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DEVELOPMENT OF MILD GASIFICATION PROCESS

Quarterly Report
for the period July-September, 1987

By

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November, 1987

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

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EXECUTIVE SUMMARY

Reactor diameter optimization testing was completed. Tests were conducted with 4-, 6-, and 8-inch non-tapered stainless steel reactor tubes. The tests have demonstrated several points. First, straight reactor tubes (stainless steel pipe) can be used in place of tapered tubes in the MGU. Also, the tests have shown that the increased heating rate obtained with the smaller diameter reactor tubes resulted in only a slight increase in the condensible yield. Finally, char and condensible quality was relatively unchanged as a function of reactor diameter.

Tests were also conducted using hot nitrogen sweep gas. The tests showed coal residence time could be reduced to as short as one hour by introducing 10 scfm of hot (~800°F) sweep gas. The effect of the hot sweep gas on product yield and quality is not known at this time due to operation problems encountered during the tests.

Based on the result of the reactor diameter optimization tests and the hot nitrogen sweep gas tests, it was decided that the MGU would be modified to be a two reactor system. Six inch diameter reactor tubes will be used with provisions made for heating sweep gas to 1000-1100°F. The char chamber will be redesigned to have water-cooled walls, controlled water-spray and nitrogen seal, and a locking cap at the bottom for sealing purposes. This design eliminates the need for a water seal in the char chamber. The coal feeding system, reactor support, and condensing system have also been redesigned for the modified MGU.

TABLE OF CONTENTS

		Page
INTRODUCTION		1
Task 1.	Test Plan for Optimization of the Mild Gasification Process	3
	Objective	3
	Discussion	3
Task 2.	Optimization of the Mild Gasification Process	3
	Objective	3
	Discussion	3
Task 3.	Evaluation of Char and Char/Coal Blends as an Industrial Boiler/Blast Furnace Fuel	11
	Objective	11
	Discussion	11
Task 4.	Analyze Test Data and Prepare Final Report	11
	Objective	11
	Discussion	11
REFERENCES		12

LIST OF FIGURES

Figure No.		Page
1.	Existing Mild Gasification Process Development Unit (MGU)	2
2.	Reactor Configuration For Hot N_2 Sweep Gas Tests	13
3.	Location Of Reactor Tube Partial Blockage During 4- and 6-Inch Reactor Tube Tests	14
4.	Coal Heating Rates For The 4-, 6-, and 8-Inch Diameter Reactor Tube Tests	15
5.	Distillation Curve Of Raw Pyrolysis Liquids Derived From UCC Wellmore No. 8 Bituminous Coal	16
6.	Location Of Unreacted Coal For Test #10	17
7.	Coal Heating Rates For The Hot N_2 Swept And Unswept Cases	18
8.	Furnace Layout For Modified MGU	19
9.	Modified MGU Flowsheet	20
10.	Liquid Recovery System For Modified MGU	21
11.	Char Receiving And Quenching Chamber For Modified MGU	22
12.	Reactor Tube Details For Modified MGU	23
13.	Floor Plan For Third Floor Of Modified MGU	24
14.	Equipment Layout For Third Floor Of Modified MGU	25

LIST OF TABLES

Table No.		Page
I.	Feedstock Analysis For MGU Tests	5
II.	Test Conditions For Reactor Diameter Optimization And Hot N_2 Sweep Gas Tests	6
III.	Product Yields For Reactor Diameter Optimization And Hot N_2 Sweep Gas Tests	8
IV.	Product Analysis For Reactor Diameter Optimization Tests	9

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 x 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 x 10^9 tons. At the current annual U.S. coal production rate of about 900 x 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, UCC Research Corporation (UCCRC) 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 is 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 four tasks; Task 1-Test Plan; Task 2-Optimization of Mild Gasification Process; Task 3-Evaluation of Char and Char/Coal Blends as a Boiler/Blast Furnace Fuel; and Task 4-Analysis of Data and Preparation of Final Report. Task 1 has been completed while work continued on Task 2.

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

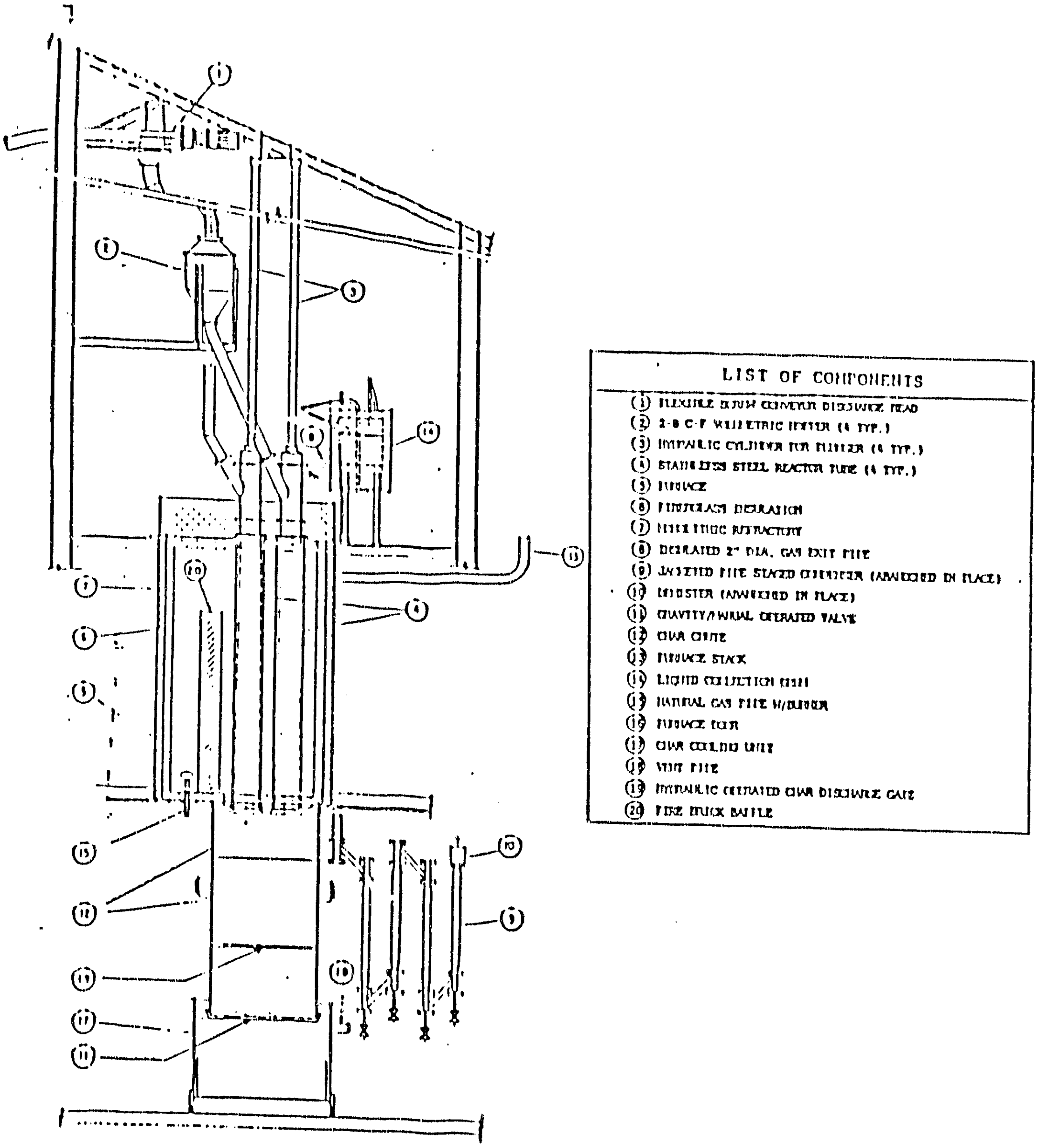

Figure 1. Existing Mild Gasification Process Development Unit (MGU)

Task 1. Test Plan

Objective

The objective was to develop a test plan for optimizing the mild gasification process.

Discussion

The test plan has been completed and was submitted to the Department of Energy in March, 1987.

Task 2. Optimization of the Mild Gasification Process

Objective

The objective of this task are 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 enough char and hydrocarbons in order to evaluate these products in various commercial applications.

Discussion

Reactor diameter optimization tests were completed during the reporting period. Also, two tests were conducted to determine the effect of hot nitrogen sweep gas on coal heating rate as well as on char and liquid quality. Although the feedstock for both sets of tests was the clean coal product, particle size of 1 inch x 0, from United Coal Company's Wellmore No. 8 coal preparation plant, the coal used for the hot nitrogen sweep gas tests was not obtained at the same time as the coal for the reactor diameter optimization tests. Therefore, the properties are slightly different. The proximate and ultimate analyses of the feedstocks are shown in Table I.

For each reactor diameter optimization test, the following test procedure was used: The furnace was preheated to temperature (1100°F) and the system purged with nitrogen. A weighed amount of coal was then charged into the reactor tube. Coal temperature was monitored with two thermocouples. One thermocouple was placed approximately four-feet high in the coal bed and one inch from the outside wall of the reactor. The second thermocouple was placed approximately four feet high in the coal bed and in the center of the coal bed (one-half the diameter of the reactor tube from the outside of the tube wall). It should be noted that the thermocouples have a certain degree of flexibility and the exact positioning of the thermocouples can vary from test to test. Therefore, we are recommending in the MGU modifications that the thermocouples be inserted laterally through the side to the reactor 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 ~ 2 hours after the maximum temperature (1100°F) was reached. After decanting excess water, the condensibles were stirred and a representative sample obtained for analysis. The analysis sample was split into two portions; a Dean-Stark distillation was performed on the first portion to determine the amount of water remaining in the "decanted" condensibles, while the second portion was dried and analyzed for carbon, hydrogen, nitrogen, oxygen, sulfur, and average molecular weight. The char was allowed to cool in the reactor tube and was discharged the next morning. The char was riffled to a sample size of ~ 30 lbs. for proximate and ultimate analysis.

The hot nitrogen sweep gas tests were conducted using a 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. For the first hot nitrogen sweep gas test, the following test procedure was used: The furnace was preheated to temperature (1100°F) and the system purged with hot nitrogen (~800°F). The nitrogen flow was adjusted to 5 scfm, and a weighed amount (55.0 lbs.) of coal was charged into the system. The nitrogen flow rate was then increased to 10 scfm. Coal temperature was monitored with two thermocouples, one placed four feet high in the coal bed and one inch from the wall of the reactor, and the other four feet high in the coal bed and in the center of the reactor (one half diameter of the reactor tube from the outside of the reactor wall). As the coal temperature in the center of the coal bed approached 1100°F, the furnace and hot nitrogen sweep gas were turned off. Condensibles were collected 2 hours after maximum temperature (1087°F) was reached. The char was allowed to cool in the reaction tube and was discharged the next morning.

The procedure for the second hot nitrogen sweep gas test was similar, except that there was not any nitrogen flow through the reactor tube during the coal charging process. The conditions for all tests conducted to date are summarized in Table II.

During the reactor diameter tests with the 4-inch reactor tube, 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 on Tests 4-6. During the last 4-inch test (Test 7), a probe was periodically inserted through the top of the gas-exit manifold into the reactor to determine, if possible, when and how the char-like bridge was formed. By using the probe, it could not be determined when or if the bridge was formed; but an inspection of the reactor after the test showed that the bridge had formed. However, the rodding action of the probe had apparently kept the center of the reactor clean and this was probably the reason for the increased yield in Test 7.

Table I. Feedstock Analysis For MGU Tests

Ultimate Analysis, dry wt.%	W#8A[1]	W#8B[2]
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
Proximate Analysis, dry wt.%*		
Ash	7.69	7.31
Volatile Matter	30.64	31.67
Fixed Carbon	61.67	61.02
*As-Received Moisture	3.94	7.69

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

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

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

Test No.[1]	Reactor Diameter (Inches)	Furnace Temp. (°F)	Final Bed Temp. (°F)	Hot Nitrogen Sweep (scfm)	Nitrogen Purge During Charging
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]Test 1-9 were reactor diameter optimization tests. Tests 10 and 11 were hot N_2 sweep gas tests.

The formation of the char-like bridge was also observed during reactor diameter optimization test with 6-inch reactor. In a procedure similar to the one used during Test 7, a probe was periodically inserted through the top of the gas-exit manifold into the reactor to determine, if possible, when the bridge was formed. However, it could not be ascertained when the bridge was formed. 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.

Proximate (dry wt.%)	
Ash	22.06
Volatile Matter	11.41
Fixed Carbon	64.51

Ultimate Analysis (dry wt.%)	
Carbon	70.68
Hydrogen	2.77
Nitrogen	1.44
Oxygen	1.88
Sulfur	0.72

Ash Composition (dry wt.%)	
SiO_2	84.44
Al_2O_3	4.70
TiO_2	0.61
CaO	0.23
K_2O	0.57
MgO	0.21
Na_2O	0.19
Fe_2O_3	4.01
P_2O_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 3 shows where the bridge formed and the area that sand was present. Aside from the bridge formmation 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 III. As shown in Table III, the condensible yields during the reactor diameter optimization tests were slightly increased as reactor tube diameter was decreased (heating rate was 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 IV.

Table III. Product Yields For Reactor Diameter Optimization And Hot N_2 Sweep Gas Tests

Test No.	Reactor Diameter, Inches	Hot N_2 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.

Table IV. Product Analysis For Reactor Diameter Optimization Tests

Char Analysis For Reactor Diameter Tests

Sample	Ash	Volatile Matter	Fixed Carbon	C	H	N	S	O	Heating Value, BTU/lb
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

Liquid Analysis For Reactor Diameter Tests

Sample	C	H	N	S	O	H/C Atomic Ratio	Molecular Weight
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

The reactor diameter optimization tests have demonstrated several points. First, straight reactor tubes can be used in place of tapered tubes in the MGU. Also, the tests have shown that the increased heating rate obtained by using smaller diameter reactor tubes results in only a slight increase in the liquid yield. Thus, it appears that heating rate, in the range studied here (~ 2.5-32°F/min), has only a minor effect on liquid yield. Finally, the product analyses show 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.

Distillation curves were obtained for coal liquids produced in UCCRC's mild gasification unit and for coal liquids produced in one of Coalite's test retorts. The MGU coal liquids were produced during recent reactor diameter testing (Test #7-4 "Ø) using a 4-inch diameter reactor tube. Both samples were produced with Wellmore No.8 clean coal feedstock. Figure 5 shows that the MGU coal liquids have similar amount of material in the naphtha boiling range (<350°F) like the Coalite coal liquids, but less material in the diesel boiling range (350-650°F). The MGU coal liquids do have more high boiling components than the Coalite liquids.

The fraction of each cut (naphtha, diesel, and heavy bottom) is shown below.

	Naphtha Fraction	Diesel Fraction	Heavy Bottoms
MGU Coal Liquids	8%	32%	60%
Coalite Liquids	7%	45%	48%

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 7). This is equivalent to an average heating rate of 16.1°F/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 the accurate determination of condensible and char yields as well as accurate char analysis. Analytical work on the condensibles has not been completed yet.

A physical examination of the lump char showed that the char looked more porous than char produced with no nitrogen sweep. It also appeared that the nitrogen gas was fairly well distributed through the bed.

The second hot N_2 sweep gas test (Test 11) was conducted to see if the results of Test 10 could be duplicated and to see if the coal could be prevented from being carried out of the furnace zone. The procedure for Test 11 was similar to Test 10 except that there was no nitrogen purge during the coal charging process. 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 7). 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.

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 reactor system. The reactors will be 6-inch diameter, straight (not tapered) type 309 schedule 40 stainless steel pipe - the same specifications that were used for the single reactor tests. Sweep gas (nitrogen or recycle flare gas) will be preheated by passage through two, 8-inch diameter pipes in the furnace. Gas will be injected at the top of the pipes and heated to 1000-1100°F for injection into the bottom of the 6-inch reactor tubes. The position of the two reactor tubes will be farthest from the burners while the sweep gas heating tubes will be nearest the burners (see Figure 8). The reactor tubes and the sweep gas heating tubes will be supported from the bottom of the furnace and will expand toward the top. This will eliminate the leaking problems encountered with the original design. The char hopper will also be redesigned. It will have water-cooled walls and will have a locking cap at the bottom for sealing purposes. The locking cap will be similar to those already in place on the existing volumetric hoppers. The locking cap will eliminate the water-seal gate of the original design. The details of the components to be modified, the MGU flowsheet, and equipment layout are shown in Figures 9-14.

Task 3. Evaluation of Char and Char/Coal Blends as an Industrial Boiler/Blast Furnace Fuel

Objective

The objective of the 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 boiler, stoker coal boilers, and as a replacement for coke in foundry/blast furnaces.

Discussion

No work scheduled during this reporting period.

Task 4. Analyze Test Data and Prepare Final Report

Objective

The objective of the task is to analyze the test data generated during MGU testing and char evaluation. The performance of the individual process elements and overall process, including potential end uses for char, will be evaluated as evidence by those data. On the basis of this evaluation, recommendations shall be made regarding further research and/or development of this mild gasification process.

Discussion

No work scheduled during this reporting period.

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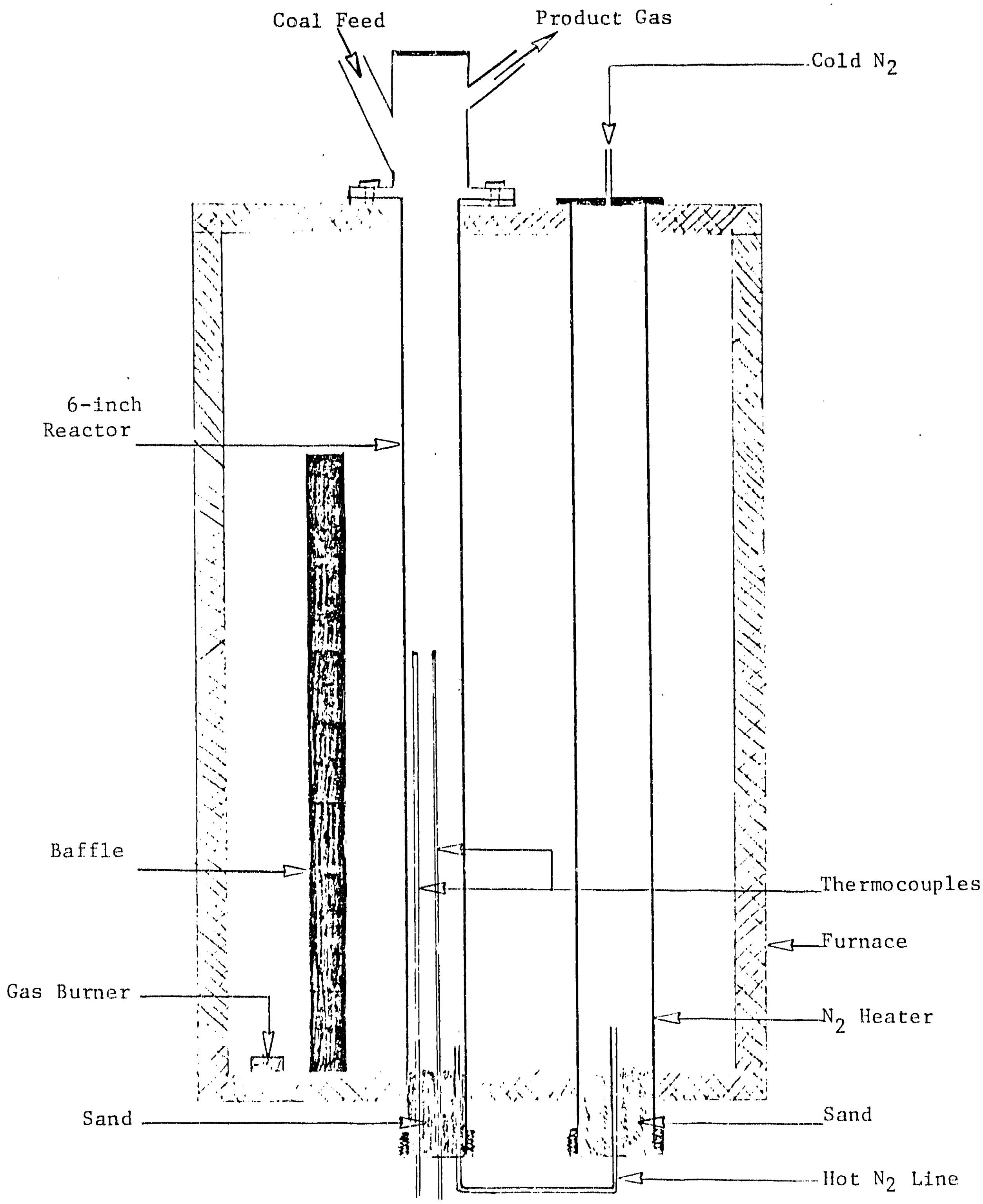

Figure 2. Reactor Configuration For Hot N_2 Sweep Gas Tests

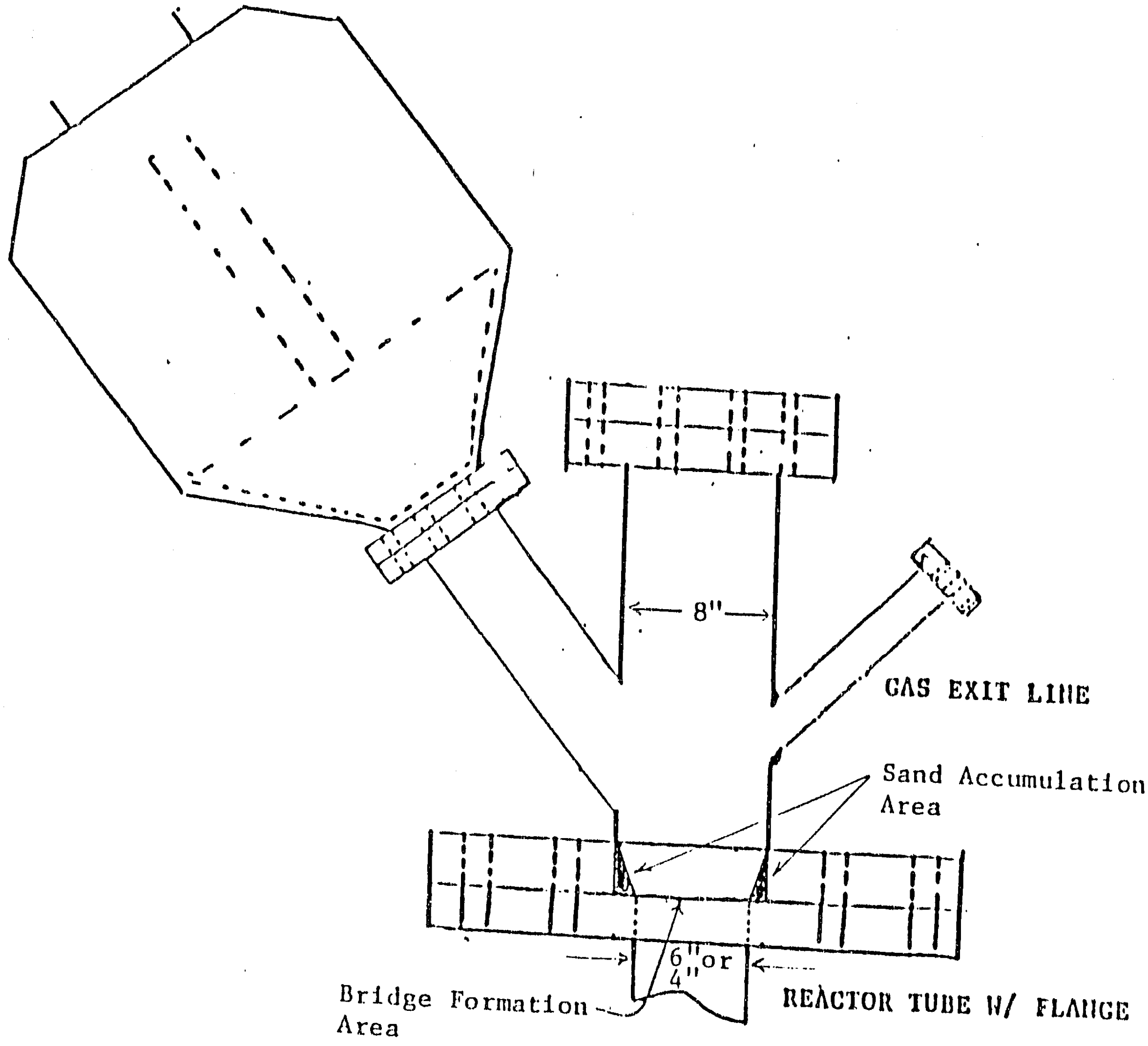

Figure 3. Location Of Reactor Tube Partial Blockage Due To Bridge Formation During 4- And 6-Inch Reactor Tube Tests

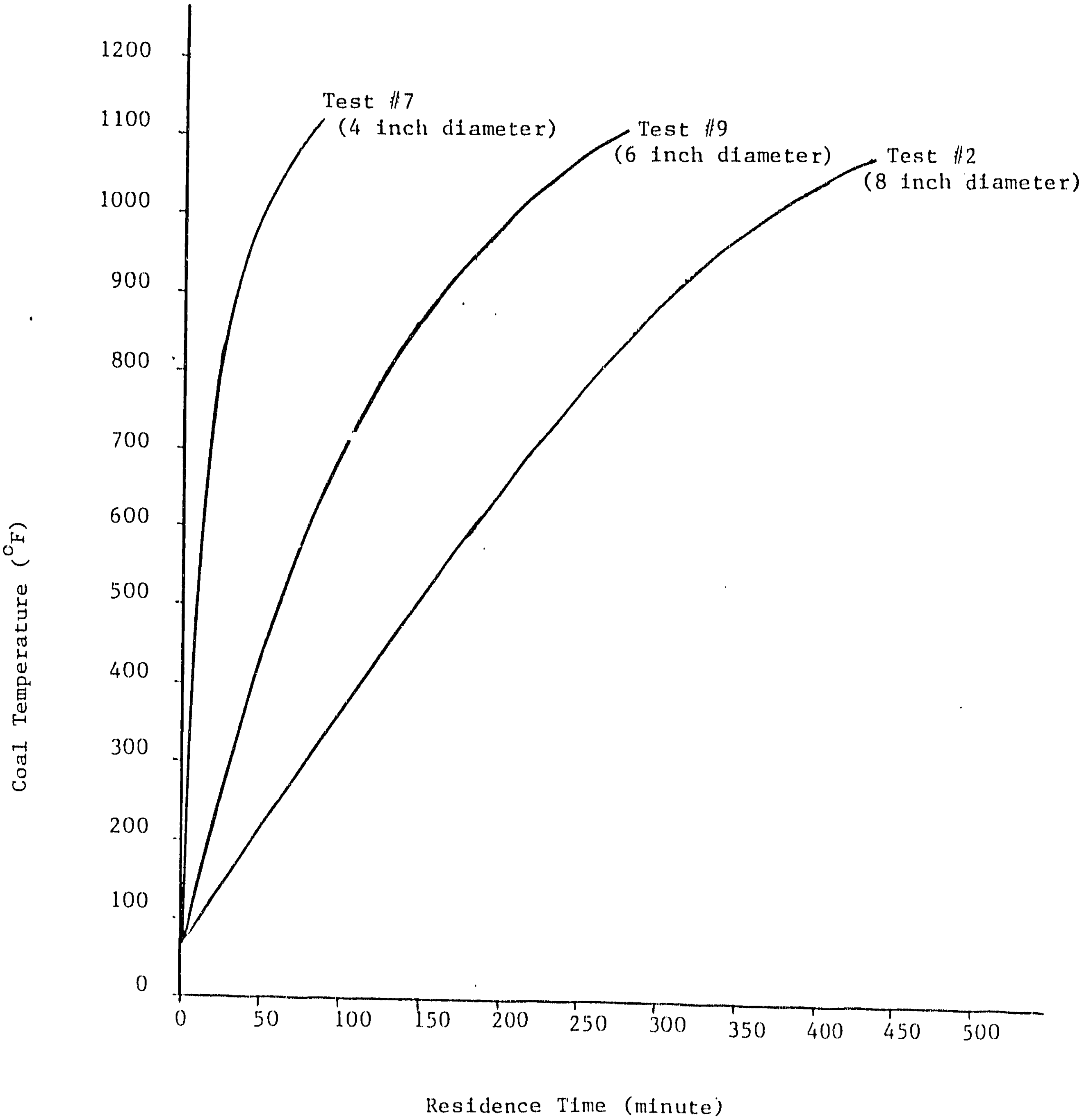

Figure 4. Coal Heating Rates (as measured in the center of the reactor tubes) for the 4-, 6-, and 8-, inch Diameter Reactor Tube Tests

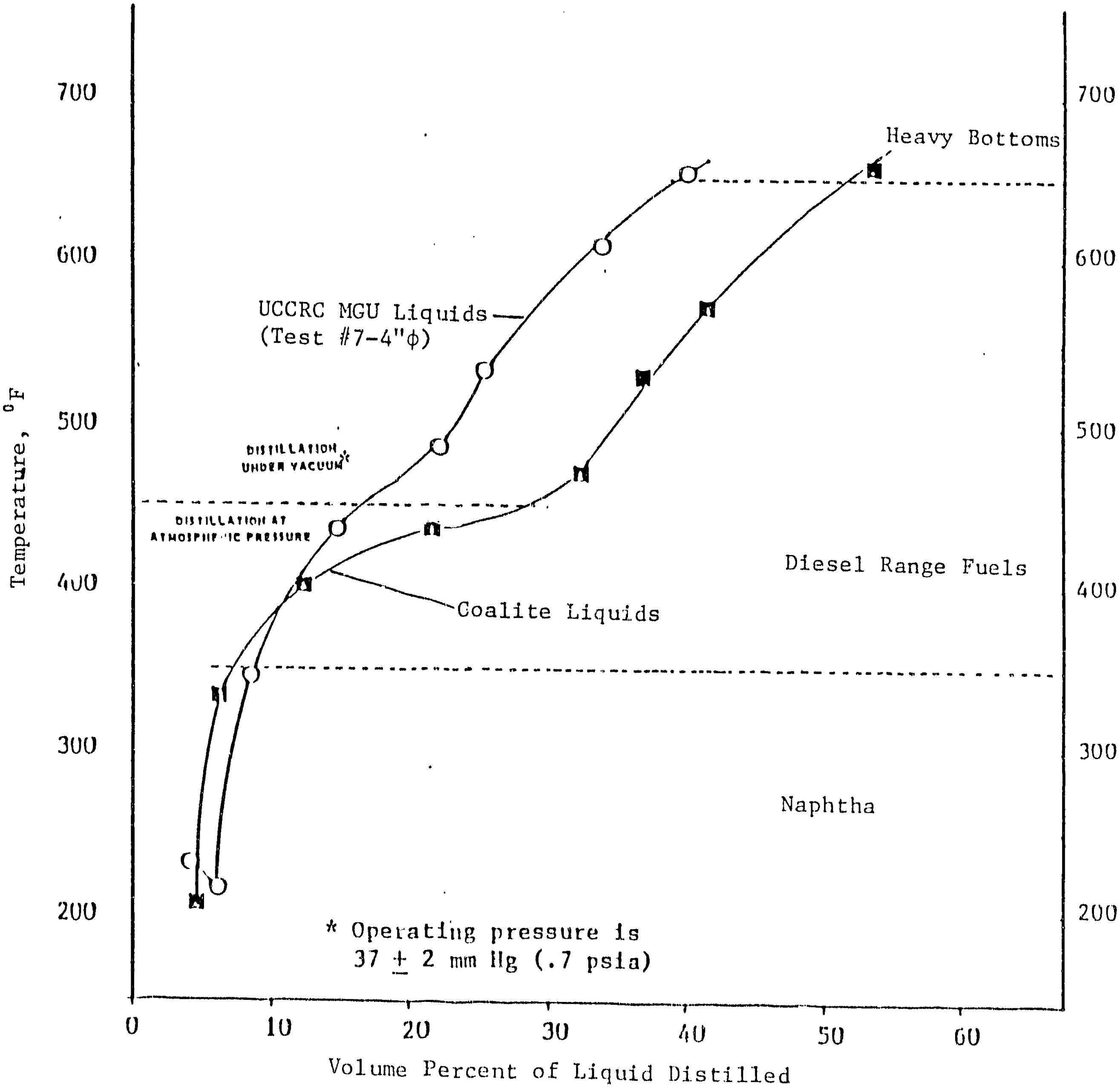

Figure 5. Distillation Curve of Raw Pyrolysis Liquids Derived from UCC Wellmore No. 8 Bituminous Coal

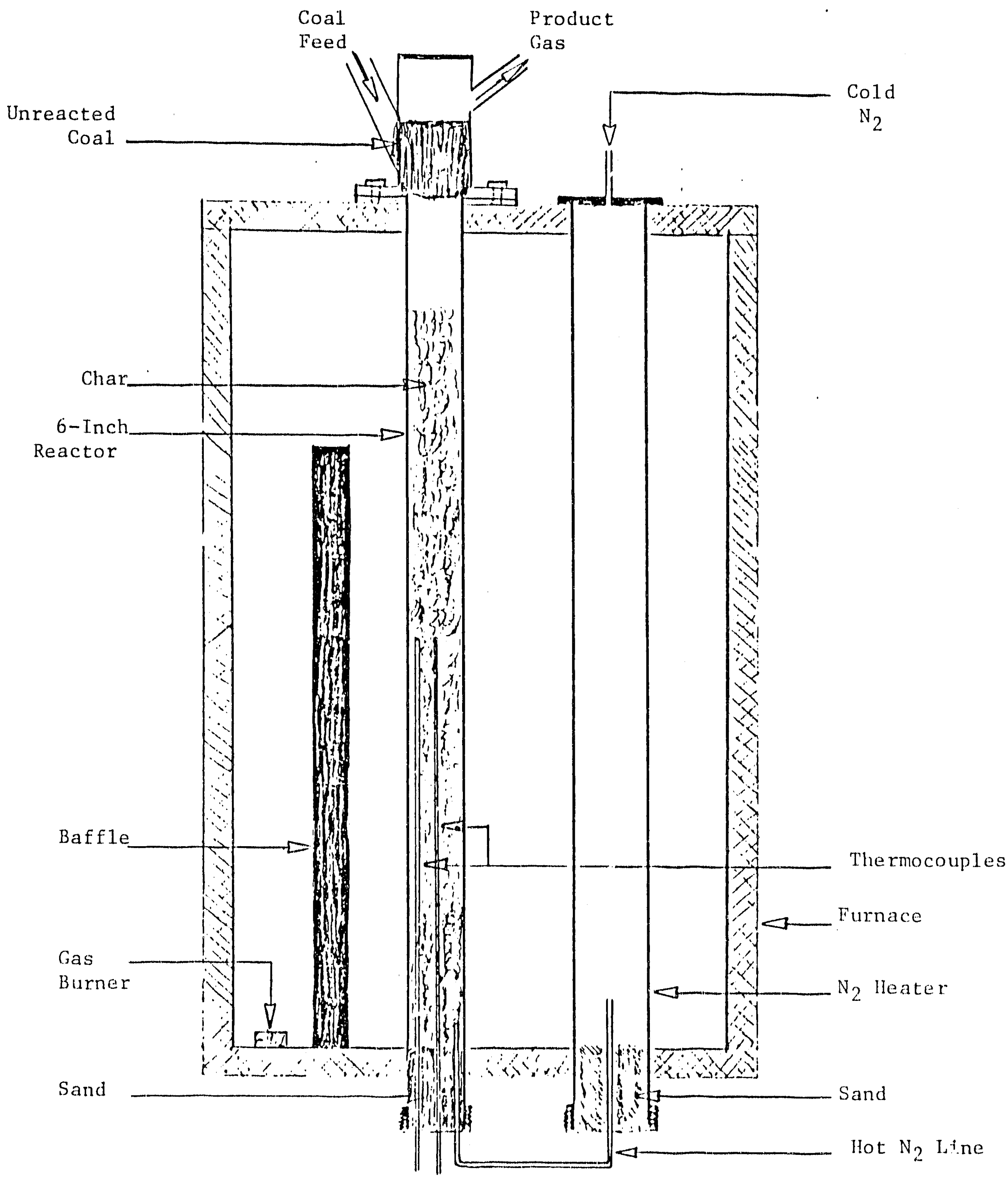

Figure 6. Unreacted Coal Location
For Test #10

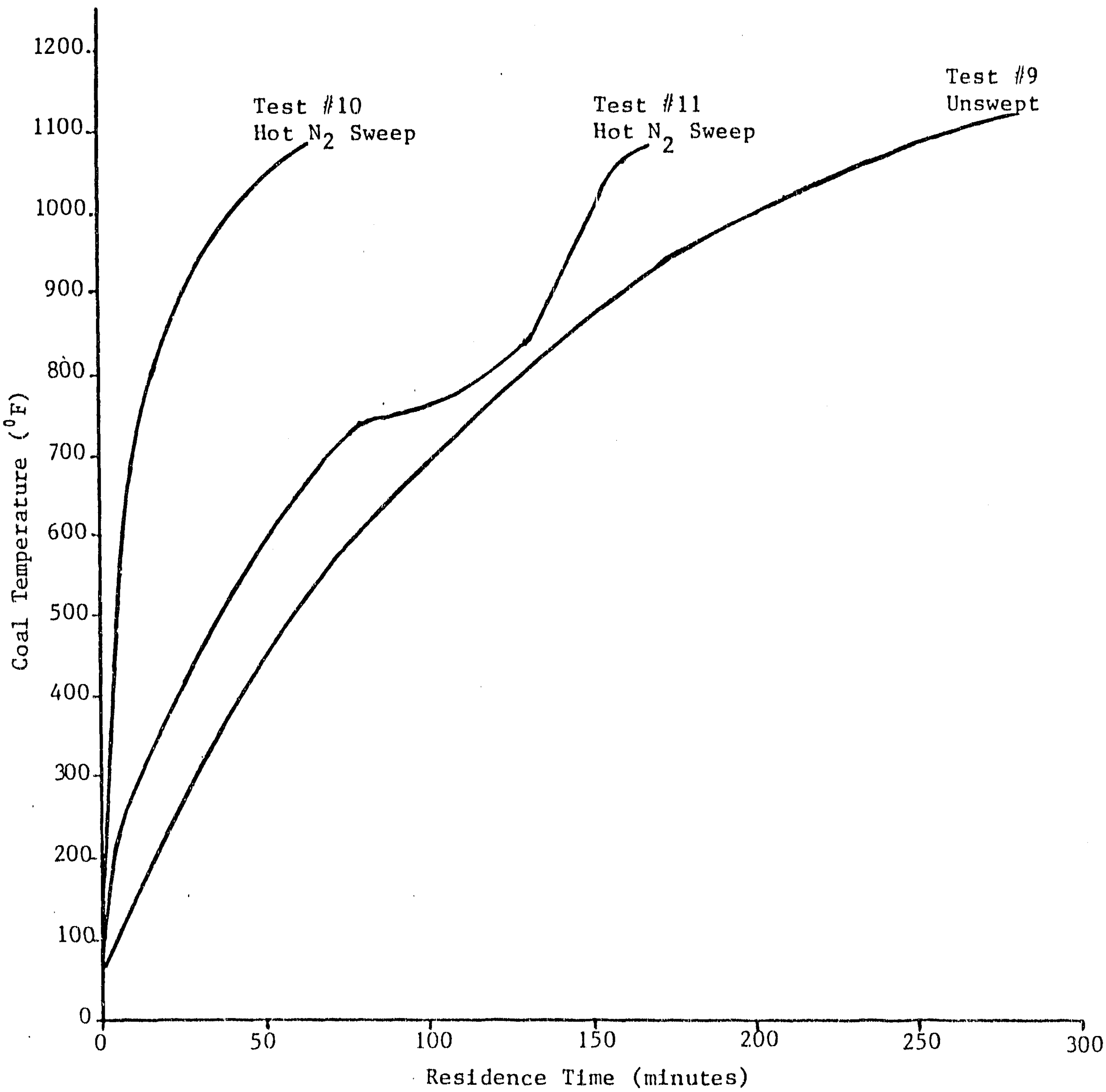

Figure 7. Coal Heating Rates (as measured in the center of the 6-inch diameter reactor tube) for the Hot N_2 Swept and Unswept Tests

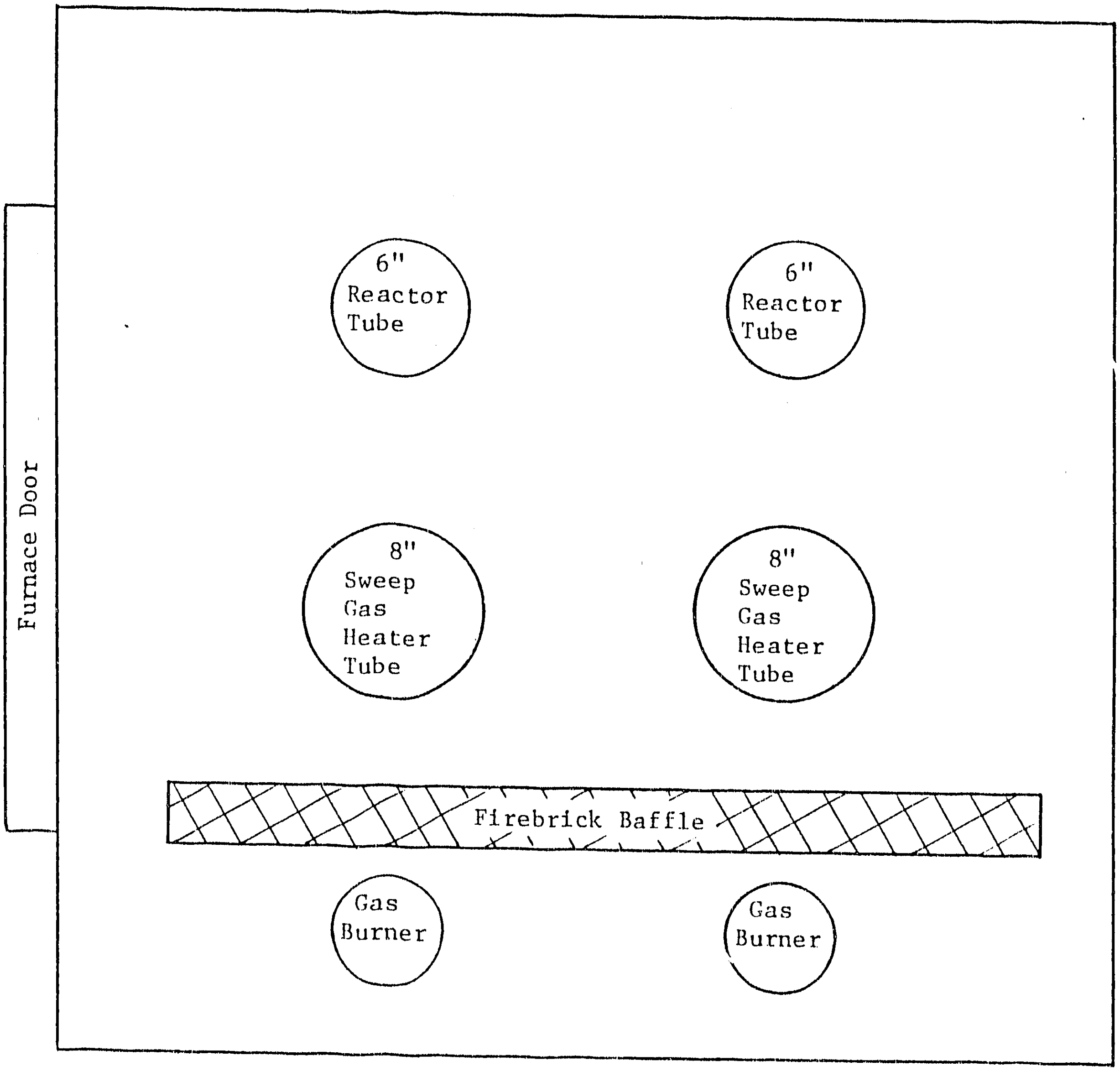

Figure 8. Furnace Layout For Modified MGU

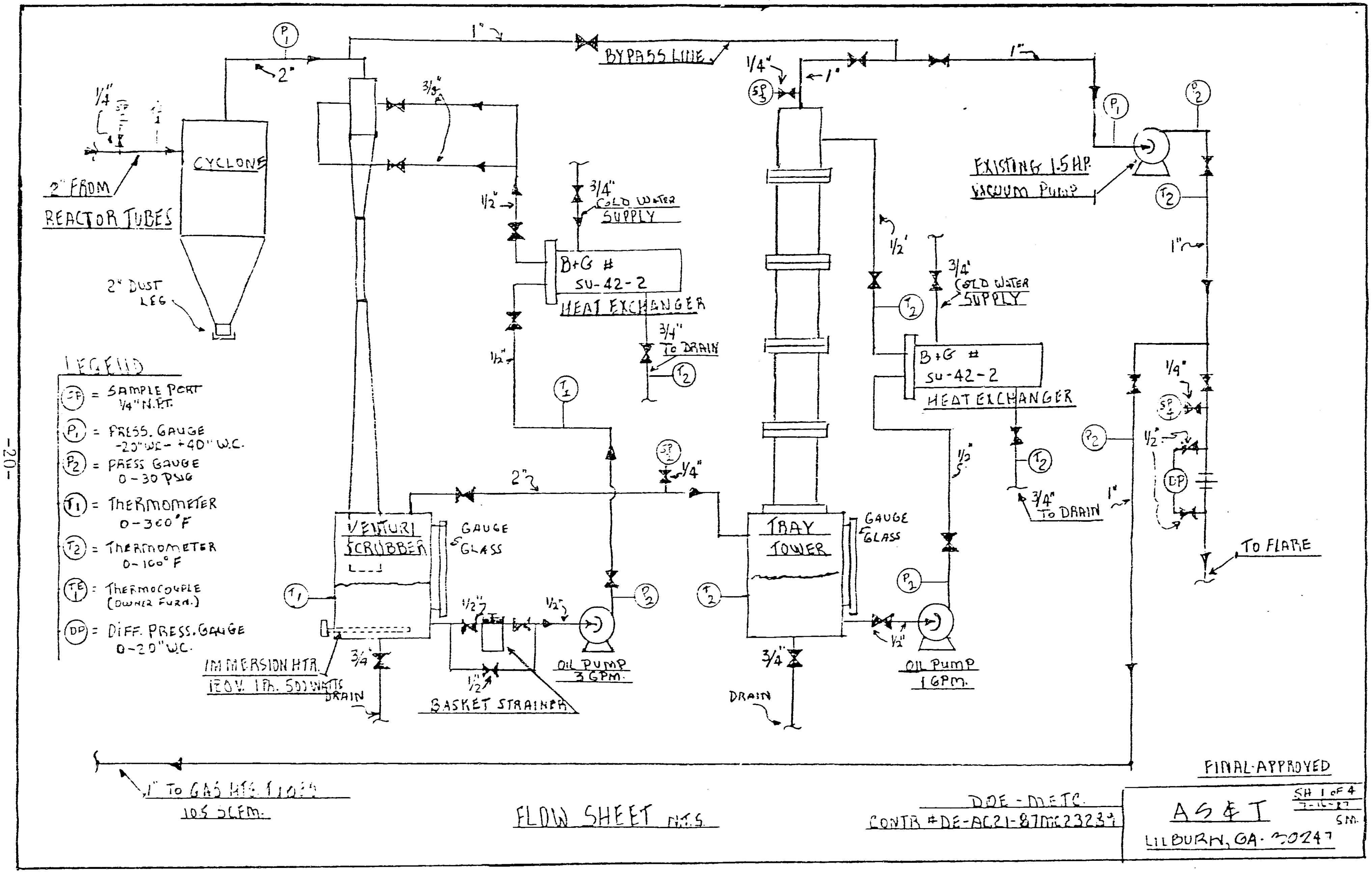

Figure 9. Modified MGU Flowsheet

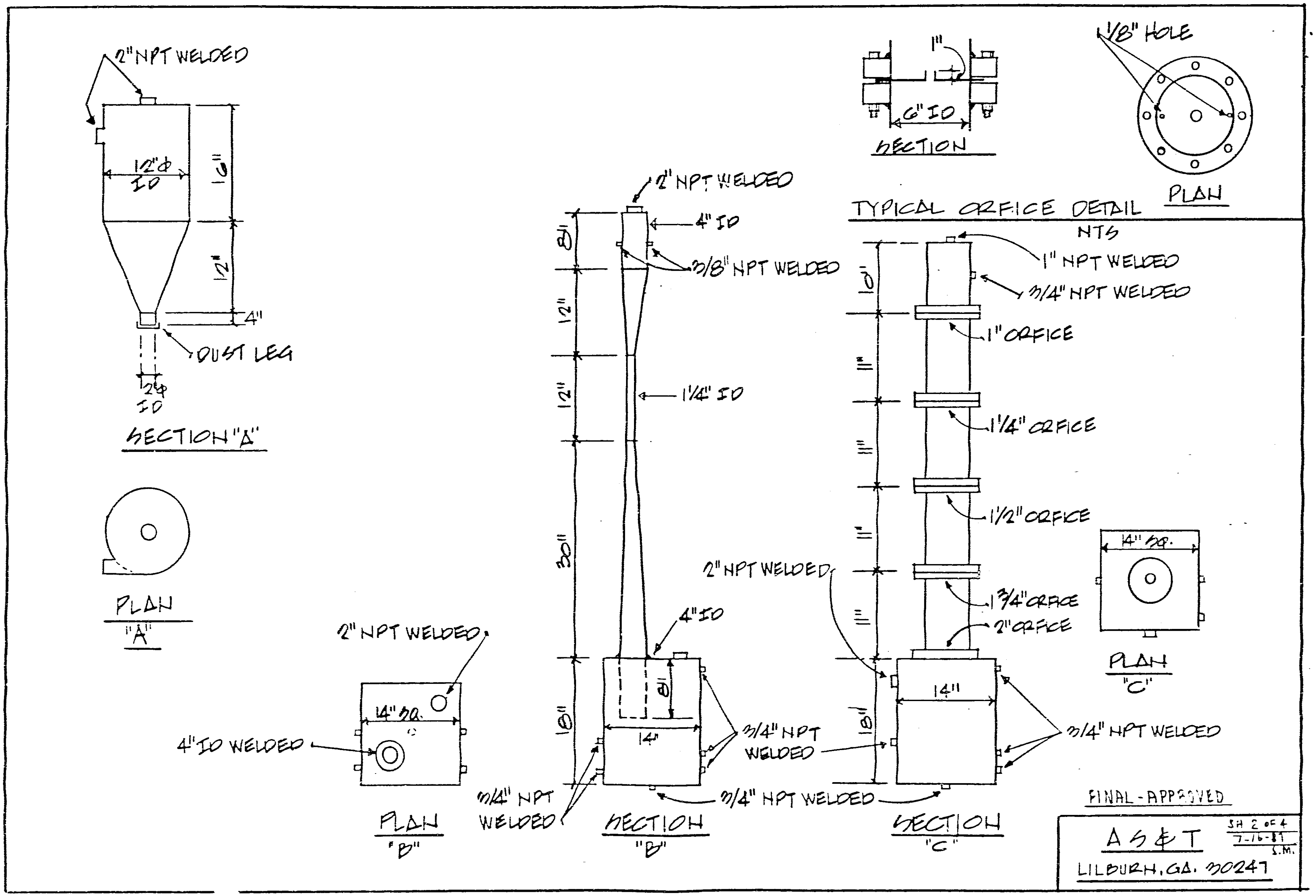

Figure 10. Liquid Recovery System For Modified MGU

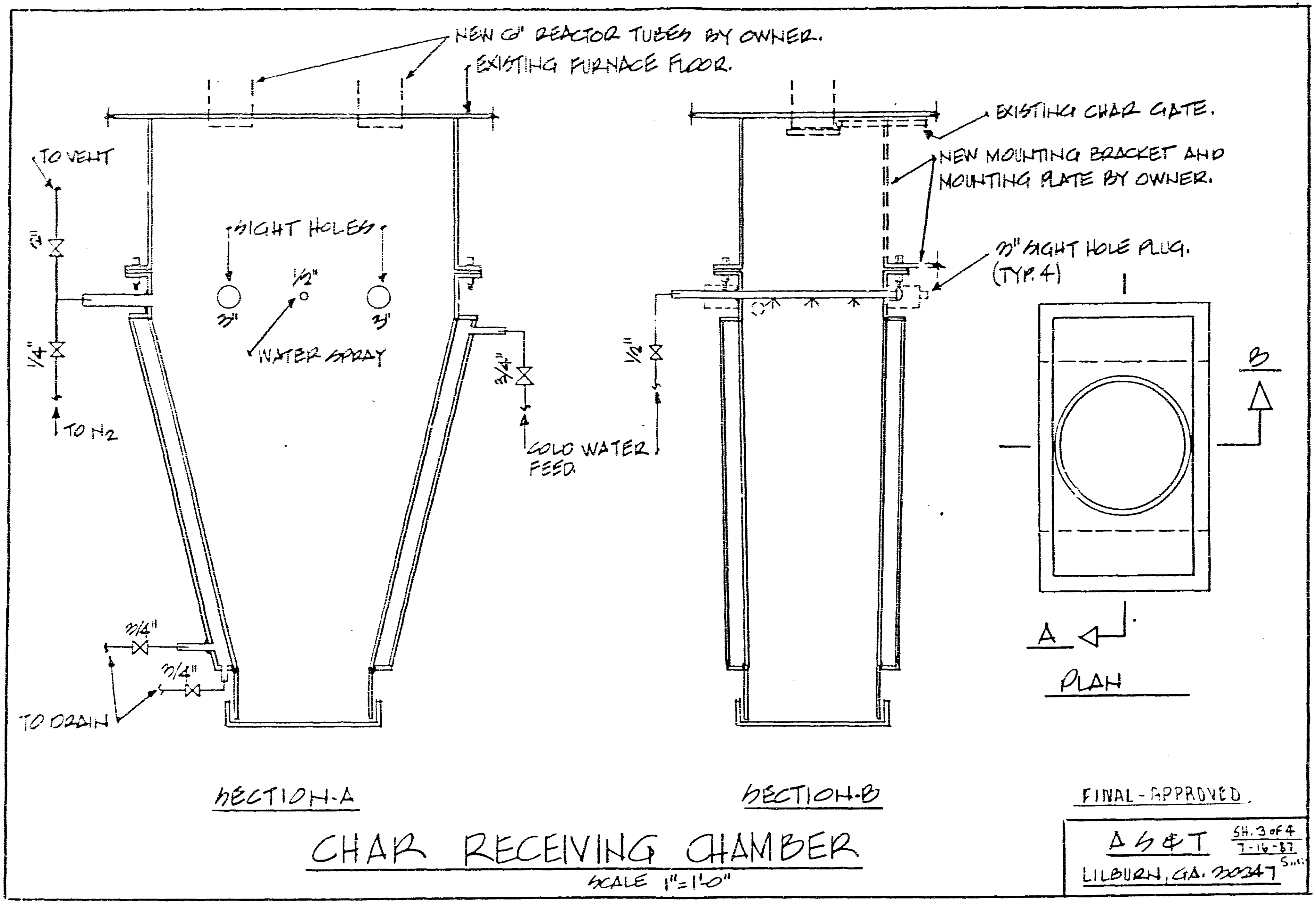

Figure 11. Char Receiving And Quenching Chamber For Modified MGU

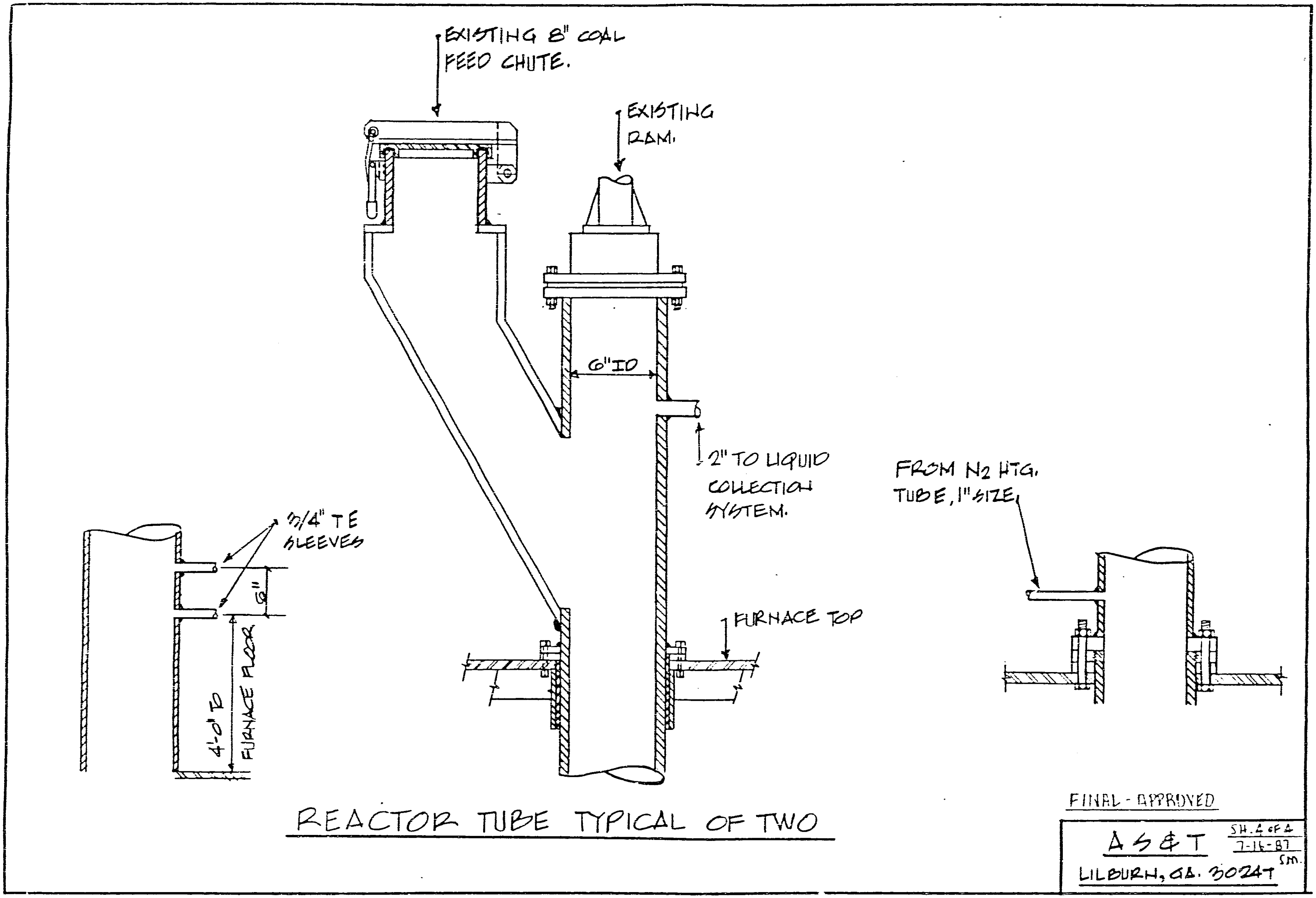

Figure 12. Reactor Tube Details For Modified MGU

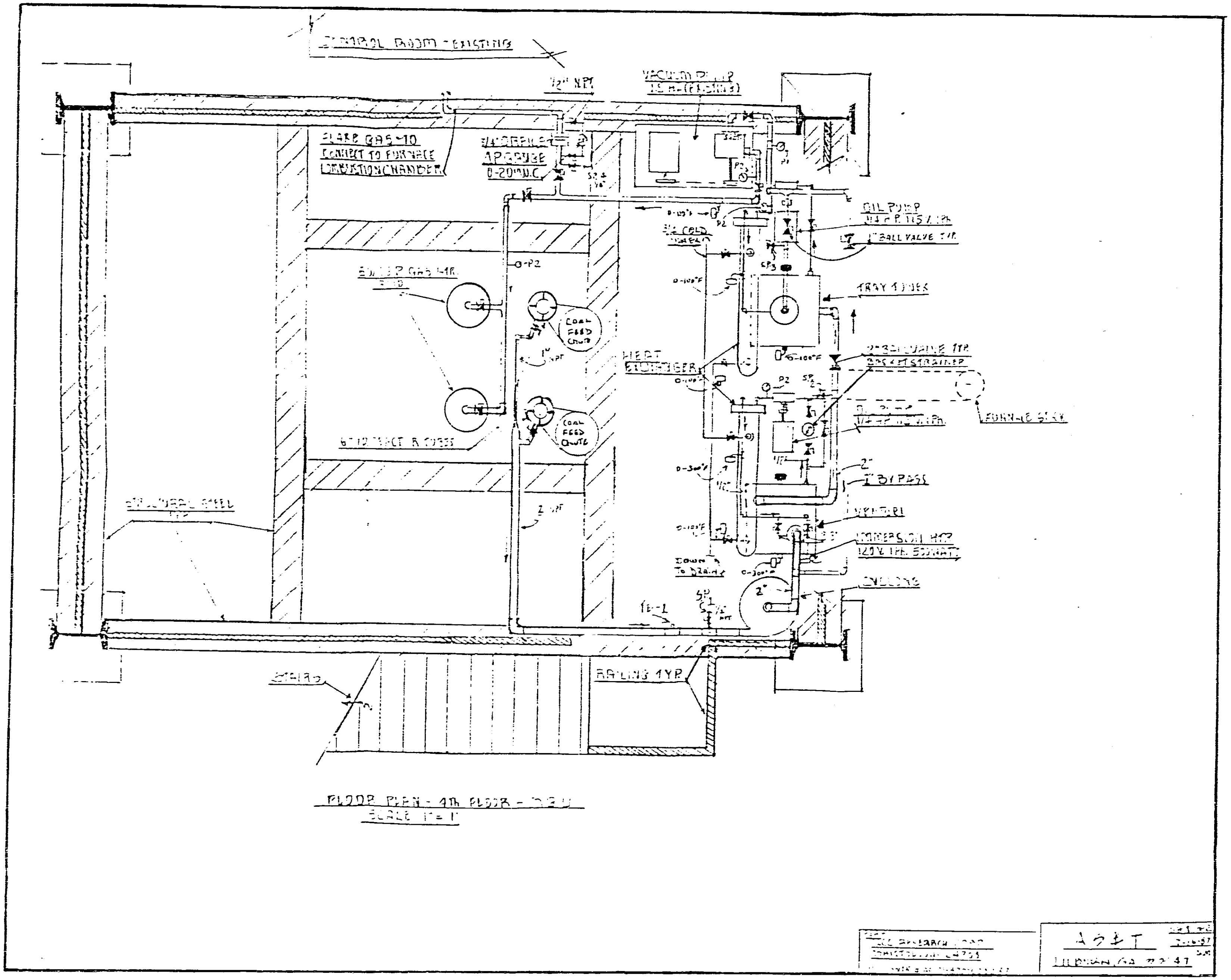

Figure 13. Floor Plan, Third Floor Of Modified MGU

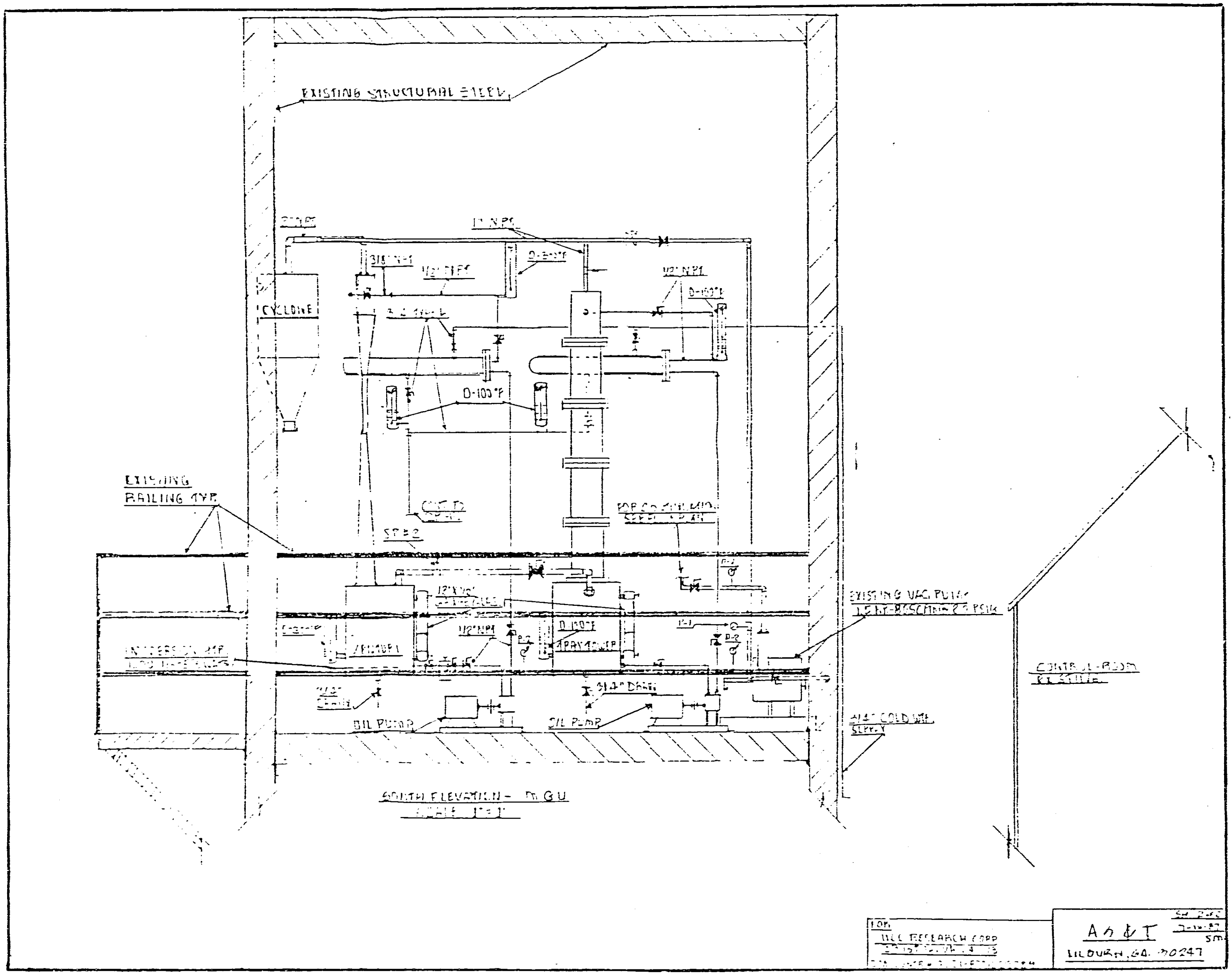

Figure 14. Equipment Layout For Third Floor Of Modified MGU

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