
- Coal Oil Yield (%)	7.8	10.0
- Water Yield (%)	6.1	5.8

Table 30.
Analytical Results for Chars of Two Production Runs
and Over-All Production Char Blend

	Test #33/PRO-1	Test #46/PRO-14	Production Blend
Moisture, %	0.29	0.58	1.54
Ash	7.90	8.16	7.83
Volatile Matter	10.22	10.27	10.73
Fixed Carbon	81.88	81.57	81.44
Carbon	85.60	86.40	78.69
Hydrogen	2.71	2.82	3.04
Nitrogen	1.85	1.83	1.91
Sulfur	0.97	1.08	0.84
Chlorine	0.097	0.09	0.086
Oxygen (by diff)	0.87	-	7.60
Btu/lb	13,714	13,932	13,737

Note: All values except % moisture are reported on dry basis.

Table 31.
Non-Condensable Gas Analysis for Two Production Runs

MGU PRODUCTION RUN #1 (Test #33/PRO-1)

Sample		VOLUME % - N2 SUBTRACTED												ISO-		TOTAL
		H ₂	CO	CO ₂	H ₂ S	CH ₄	C ₂ H ₆	C ₂ H ₄	C ₃ H ₈	C ₃ H ₆	n-C ₄ H ₁₀	1-C ₄ H ₈	BUTANE	C ₄ &C ₅		
#	TIME	17.05	0.00	8.57	1.74	45.34	14.53	1.97	4.79	2.32	1.22	0.10	0.22	2.14	100.00	
1	60	22.96	0.00	4.54	2.52	36.95	18.02	2.02	5.95	2.52	1.45	0.18	0.28	2.62	100.00	
2	120	24.16	0.59	3.55	2.52	34.95	18.13	2.20	6.31	2.71	1.54	1.00	0.30	2.05	100.00	
3	180	25.60	0.61	3.53	2.63	35.61	17.41	1.91	5.77	2.39	1.46	0.68	0.26	2.15	100.00	
4	255	30.30	0.63	3.88	2.14	39.88	13.19	1.28	4.00	1.53	1.00	0.60	0.19	1.37	100.00	
5	300	23.22	0.00	5.58	0.00	45.63	15.87	1.01	4.66	1.47	0.97	0.50	0.21	0.88	100.00	
6	360															
7	420															

MGU PRODUCTION RUN #14 (Test #46/PRO-14)

Sample #	TIME	VOLUME % - N2 SUBTRACTED											ISO-		TOTAL
		H ₂	CO	CO ₂	H ₂ S	CH ₄	C ₂ H ₆	C ₂ H ₄	C ₃ H ₈	C ₃ H ₆	n-C ₄ H ₁₀	1-C ₄ H ₈	BUTANE	C ₄ &C ₅	
1	83	20.44	0.00	5.59	2.46	40.28	16.28	2.65	5.29	2.83	1.27	0.00	0.24	2.67	100.00
2	138	23.01	0.67	5.07	2.50	39.17	15.21	2.57	4.92	2.70	1.18	0.90	0.21	1.90	100.00
3	195	24.27	0.47	4.44	2.47	39.02	15.00	2.67	4.83	2.79	1.12	0.92	0.20	1.79	100.00
4	255	26.12	0.00	3.29	2.50	41.77	13.71	2.30	4.35	2.39	1.04	0.79	0.20	1.54	100.00
5	318	27.03	0.35	2.90	2.68	43.30	12.54	1.95	3.96	2.01	0.96	0.70	0.19	1.44	100.00
6	375	30.57	0.45	2.50	2.30	44.53	11.08	1.57	3.30	1.63	0.77	0.00	0.16	1.15	100.00
7	416	31.71	0.00	3.24	2.23	47.53	8.63	0.79	2.54	0.96	0.66	0.00	0.96	0.74	100.00

2.2.4.1 Quality Comparison of MGU Production-Blend (CTC Pro-Blend) and COALITE Coal Liquids

Currently, the only commercial mild gasification plant operating in the world is the COALITE plant in England. As a result, the performance and products of this plant have become the focal reference of literally all mild gasification researchers around the world. Since the beginning of this project, one of CTC's primary goals has been to produce mild gasification products which are comparable or better in quality than the commercial COALITE products.

As the CTC mild gasification unit (MGU) is the largest operating mild gasification facility in the U.S., it is also instructive to gauge its performance against a commercial plant to see if it is favorable for the U.S. to vigorously pursue the next logical step toward commercial development of the mild gasification technology. Furthermore, the MGU process is similar in nature to the COALITE process. Therefore, a comparison of the MGU production-blend liquid (produced under the optimum conditions described above) and COALITE liquid is in order. Under a separate program sponsored by a DOE/METC grant for locomotive diesel fuel development, CTC acquired a substantial quantity of raw COALITE liquid. Table 32 and Figure 18 show the comparison of CTC Pro-Blend and raw COALITE coal liquids in terms of ultimate and NMR analyses as well as distillation data.

As can be seen in Table 32, the CTC Pro-Blend liquid has substantially less hetero-atom (S, N, O) content and has a higher H/C atomic ratio than the COALITE liquid. The NMR analysis and distillation data also indicate the MGU liquid has substantially more lighter transportation type fuel constituents. All of these data appear to support the conclusion that the CTC Pro-Blend liquid is superior in quality to the COALITE liquid. Although the average molecular weight data for the COALITE liquid (218) and the CTC Pro-Blend liquid (220) are essentially identical, all of the other data indicate that the COALITE liquid is substantially heavier than the CTC Pro-Blend. Thus, it is possible that the one time molecular weight measurement of 218 for the COALITE liquid may be in error. Repeated molecular weight measurements have been conducted on the CTC Pro-Blend liquid with quite consistent results. However, due to the fact that there is no mechanical means in place to thoroughly mix the COALITE liquid in storage as well as the fact that no substantial quantities have been withdrawn from the storage tank since the last testing (as of this writing), it has not been possible to obtain a representative sample of the COALITE liquid for additional molecular weight measurements.

Table 32.

Comparison of CTC Pro-Blend and COALITE Coal LiquidsI. Ultimate Analysis,* Wt%

	<u>CTC Pro-Blend</u>	<u>Coalite</u>
Carbon	85.01	84.49
Hydrogen	9.16	8.48
Oxygen	4.68	4.83
Nitrogen	0.67	1.03
Sulfur	0.50	0.97
H/C Atomic Ratio	1.284	1.196
Moisture	0.94	1.57
Molecular Weight	220	218
Btu/lb	16,736	-

*NOTE: Data on dry basis except moisture, molecular wt., and Btu/lb, which are "as received".

II. Proton (¹H) Distributions (NMR Analysis)

	<u>CTC Pro-Blend**</u>	<u>Coalite</u>
<u>Alkyl Proton, %</u>	77	69
<u>Aromatic Proton, %</u>		
Mono-Cyclic	17	5
Di-Cyclic	3	21
Tri-Cyclic	3	5

**NOTE: In addition to the Proton (¹H) distribution shown above, the LC-NMR analysis also revealed the molecular chemical component distribution in the CTC Pro-Blend as:

Alkanes	44.3%
Mono-cyclic Aromatics	34.3%
Di-cyclic Aromatics	13.9%
Tri-cyclic Aromatics	7.6%

No similar data for the COALITE liquid are available for comparison at this writing.

III. Distillation Data (Moisture Free Basis), Vol %

	<u>CTC Pro-Blend</u>	<u>COALITE</u>
Naphtha (IBP-350°F)	4.7	1.4
Diesel (350°-650°F)	60.1	49.1
Residual (B.P.>650°F)	35.2	49.5

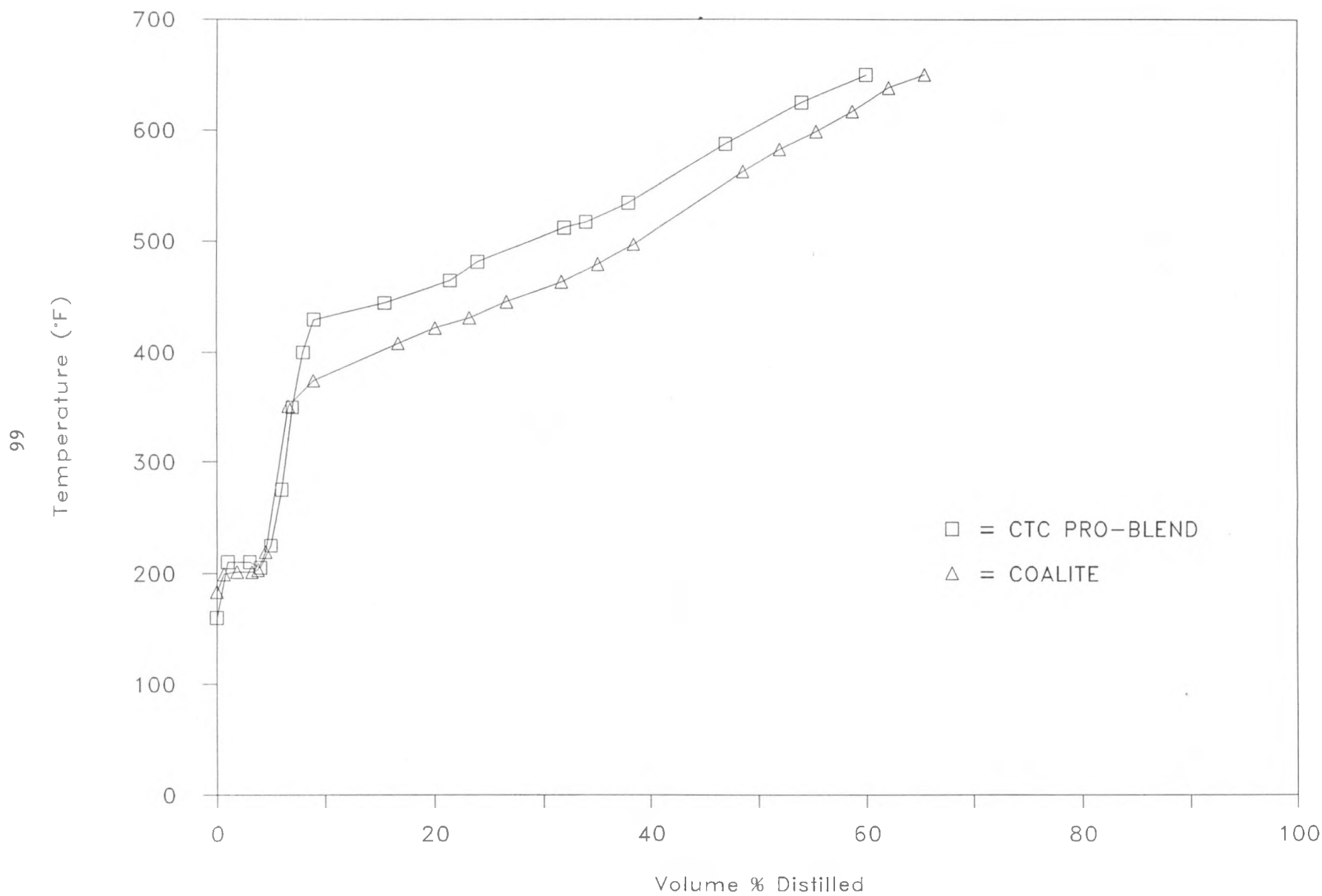


Figure 18. CTC Pro-Blend & COALITE Raw
Coal Liquid Distillation Curves

2.3 TASK 3 - EVALUATION OF CHAR & CHAR/COAL BLENDS AS AN INDUSTRIAL BOILER/BLAST FURNACE FUEL

2.3.1 Objective

The objective of this task is to evaluate the MGU char product in three commercial applications. Tests will be conducted to determine the suitability of char in industrial/utility pulverized coal boilers, stoker coal boilers, and as a replacement for coke in foundry/blast furnaces.

2.3.2 Results and Discussion

2.3.2.1 Test Plan for Char Evaluation as an Industrial Boiler/Blast Furnace Fuel

The preliminary test plan was submitted to DOE/METC on April 15, 1987 and is enclosed in this report in Appendix B.

The test plan called for the char burning tests to be carried out with the following test equipment and locations:

- [1] Industrial/Utility Boiler
 - TAS-System of TAS-COAL
 - Magna, Utah
- [2] Foundry Furnace
 - Cupola of Tenetec Corporation
 - Johnson City, Tennessee
- [3] Stoker Boiler
 - Coal Technology Corporation
 - Bristol, Virginia

For the stoker boiler test above, it was originally planned to use a larger existing stoker boiler located at United Coal Company's corporate office. The char feed rate for this unit would be approximately one ton per hour. However, due to the fact that this unit had been idle for a number of years and would likely have required significant repairs/preparation to get it back in operating condition, it was decided, with DOE's approval, to switch the testing to a smaller residential stoker boiler. The smaller residential boiler functions quite similar to the larger unit, only on a much smaller scale.

The original test plan misquoted the char requirement for the foundry furnace test as being 1200 lbs for the initial cupola bed and 1875 lbs for twenty-five, additional 75 lb charges. On confirmation with Tenetec Corporation before the initiating the test, it was learned that the testing would require only about 600 lbs for the initial cupola bed and 525 lbs for seven additional 75 lb charges.

2.3.2.2 Characterization of powdered char as an Industrial/Utility Boiler Fuel

The combustion tests to investigate the possible use of char in industrial/utility pulverized coal boilers were conducted at TAS Inc.'s

combustion research facility in Magna, Utah. Schematic diagrams of the test unit are shown in Figures 19 and 20. Basically, the unit operates as follows: Char (or coal) is conveyed from the feed hopper to the TAS mill by a screw conveyor. As the feed enters the grinding mill it is mixed with primary combustion air. The micronized feed, suspended in the primary combustion air stream, is conveyed to the burner assembly. Secondary and tertiary combustion air is introduced at this point and the micronized feed is combusted in the kiln. Ash and other particulate material suspended in the combustion gas stream are removed in a bag house before the gases pass to the exhaust stack.

Two tests were conducted in the TAS unit, one with coal and one with char. Proximate and ultimate analyses for both feedstocks are shown in Table 33. The particle size distribution of the feed material (after grinding) is also shown in Table 33. Prior to initiating these tests, the instrumentation for measuring the combustion gases was calibrated (in each test the combustion gas stream was analyzed for SO_2 , CO , NO_x , and O_2). The precise feed rate of the 6-inch screw conveyor, which fed the coal (or char) to the TAS mill, was determined by collecting and weighing the material conveyed during a known period of time.

For each test, the coal/char was placed in the storage hopper and fed to the TAS mill at the desired rate. In Test #1 (Wellmore #8 bituminous coal), the coal firing rate was adjusted to the maximum heat loading of the facility's combustion chamber (approximately 8 MMBtu/hour). This loading is referred to as the *high fire rate* segment of the test. After the necessary stabilization period (in which adjustments were made to minimize the air flow without affecting the CO concentration), data was gathered for 30 minutes. Ash collected in the bag house was collected for analysis.

After completion of the high fire rate segment, the feed rate was reduced to approximately 1/6 of the original rate. This loading is referred to as the *low fire rate* segment of the test. After the appropriate stabilization period, data was gathered and ash samples collected as before. At the end of the low fire rate test period, the feed rate was returned to the original high fire rate (8 MMBtu/hour) and a grind sample was obtained.

The procedure for Test 2 (char) was the same as that used for Test 1 (coal), with the exception that a support fuel was required to stabilize the char flame during the low fire rate test.

The results of the coal and char combustion tests are shown in Table 34. The Wellmore #8 coal produced a stable flame at oxygen concentrations of greater than 2%. At oxygen concentrations of less than 2%, the concentration of carbon-monoxide (CO) in the combustion gas was very unstable. It was also noted that the char flame (Test 2) had a tendency to move out of the burner head at the high fire rate. It was necessary to reduce the combustion air in the burner head to maintain the flame in the proper burner area.

The major differences in the high fire rate tests with coal and char were in the NO_x and SO_2 emissions. The NO_x emission for the char was approximately twice as great as that for the coal, while the SO_2 emission for the char was approximately half of that of the coal. The average flame temperature and combustion efficiency of the char at the high fire rate were only slightly less than those of the coal.

Table 33.
Analyses of TAS Systems Feedstocks

<u>Proximate</u> (dry wt%)			<u>Ultimate</u> (dry wt%)		
	<u>Coal</u>	<u>Char</u>		<u>Coal</u>	<u>Char</u>
Ash	7.69	17.57	Carbon	78.62	74.99
Volatile Matter	30.64	11.42	Hydrogen	5.09	2.41
Fixed Carbon	61.67	71.01	Nitrogen	1.53	1.43
			Oxygen	5.47	2.32
			Sulfur	1.49	1.10
			Chlorine	0.11	0.18
<u>Particle Size</u> (microns)					
	<u>Coal</u>	<u>Char</u>			
90% less than	60.85	61.80			
50% less than	26.59	19.72			
10% less than	5.52	4.34			
Mean Size	30.75	27.01			

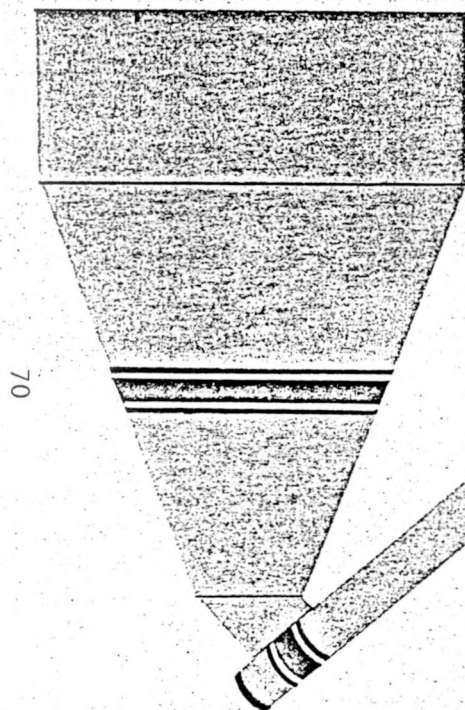
Table 34.
Operating Data from TAS System Tests

<u>Test Parameter</u>	<u>High Fire Rate^a</u>		<u>Low Fire Rate^b</u>	
	<u>Coal</u>	<u>Char</u>	<u>Coal</u>	<u>Char^c</u>
Average Flame Temp. (°F)	2867	2683	2333	2400
Primary Air Flow (scfm)	360	302	279	279
Secondary Air Flow (scfm)	976	829	890	579
Tertiary Air Flow (scfm)	549	552	501	346
<u>Combustion Gas Analyses</u>				
SO ₂ (ppm)	959	468	164	41
CO (ppm)	302	259	84	95
NO _x (ppm)	367	621	410	108
O ₂ (wgt %)	2.39	4.25	10.43 ^d	11.35 ^d
<u>Emission Data</u>				
SO ₂ (lb/MMBtu)	1.636	0.953	0.460	0.145
CO (lb/MMBtu)	0.240	0.231	0.124	0.148
NO _x (lb/MMBtu)	0.484	0.909	0.947	0.276
<u>Combustion Efficiency (%)</u>	88.9	86.8	88.5	86.5

Notes:

- a High fire rate is equivalent to the highest loading for the facility's combustion chamber (~ 8 MMBtu/hour). However the mill and burner are capable of firing to a maximum of 15 MMBtu/hour.
- b Low fir rate was at a 6 to 1 turndown ratio (~ 1.3 MMBtu/hour). Therefore, the turndown rate from maximum fire was ~ 12 to 1.
- c Support fuel (2000,000 Btu/hour) required for this test to stabilize the flame.
- d Air leakage across primary, secondary, and cooling dampers resulted in high oxygen concentrations on these burns.

TAS-COAL



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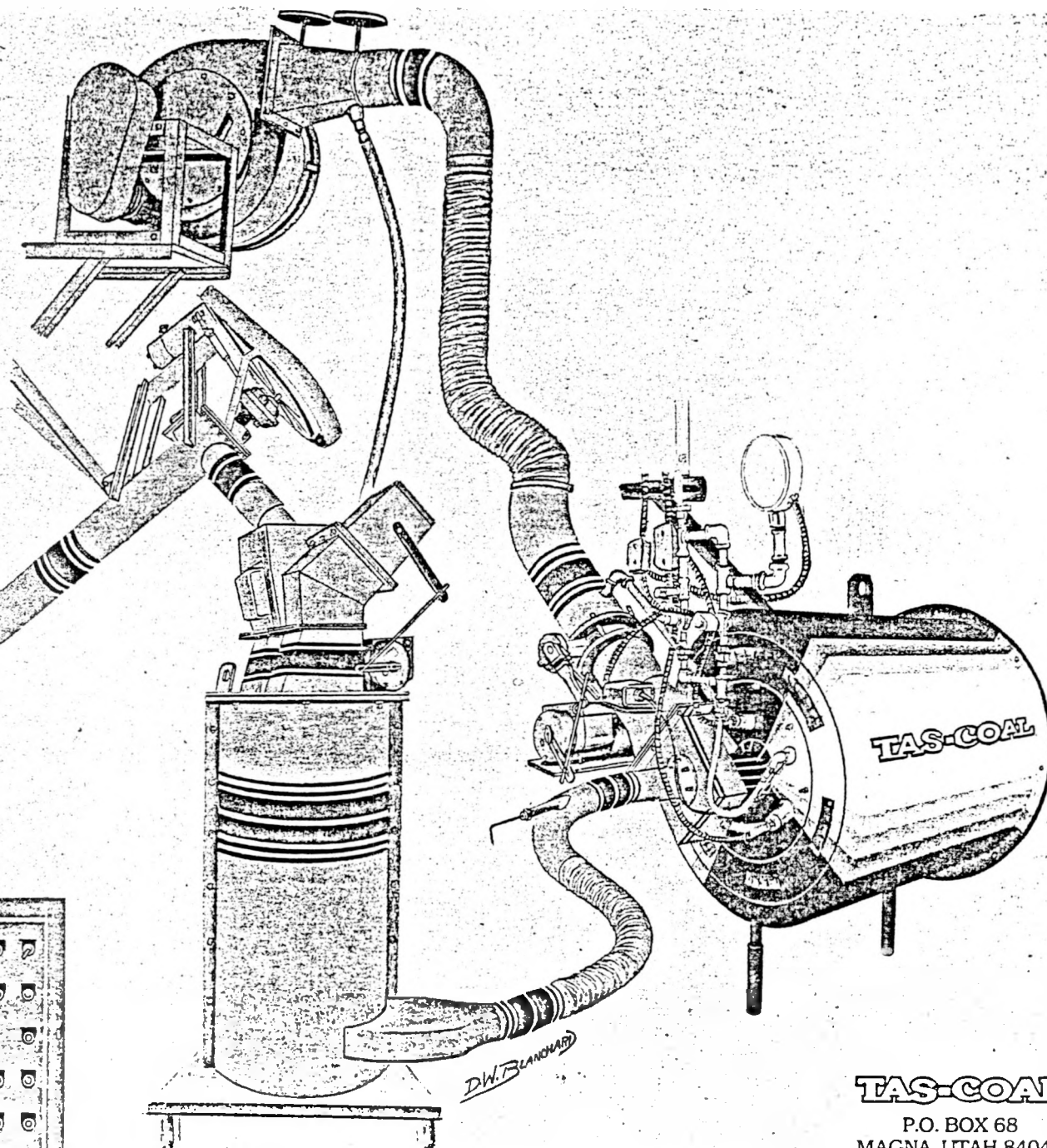
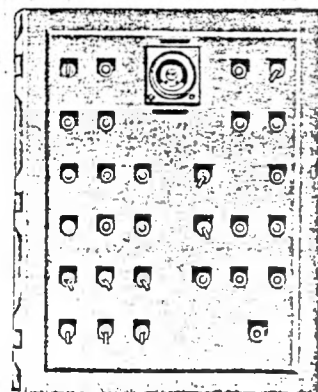


Figure 19. TAS-System Unit

TAS-COAL

P.O. BOX 68
MAGNA, UTAH 84044

Enquiries please call:
Kent White

1-801-250-2908

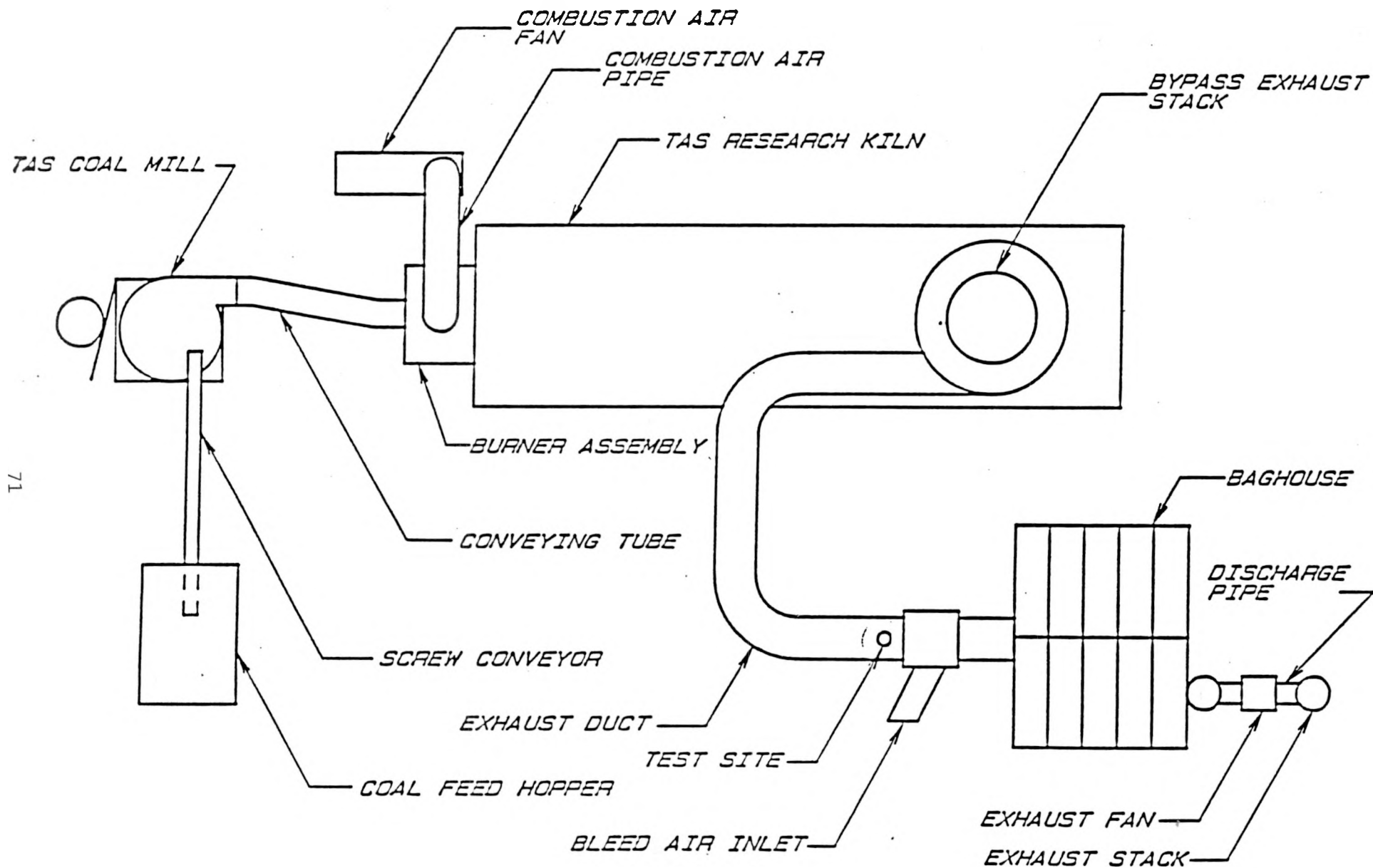


Figure 20. Schematic of TAS Inc.'s Combustion Research Facility

The SO₂ emission of the char was approximately 1/3 that of the coal at the low fire rate. The NO_x emission for the char was also approximately 1/3 that of the coal. The flame temperatures and combustion efficiencies for the char and coal at the low fire rate were approximately equal. It should be noted that roughly 200,000 Btu/hour of support fuel was required to produce a stable flame with the char at the low fire rate. This was the only time that support fuel was required.

In summary, the char burned successfully in the TAS system. Although a support fuel was required to maintain a stable flame with the char at the low fire rate, none was required at the high fire rate. The combustion efficiency of the char was only 2% less than that of the coal. The char flame temperature was also only slightly lower than that of the coal. The SO₂ emissions of 0.95 and 0.15 lb/MMBtu under the high and low fire rates were well below the Federal New Source Performance Standard of 1.20 lb/MMBtu. In addition, the engineers at TAS Inc., believe that the char be an acceptable fuel in numerous applications - such as cement plant kilns and asphalt plants.

2.3.2.3 Characterization of Char as a Foundry Furnace Fuel

The commercial foundry application burning test for the char was conducted at the Tenetech Foundry located in Johnson City, Tennessee. A cupola furnace was used for this test (see Figure 21). Basically the cupola was a vertical cylindrical stack of mild steel plate, elevated on columns above the ground, with semi-circular doors at the bottom. The inner stack surface was lined with refractory brick from top to bottom. Forced air from a fan blower was delivered to the wind belt and entered the cupola through the tyres at the bottom of the furnace. The coke (or char) and scrap cast metal charges were hoisted up to an opening approximately 1/3 of the way up the side of the stack (~25 feet above the ground). In normal casting operations, the coke imparts a portion of its carbon to the casting metal. Additives are often used to regulate the percentage of carbon donation to meet the desired specifications (generally about 2 - 4%).

The procedures followed for the char burning test were identical to those utilized when coke is used as the cupola fuel. The lump char feedstock utilized in this test was obtained during the previous MGU production runs conducted under Subtask 2.2.4. Approximately 300 lbs of char was first ignited in open drums outside the cupola. After this char was red hot, it was hoisted into the cupola, followed by an additional 400 lbs of char to complete the initial bed charge. The Tenetech personnel stated that the char appeared to burn somewhat hotter than coke and at a somewhat faster rate. After the additional char was added to the cupola bed, the air blower was started. Flames immediately belched from the cupola combustion chamber and red hot particles of char (brittles) blew out of the charging port and exhaust stack. This phenomenon did not occur when coke was used as the cupola fuel. Shortly after the flames died down, the first charge of scrap metal was dropped onto the char bed, along with an additional 75 lb charge of char. A second charge of scrap metal and char was added approximately 20 minutes later.

After the second charge, the char bed was observed through a sight glass in the tyre near the bottom of the cupola. It was immediately noticed that the char had literally disappeared, allowing the scrap metal to settle into the ashes. It appeared that the char had burned so quickly that the metal was

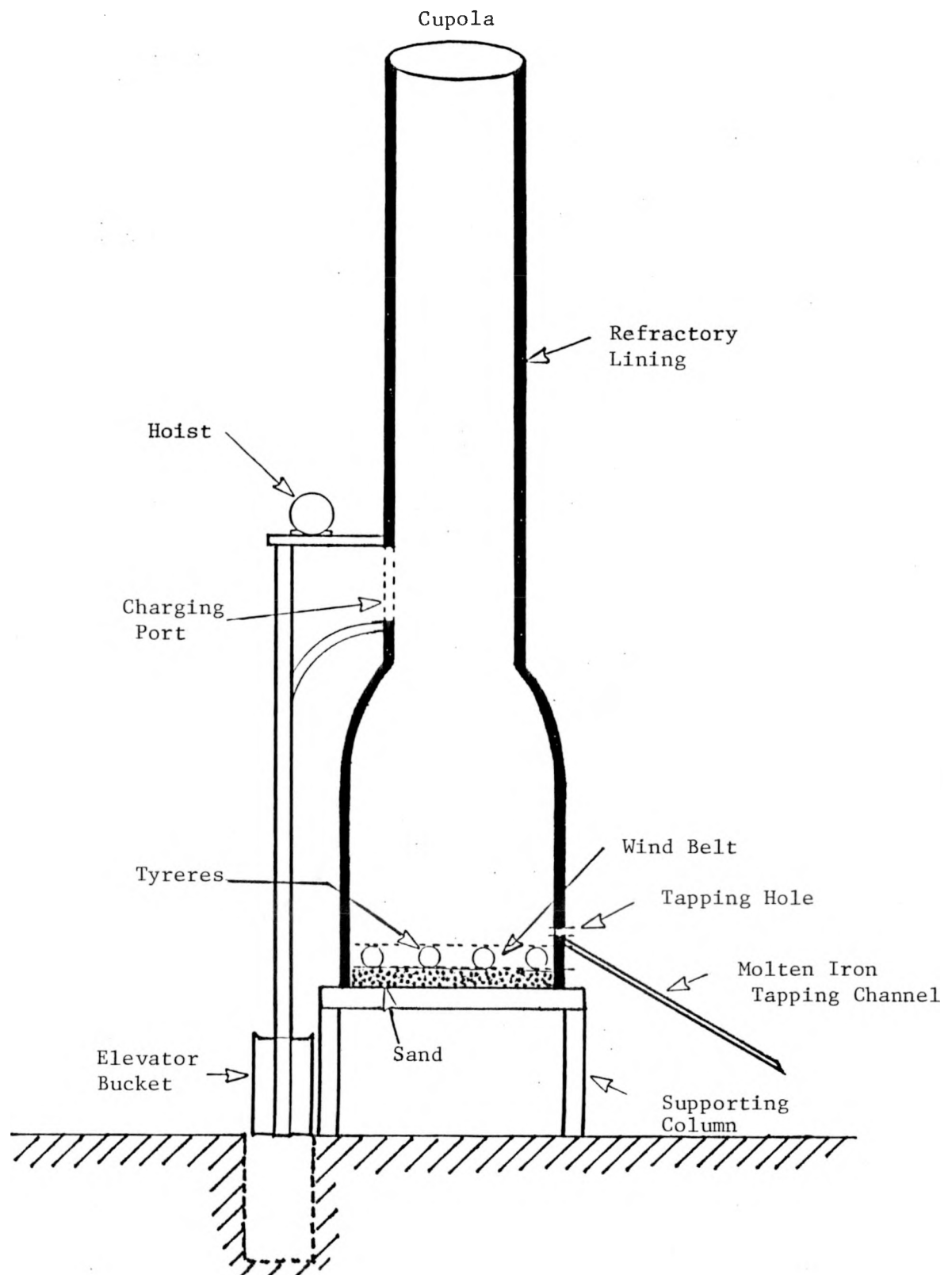


Figure 21. Foundry Cupola - Tenetek Corporation

not exposed to an adequate amount of heat to reach its melting point. The test was terminated at this point. After the scrap cast metal and ash were discharged from the cupola, it was noted that the metal from the first charge was only red hot in spots and that from the second charge appeared to be virtually unaffected. Due to the failure of this test, it was not possible to obtain any meaningful data as was originally planned.

After discussions with the Tenetek engineers, it was concluded that the char did not appear to have the strength nor the burning characteristics needed for the foundry cupola application without further processing. The Tenetek engineers did not feel that blending the char with coal or coke would produce satisfactory results either.

2.3.2.4 Characterization of Char as a Stoker Fuel

As stated previously, a residential stoker boiler unit was utilized for the char stoker tests (see Figure 22). In preparation for these tests, a water meter was installed on the boiler water supply inlet. This was done to obtain an accurate measurement of the boiler water consumption during each stoker test. In each test, the water consumption, steam pressure, weight of char or coal burned, and the Btu/lb value for the feedstock were used to calculate the boiler efficiency with each feedstock (coal and char).

In each of the coal and char combustion tests, the boiler was brought to a steady steam pressure using a coal feedstock. After achieving steady state, the feed hopper was emptied and a known weight of coal (or char) was loaded into the hopper. During the transition from coal to char, a period of approximately one hour was utilized to insure that no coal remained in the screw conveyor and/or tyrere. Water consumption, pressure, and temperature measurements were recorded throughout each of the tests. The temperatures of the combustion chamber, coal/char bed, and stack gases were recorded.

The char feedstock utilized in the stoker boiler tests was that produced during the MGU production runs. The coal feedstock used as the "base case" for these tests, was the same H&K coal used to produce the MGU production char. Both the coal and char feedstocks were crushed to a nominal 1/2" x 0 stoker feed size.

During the stoker tests, it was noted that the coal did agglomerate to form fairly large "clinkers". These clinkers were slowly pushed away from the tyrere (by the fresh coal from the screw auger) and created large open areas through which the forced air/flame could easily penetrate. However, the char did not agglomerate as it burned, and thus produced a more tightly packed bed. Due at least in part to this tighter packed bed (which somewhat restricted the forced air flow), the flame height with the char was much smaller than that produced with the coal. The tighter packed char bed also tended to retain more of its heat energy in the bed rather than transferring it to the air and walls inside the boiler.

The results of the stoker boiler tests are shown in Table 36. During the first two tests, the "medium" auger speed (1 rpm) was utilized with both coal and char. It was found that the char produced a higher boiler efficiency than did the coal (59.5% for char vs. 32.8% for coal). However, it was believed that the large difference in efficiencies was due primarily to the higher bulk density and Btu/unit volume of the coal and the fact that the boiler has a

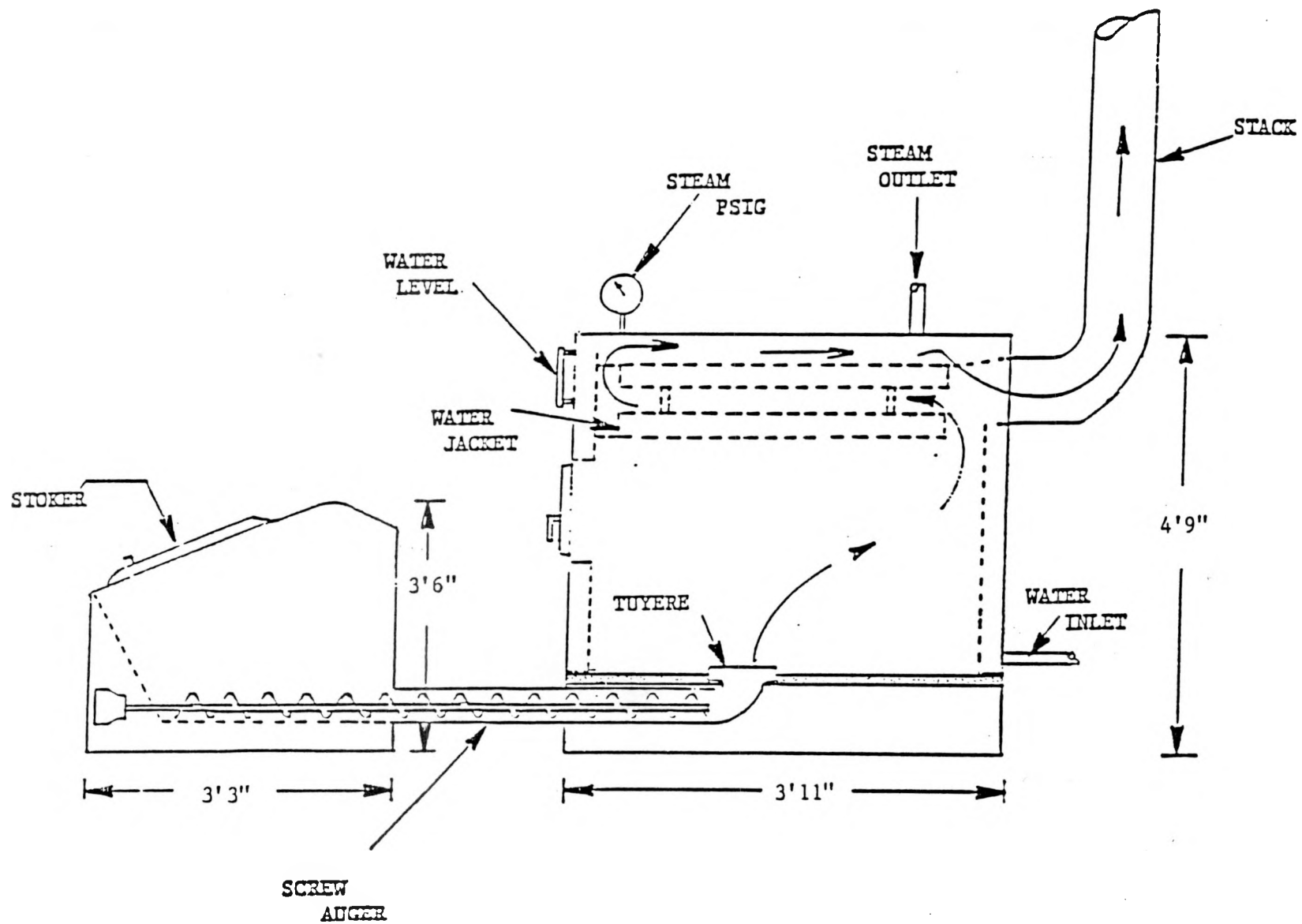


Figure 22. Residential Stoker Boiler - Coal Technology Corporation

fixed heat transfer surface area. That is, at the same feed auger speeds, a greater amount of energy per unit of time will be produced with coal and thus a larger portion of energy will be lost out the stack - thus resulting in a lower boiler efficiency. In order to effectively compare the performances of the coal and char, the energy input per unit of time should be the same. This theory was substantiated in the second coal stoker test, in which the auger speed was reduced to 0.6 rpm (slow rate). As shown in Table 36, the efficiency (39.4%) obtained in this second test was indeed improved over that obtained in the first (32.8%). As the stoker feed auger only had three speeds - 0.6 rpm (slow), 1.0 rpm (medium), and 1.33 rpm (high) - it was not possible to set the coal and char feed rates such that the energy input per unit time for both were equivalent.

For the purpose of comparing the degrees of combustion obtained in the coal and char tests, the combustion residue from both were sampled and analyzed (see Table 37). The analyses indicate that the coal underwent more complete combustion than did the char. This can easily be seen through the changes in the key data components (C, H, and Ash) between the feedstock and the corresponding residue as shown in Tables 35 & 37 for both the coal and char.

Table 35.
Ultimate & Proximate Analyses of Stoker Boiler Feedstocks

	H&K Coal <u>Feedstock</u>	Production <u>Char Feedstock</u>
Moisture, Wgt %	1.91	1.54
Ash	6.85	7.83
Volatile	33.64	10.73
Fixed Carbon	59.51	81.44
Carbon	79.14	78.69
Hydrogen	4.91	3.04
Nitrogen	1.26	1.91
Chlorine	0.15	0.086
Sulfur	0.98	0.84
Oxygen (by difference)	6.71	7.60
Btu/lb	14,723	13,737

Note: All values are reported on a dry basis (except moisture).

Table 36.
Comparison of Stoker Boiler Burning Tests with Char and Coal

	<u>Coal Test #1</u>	<u>Coal Test #2</u>	<u>Char Test</u>
Water Consumed (gallons)	35.4	52.0	33.6
Feed Consumed (lbs)	71.75	87.5	40.0
Steam Pressure (psig)	5.0	3.1	2.5
Visible Stack Emission	Slight (light gray)	Slight (light gray)	None
Feed Auger Speed (rpm)	1 (Medium)	0.6 (Low)	0.6 (Low)
Test Duration (minutes)	180	360	180
Boiler Efficiency * (%)	32.8	39.4	59.5

* Note: Boiler Efficiency = $\frac{(\text{Total Steam Energy Output})}{(\text{Total Fuel Energy Input})} \times 100$

Table 37.
Ultimate Analyses Comparison of Residue (Ash)
from Stoker Boiler Tests

	<u>Coal Test #1 Residue</u>	<u>Production Char Residue</u>
Moisture	0.86	0.51
Carbon	72.01	76.16
Hydrogen	0.25	0.23
Nitrogen	0.94	1.16
Chlorine	0.010	0.016
Sulfur	0.81	0.69
Ash	26.23	21.81
Oxygen	< 0.10	< 0.10
(by difference)		

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4.0 APPENDICES

APPENDIX A:

**Test Plan
for
Development of Mild Gasification Process**

March 4, 1987

Work Performed Under
Contract No. DE-AC21-87MC23289

For:
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia 26505

By:
UCC Research Corporation
of
United Coal Company
Bristol, Virginia 24201

TEST PLAN

The objective of this task is to develop a test plan for optimizing the Mild Gasification Process. Optimization will be accomplished in two phases. Phase I will be modification of the Mild Gasification Process Development Unit, MGU, to improve unit operation. The MGU was built and initial testing conducted during research performed by UCC Research Corporation (UCCRC) under Department of Energy Contract No. DE-AC21-84MC21108, "Management of Coal Waste by Energy Recovery: Mild Gasification (Pyrolysis) of Coal Preparation Wastes." Phase II of the optimization process will be parametric testing with the modified MGU. Tests will be conducted to study the effects of temperature, particle size, sweep gas, coal type, and lime additive on the quantity and quality of the liquid, solid, and gas products.

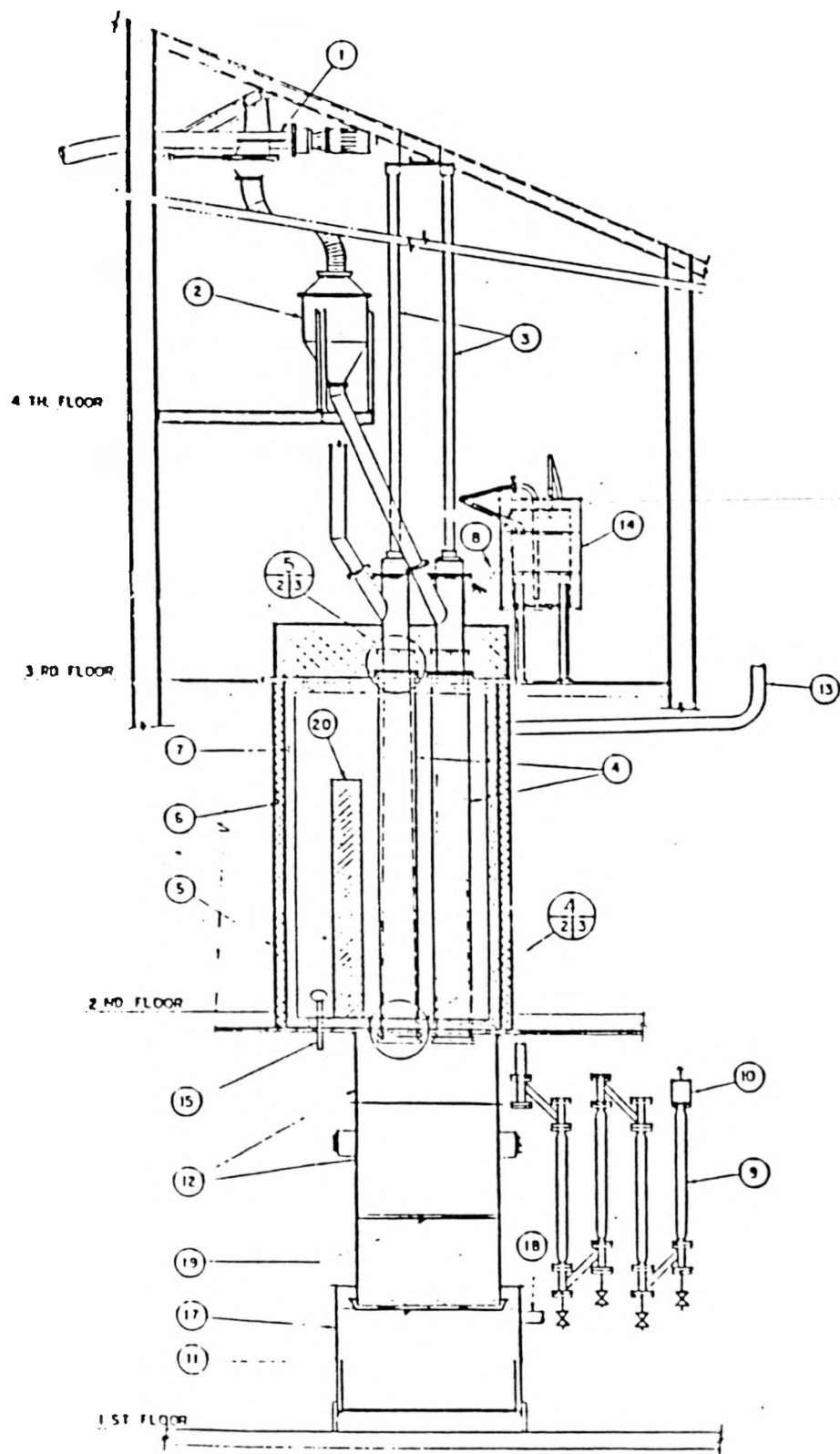
● Experimental Equipment and Facility Modifications

Figure 1 shows the MGU as built, and does not include proposed modifications. Parametric testing will be performed in the MGU after the facility is modified. Four major areas have been identified as needing modification.

The first major area of modification is the reactor tube diameter. The present eight inch diameter reactor tubes cause extremely slow heating rates ($\sim 5^{\circ}\text{F}/\text{min}$) in the center of the coal bed. In general, lower heating rates result in lower liquid yields and better liquid quality. Conversely, higher heating rates generally result in higher liquid yields and poorer liquid quality. While the quality of liquids from prior MGU tests have been good, liquid yields have been lower than expected. Yields were expected to be in the 12-15% range, but tests to-date have been in the 5-7% range. One contributing factor to the low liquid yield is the very slow heating rate. To increase the heating rate and thus improve the liquid yield, new reactor tubes with a smaller diameter will be installed. However, since liquid quality generally decreases with increasing heating rates, a compromise between liquid yield and liquid quality must be achieved. To determine the optimum reactor tube diameter, preliminary tests will be conducted in the current MGU with the char chamber removed temporarily, using non-tapered stainless steel reactor tubes. Four different diameters, 8, 6, 5, and 4 inches, will be tested. The reactor will be supported (fixed) for the time being at the top and float at the bottom for thermal expansion as is the case currently. The bottom of the reactor will be sealed with a blind flange. Char will be removed manually from the reactor tube after the reactor has cooled to ambient temperature. To prevent delay in this reactor diameter testing, the condensing system will also temporarily be the same indirect cooling system used during prior test runs.

The second area of modification is the coal feed system. Previous tests were hampered by the blockage of coal feed chutes, coal sticking in the volumetric hoppers, and with incomplete feeding of coal into the reactor tubes. Also, the flexible screw conveyor designed to carry coal from ground level to the volumetric hoppers on the fourth floor of the MGU never worked properly. The flexible screw broke during every attempt to convey coal to the volumetric hoppers. Therefore, a bucket

Figure 1. Mild Gasification Process Devevelopment Unit



LIST OF COMPONENTS

- (1) FLEXIBLE SCREW CONVEYOR DISCHARGE HEAD
- (2) 2-B C.F. VOLUMETRIC FEEDER (4 TYP.) SEE DET. $\frac{6}{2/3}$
- (3) HYDRAULIC CYLINDER FOR PLUNGER (4 TYP.)
- (4) STAINLESS STEEL REACTOR TUBE (4 TYP.) SEE DET. $\frac{4}{2/3}$ & $\frac{5}{2/3}$
- (5) FURNACE
- (6) FIRECLAY INSULATION
- (7) MONOLITHIC REFRACTORY
- (8) INSULATED 2" DIA. GAS EXIT PIPE
- (9) JACKETED PIPE STAGED CONDENSER (ABANDONED IN PLACE)
- (10) BENZINE (ABANDONED IN PLACE)
- (11) GRAVITY/PISTON OPERATED VALVE
- (12) COW CHUTE. SEE DET. $\frac{1}{2/4}$
- (13) FURNACE SACK
- (14) LIQUID COLLECTION OPEN
- (15) NATURAL GAS PIPE 1/2" BURNER
- (16) FURNACE DOOR
- (17) COW COOLING UNIT. SEE DET. $\frac{1}{2/4}$
- (18) VENT PIPE
- (19) HYDRAULIC OPERATED COW DISCHARGE GATE. SEE DET. $\frac{1}{2/4}$
- (20) FIRE BRICK BRILE

elevator system will be used to convey coal from ground level to the volumetric hoppers. In order to correct the remaining coal feeding problems, the following modifications will be made: The four existing volumetric hoppers and corresponding feed chutes will be removed. Four new volumetric hoppers will be installed on the third floor directly next to the reactor tube top assembly. This will eliminate the plugging of coal feed chutes and incomplete feeding of coal into the reactor tubes. In addition, a valve will be installed just below the coal feed entrance to the reactor tubes. This will separate the feed system from the reactor system and prevent gases and tars from accumulating in the coal feed system. The valve will be located such that the volume above the valve to the top of the volumetric hopper is equal to the volume of the reactor tube. The location of the new volumetric hoppers and valve is shown in Figure 2.

The third area of modification is the condensing system. Currently, the MGU uses indirect cooling to recover the condensable hydrocarbons. Indirect cooling is inefficient for condensing the aerosol-type vapor from this type of Mild Gasification Process. In addition, the current barrel system is cumbersome and inconvenient to use. To improve condenser efficiency, the current condensers will be replaced with a two-stage, direct-quenching, liquid recovery system. The new system will be a venturi scrubber followed by a tray tower and is shown in Figure 3.

The fourth major area of modification is the reactor tube support system. Currently, the reactor tubes are supported (fixed) at the top of the furnace and are free at the bottom to allow for thermal expansion. A satisfactory seal between the char chamber and furnace was never achieved. With the slight pressure conditions present at the beginning of the test run, coal gas escaped into the furnace chamber and was lost. With the slight vacuum conditions present during the middle and end of a test run, furnace gases as well as air from the char chamber were pulled into the reactor tubes causing coal oxidation and other undesired reactions in the coal bed. The leaking problem between the furnace and char chamber was especially damaging since the bottom reactor gate was not designed to form a gas-tight seal. To prevent this type of leaking problem, the support system for the reactor tubes will be changed to allow the reactor tubes to float at the top (for thermal expansion). The reactors will be fastened at the bottom to the furnace floor. The exact mechanism for fastening the reactor tubes is still to be determined, pending recommendations from the reactor tube manufacturer and design subcontractor. It is also planned to redesign the bottom reactor gate in an effort to prevent coal gases from leaking from the bottom of the reactor tube into the char chute. The new reactor gate design has not yet been determined.

● Brief MGU Test Procedures

The sized feedstock is conveyed by bucket elevator from the ground floor to the third floor of the MGU where it is loaded into four volumetric hoppers. From the volumetric hoppers the coal is gravity fed into the reactor tubes where it remains until a predetermined temperature is reached at the center of the coal bed. The gaseous products are withdrawn continuously via a vacuum pump and pass through the condenser system. Non-condensable gases are sent to a flare. The

Figure 2. New Volumetric Hopper Location

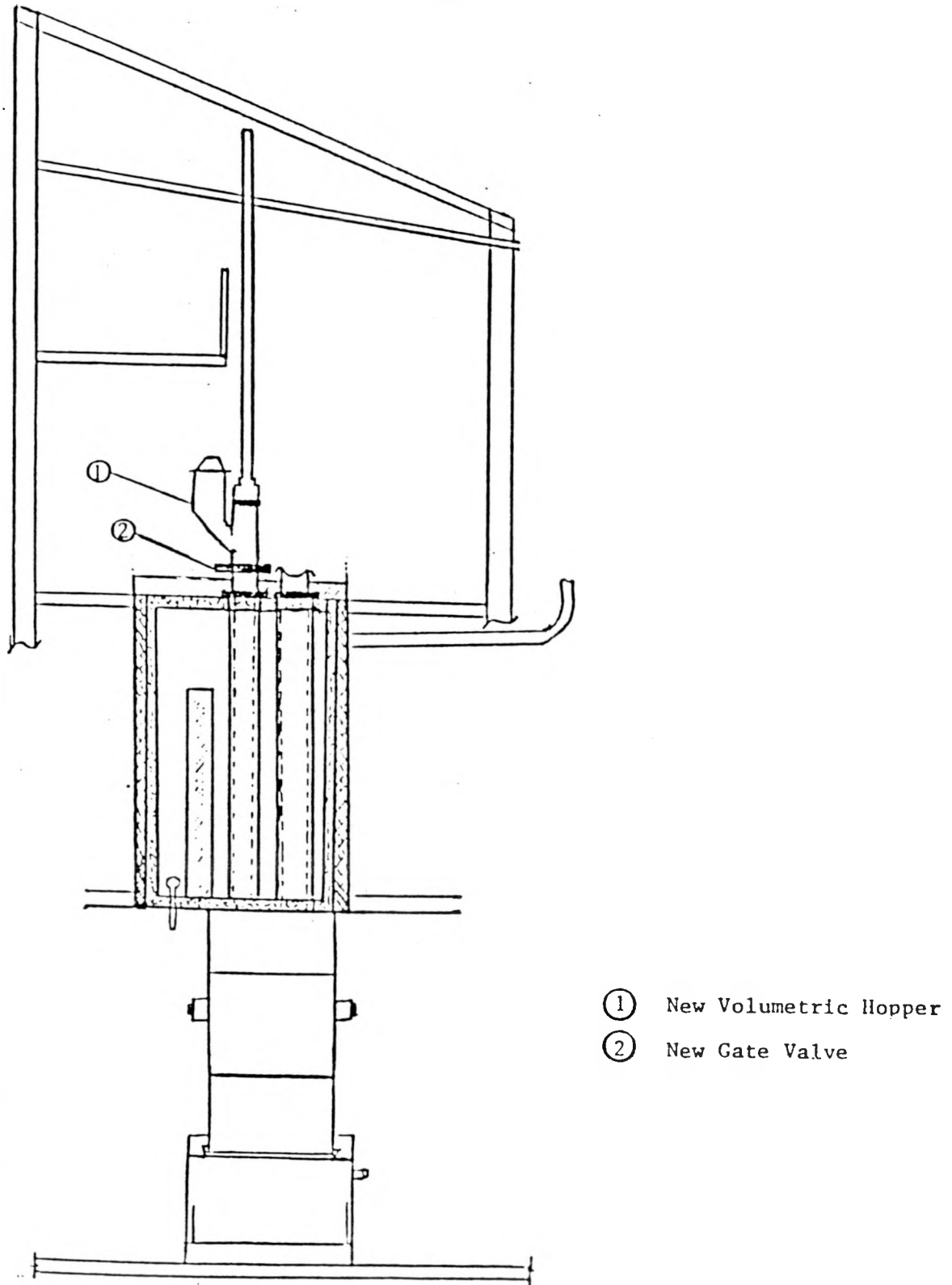
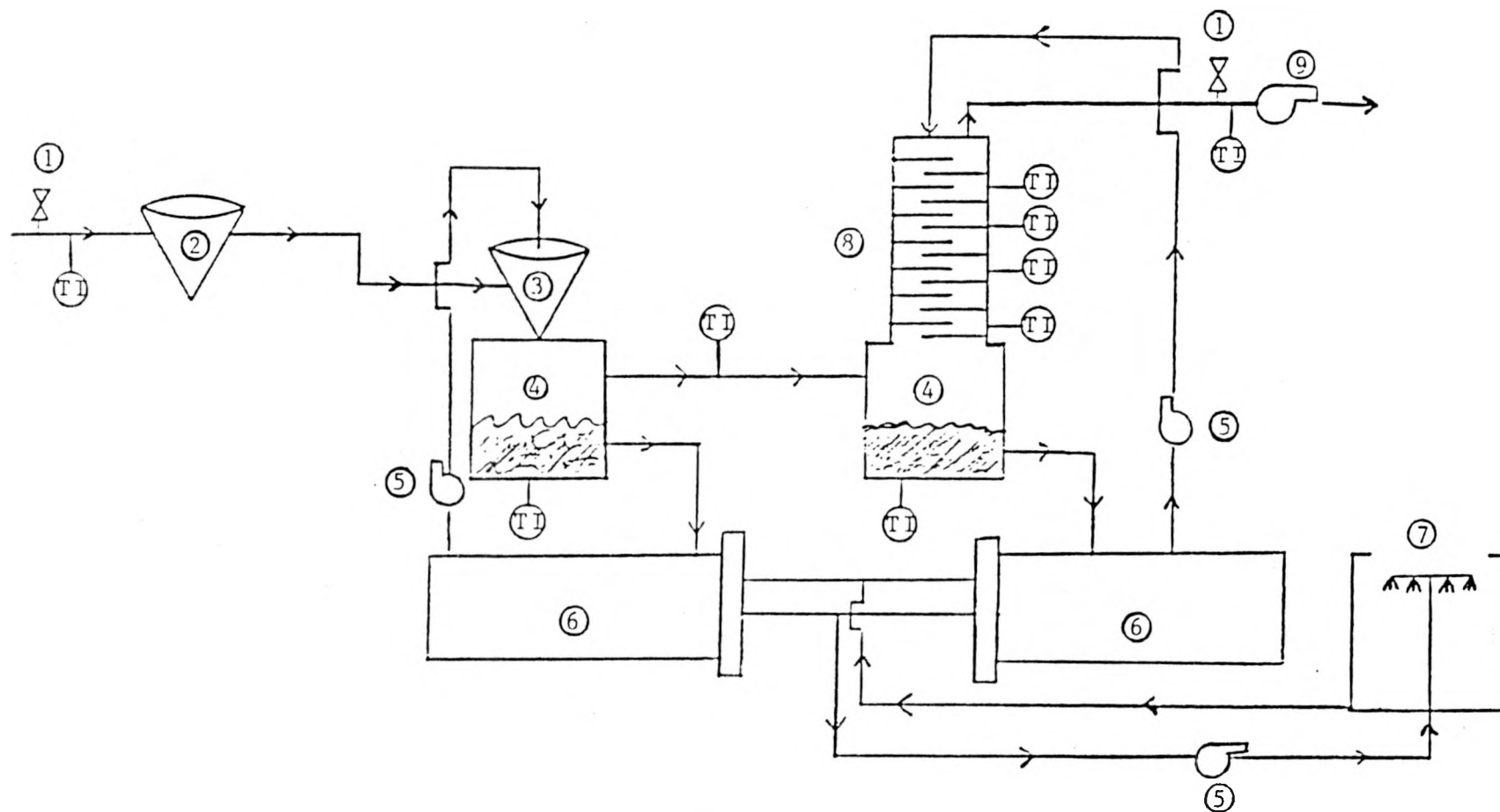


Figure 3. Proposed MGU Condensing System



- ① Gas Sampling Valve
- ② Cyclone
- ③ Venturi Scrubber
- ④ Holding Tank
- ⑤ Liquid Circulation Pump

- ⑥ Heat Exchanger
- ⑦ Cooling Tower
- ⑧ Tray Tower
- ⑨ Vacuum Pump
- ⓐ Temperature Indicator

char from the process is discharged by hydraulic ram into an atmospheric chamber and water quenched.

● FEEDSTOCK SELECTION

A total of five feedstocks will be used during testing. The feedstocks are listed below.

1. UCC Wellmore No. 8 "Ky Blend" -bituminous coal.
2. UCC coal (with different macerals) or Pittsburgh No. 8, (selection to be determined) -bituminous coal.
3. Rosebud Coal -subbituminous coal
4. Mississippi Lignite
5. UCC Wellmore No. 8 coal preparation waste
-1/4" x 0 size fraction.

A proximate and ultimate analysis will be performed on each feedstock used during testing. The maceral composition of each feedstock will also be determined.

● PARAMETRIC TEST CONDITIONS

Reactor Tube Diameter Testing

A total of nine tests will be conducted, with the non-tapered stainless steel reactor tubes. The main objective of this part of testing is to determine the optimum reactor tube diameter for the MGU. Test conditions are summarized in Table I.

The effects of the following operating parameters on product yield and quality will be determined: reactor tube diameter (4, 5, 6, and 8 inches) and bed height (2, 5, and 8 feet). Bituminous coal with an approximate particle size of 1" x 0 will be used for these tests.

From the results of the testing mentioned above, the best operating parameters will be used to determine the effect of coal weathering on the Mild Gasification Process. For the testing, a bituminous coal sample will be taken and separated into three categories: Unweathered, mildly weathered, and highly weathered. The unweathered coal will be prepared by storing one third of the original coal sample under a nitrogen blanket in an airtight container. The mildly weathered coal will be prepared by storing one third of the original coal sample in a 55-gallon drum. The drum and lid will not be airtight and will not be flushed or blanketed with nitrogen. The highly weathered coal will be prepared by taking the remaining one-third of the coal sample and storing it outdoors, exposed to wind, rain, and other normal weather conditions.

MGU Parametric Testing

Thirteen tests will be conducted, ten with bituminous coal, one with subbituminous coal, one with coal preparation waste, and one with lignite. After completion of the initial thirteen tests, there will be

an option to conduct up to ten additional tests with the feedstock and test conditions determined from the results of the initial tests. The tests are summarized in Table 2.

The first nine tests are designed to determine the effects of the following operating parameters on unit performance and on product yield and quality: temperature (1000-1300°F), particle size (1" x 0 and 1/8" x 0), sweep gas (nitrogen and steam), and lime additive 10 wt.% and 20 wt.%). The bituminous coal feedstock used for parametric testing will be chosen based on ultimate and proximate analyses, and maceral components.

From the results of the parametric testing mentioned above, the optimum temperature and particle size will be chosen to test on the second bituminous coal, the subbituminous coal, the lignite, and the coal preparation waste.

After evaluating results from parametric testing and from feedstock testing, up to ten additional tests of interest will be conducted. Parameters will be those with the most prominent effect on product quantity or quality.

Table 1. Reactor Tube Diameter Testing

<u>Test No.</u>	<u>Coal Type</u>	<u>Temp^o F</u>	<u>Particle Size, Inches</u>	<u>Reactor Diameter, Inches</u>	<u>Bed Height, Ft.</u>
1	HVB ^a #1	1100	1 x 0	8	8
2	HVB #1	1100	1 x 0	6	8
3	HVB #1	1100	1 x 0	5	8
4	HVB #1	1100	1 x 0	4	8
5	HVB #1	1100	1 x 0	TBD ^b	5
6	HVB #1	1100	1 x 0	TBD	2
7	HVB #1 (NW) ^c	1100	1 x 0	TBD	8
8	HVB #1 (MW) ^d	1100	1 x 0	TBD	8
9	HVB #1 (HW) ^e	1100	1 x 0	TBD	8

^a HVB = High volatile bituminous coal.

^b TBD = To be determined based on results of previous tests.

^c (NW) = Not Weathered; kept under nitrogen blanket.

^d (MW) = Mildly Weathered; limited exposure to air.

^e (HW) = Highly Weathered; exposed to wind, rain & other normal weather conditions.

Table 2. MGU Parametric Testing

<u>Test No.</u>	<u>Coal Type</u>	<u>Temp⁰ F</u>	<u>Particle Size, Inches</u>	<u>Sweep Gas</u>	<u>Additive</u>
1	HVB #1 ^a	1000	1 x 0	-	-
2	HVB #1	1100	1 x 0	-	-
3	HVB #1	1200	1 x 0	-	-
4	HVB #1	1300	1 x 0	-	-
5	HVB #1	TBD ^b	1/8 x 0	-	-
6	HVB #1	TBD	TBD	N ₂	-
7	HVB #1	TBD	TBD	H ₂ O	-
8	HVB #1	TBD	TBD	-	Lime, 20%
9	HVB #1	TBD	TBD	-	Lime, 10%
10	HVB #2	TBD	TBD	-	-
11	SBT #1 ^c	TBD	TBD	-	-
12	LIGNITE	TBD	TBD	-	-
13	CPW ^d	TBD	TBD	-	-
14-23 ^e	TBD	TBD	TBD	TBD	TBD

^a HVB = High volatile bituminous coal.

^b TBD = To be determined based on results of previous tests.

^c SBT = Subbituminous coal.

^d CPW = Coal preparation waste.

^e Up to 10 optional tests to be conducted. Parameters based on results of previous tests.

● DESIRED EXPERIMENTAL DATA

One objective during parametric testing will be to obtain more detailed information about what is occurring in the MGU during the Mild Gasification Process. Toward this end, the following process and operating data will be collected during each run:

1. Coal temperature vs. time (at the center of the coal bed).
2. Coal temperature vs. time (at the midpoint between the center of the coal bed and the reactor wall).
3. Furnace temperature vs. time.
4. Gas temperature vs. time (at the reactor tube exit).
5. Gas temperature vs. time (between the venturi scrubber and tray tower).
6. Gas temperature vs. time (just before the flare)
7. Feedstock weight.
8. Additive weight.
9. Char weight.
10. Liquid product weight.
11. Non-condensable gas flow rate and total flow.
12. Gas pressure vs. time (at same points gas temperature is measured).
13. Flow rate of the sweep gases.

Also, the operators will record general and specific observations on equipment performance during each test run.

A log book for the collection of the above data will be prepared.

● SAMPLING METHODS AND PRODUCT ANALYSIS

Char samples will be taken at the end of each test run after the char has been discharged, collected, and cooled. The char will be riffled down to a sample size of 10-15 lbs. The char sample will then be crushed and sent for analysis. For each char sample, ultimate analysis, proximate analysis, and calorific value will be determined.

Liquid samples will also be taken at the end of each run. Excess water will be decanted and the liquid thoroughly mixed. A representative sample will be taken and any remaining water removed by heating the liquid to $\sim 110^{\circ}\text{C}$. Any light oils removed during the process will be collected and recombined with the coal oil. For each liquid sample, elemental composition (carbon, hydrogen, nitrogen, sulfur, and oxygen) and molecular weight will be determined. Boiling point distribution and LC- ^1H NMR data will be obtained as required.

Gas samples will be collected periodically from two sample points in the gas stream. The first sample point will be located immediately after the gases exit the reactor tube. Sample point number two will be located ahead of the vacuum pump. The samples will be analyzed for light gases H_2 , O_2 , N_2 , CH_4 , CO , CO_2 , and heavier hydrocarbons.

APPENDIX B:

Test Plan
for
Evaluation of Char as Industrial Boiler/Blast Furnace Fuel

April 14, 1987

Work Performed Under
Contract No. DE-AC21-87MC23289

For:
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia 26505

By:
UCC Research Corporation
of
United Coal Company
Bristol, Virginia 24201

Objective:

The objective is to evaluate the char produced in the Mild Gasification Process Development Unit (MGU) for potential application in end uses by conducting three utilization tests.

Kinds of Utilization Tests:

- (1) Powdered char as industrial and utility boiler fuel.
- (2) Lump char as stoker boiler fuel.
- (3) Lump char as foundry furnace fuel.

Note: Originally it was planned to use a blast furnace for test (3), however, due to uncertain availability of a blast furnace, a foundry furnace will be used instead.

The main purpose of this test is to use the char as a coke substitute. By testing in a foundry furnace, we believe that the primary objectives of the test will be accomplished.

Test Equipment and Locations:

- (1) Industrial and Utility Boiler
Tas-System of TAS-COAL, Magna, Utah.
- (2) Stoker Boiler
Steam boiler of United Coal Company, Bristol, Va.
- (3) Foundry Furnace
Cupola of Tenetek Corporation, Johnson City, Tenn.

Description of Experimental Equipment

(1) TAS - System

A pilot unit of the Tas - System, shown in Figure 1, is a micronized coal combustion system. Coal, 2" x 0, is fed through a screw conveyor into a rotary pulverizer. The pulverizer is belt driven from a vertically positimed, TEFC, 1800 rpm, 30 hp motor. Primary air is supplied as the coal enters the pulverizer. Primary air is introduced in a very turbulent manner allowing particle size reduction of the coal.

The coal particle size is reduced in a single pass to 80% passing 325 mesh. The discharged micronized coal, suspended in the primary air stream, is delivered to the burner head through a steel delivery tube. The secondary and tertiary air streams are mixed with the micronized coal at this point to maximize the combustion efficiency.

(2) Steam Boiler

A 30,000 lb/hr (maximum steam output) stoker boiler will be used. The system consists of a loading hopper which dumps into a storage bunker, the coal is screw fed into a small hopper that discharges to the boiler. At maximum boiler output, the coal feed rate is approximately 1 ton/hr.

(3) Foundry Furnace

The equipment is a cupula with an operating capacity of a 1200 lb coke bed and 75 lb coke charges during operation.

Experimental Tests and Desired Data

(1) Industrial and Utility Boiler Fuel

(A) Test Procedures and Conditions

Two tests will be conducted in the TAS - System. The first test will be with UCC Wellmore No. 8 bituminous coal to establish a baseline set of data. The second test will be with char from UCC Research's MGU. The feedstock for producing the char will be UCC Wellmore No. 8 bituminous coal. The results of the char combustion test will be compared with the results of the coal combustion test to determine the char performance in the TAS-System relative to coal.

Each test will require approximately two 55-gallon drums of sample (~500-600 lbs). The test sample will be fed by screw conveyor from a feed hopper to the rotary mill. Screw rpm will be controlled by pressure and temperature sensors on the combustor. After start-up has been achieved and a stable flame established, the turn down ratio for the fuel will be determined. Then for a fixed feed rate, combustion gas samples, micronized feed samples, and ash samples from the bag house will be collected. Feed rate, flame temperature, flame characteristics, (color, length, shape, etc.), and percent air in primary, secondary, and tertiary air streams will be monitored throughout each test.

Based on analysis of char produced in previous MGU tests, TAS-COAL believes the char "will definitely burn well," in their system. However, should the char not burn satisfactorily in Test 2, an optional Test 3 will be performed using a char/coal blend. The exact char/coal blending ratio will be determined later depending on the results from Test 2.

(B) Desired Experimental Data

- Flame temperture.
- Flame characteristics (color, size, shape).
- Percent air in the primary, secondary, and tertiary. air streams.

- Turn down ratio.
- CO, SO₂, and NO_x, (lbs/ton or lbs/10⁶ Btu).
in combustion gas stream (for given feed rate
and amount of combustion air).
- Ultimate and proximate analysis of feed material.
- Ash analysis (including carbon content of ash).

(2) Stoker Boiler Fuel

(A) Test Procedure and Conditions

For this test the boiler will be operated at one-half capacity to conserve fuel and conduct a longer test. Even at one-half ton/hour the test will be of relatively short duration and will be more qualitative in nature, obtaining only general combustion characteristics and burner response to the char fuel.

To conduct the test, the boiler will be started up and brought to a stable operating condition using coal as the fuel source. After obtaining a stable operating condition with coal, char will be added to the feed bin. The char (1" x 0) will be added to the feed bin only after almost all of the coal has been used. This should allow a smooth and sharp transition to char as the fuel source. General observations to be made during the test run will be boiler firing and response with char as compared to coal. Char and ash samples will be collected before and after the test respectively.

(B) Desired Experimental Data

- Char analysis, including proximate and ultimate analysis, and size distribution.
- Ash composition.
- Boiler temperature, flame appearance (color, size, etc.).
- Char feed rate.
- Turn down ratio.
- Visual emissions.

(3) Foundry Furnace Fuel

(A) Test Procedure and Conditions

Feedstock for the char will be chosen based on the fuel requirements requested by Tenetec. At this time, those requirements have not been specified. Typical cokes used in foundries have the following composition range:

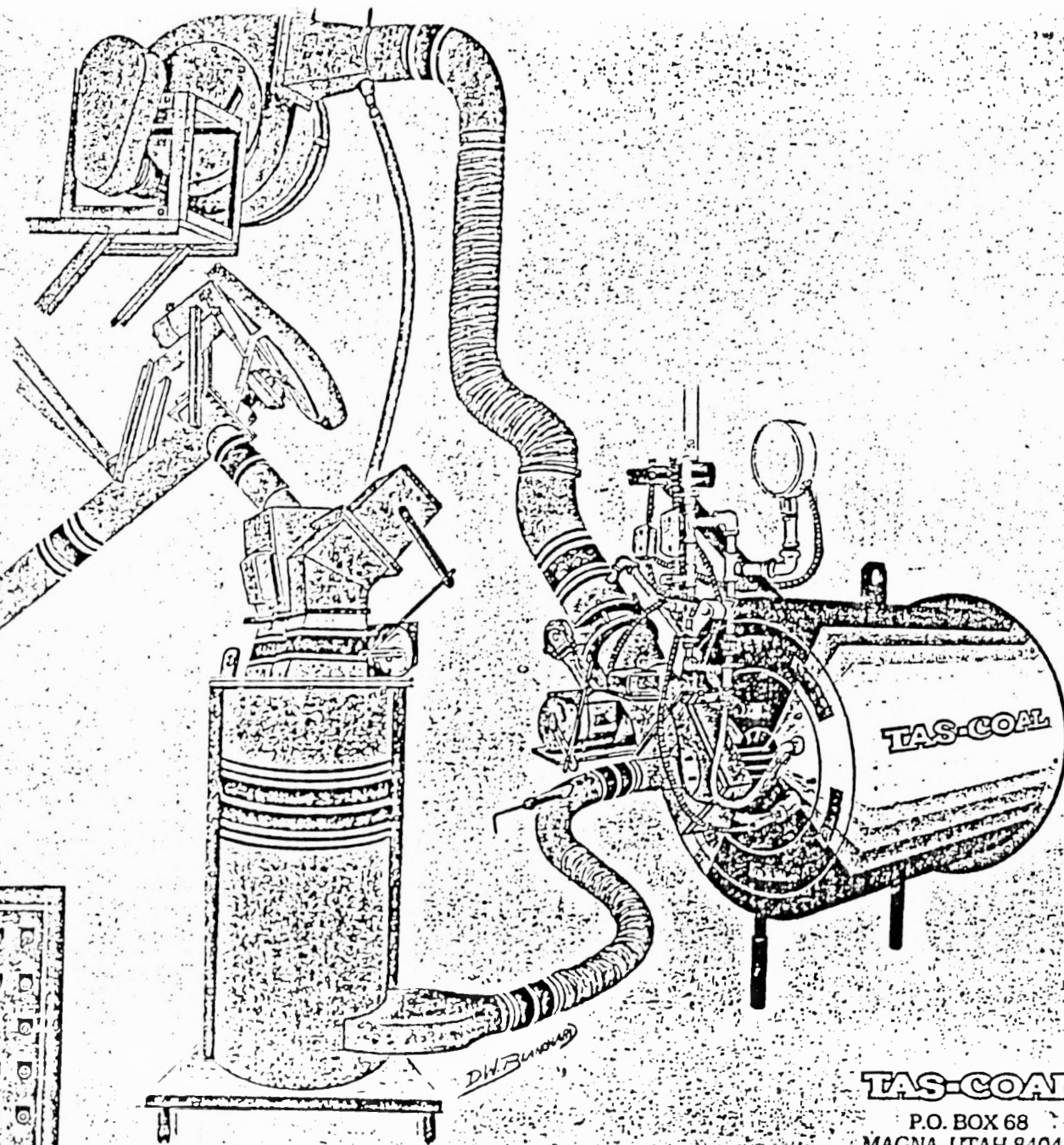
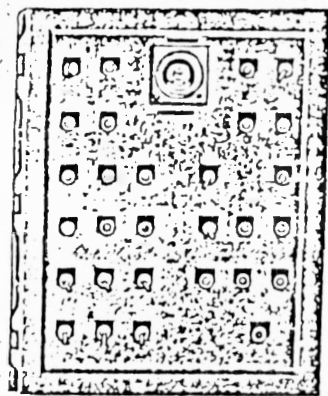
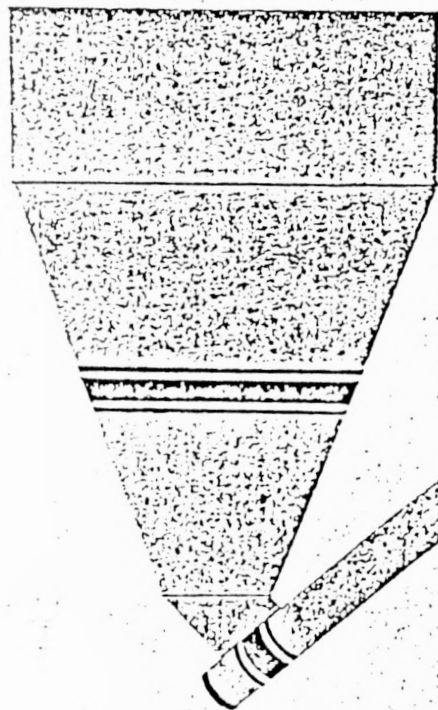
Sulfur	0.7 - 1.3%
Ash	6.0 - 13.0%
Moisture	3.0 - 8.0%
Volatile Matter	0.7 - 1.6%

The sulfur and ash requirements are the most critical and the ones which are dependent on the parent coal. The sulfur and ash requirement for Tenetec is generally close to 0.7% and 6.0% respectively. Therefore, a low ash, low sulfur coal will most probably be used during MGU production of the char. The test char will first be screened to remove the fine material (< 1 inch). Only the + 1 inch char will be used. Approximately 1.5 tons of char will be required for a one day test, 1200 lbs for the bed and 1875 lbs for twenty-five, 75 lb charges. These amounts represent typical one day fuel use for Tenetec. Normal operating procedures used by Tenetec will be followed during the test.

(B) Desired Experimental Data

- Cupola temperatures.
- Char analysis including ultimate and proximate analysis, particle size distribution, and char strength.
- Gaseous emissions (SO₂) during test.
- Heat produced by char fuel.
- Smoke/particulates in the bag house.
- Comparison data gathered from normal operations using coke.

TAS-COAL



TAS-COAL

P.O. BOX 68
MAGNA, UTAH 84044

Enquiries please call:
Kent White

1-801-250-2908

Figure 1. TAS-SYSTEM Unit