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DEVELOPMENT OF AN ADVANCED, CONTINUOUS
MILD GASIFICATION PROCESS
FOR THE PRODUCTION OF CO-PRODUCTS

PROJECT 61089 QUARTERLY REPORT
For the Period July-September 1989

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SUMMARY

During the past quarter, 10 mild gasification tests were conducted in the 8-inch-I.D. process research unit (PRU). Modifications to the PRU were made during this period to improve mixing and to overcome the caking tendency of the Illinois No. 6 coal. Coal or coal/coke mixture feed was introduced to the fluidized bed in 9 of the 10 tests, and freeboard feeding was used in one test. In all but two of the tests, the feed coal was blended with coke breeze; the ratio of coal to coke was 1:1 in all of these tests except one, in which a 2:1 coal to coke ratio was used.

Six of the tests resulted in satisfactory operation at steady conditions for 2.25 to 3.25 hours. Samples of char, gas, water, and organic condensables were collected over a one-hour period from each of these successful tests and analyzed. The resulting data show trends in co-product yields and characteristics that are valuable for scale-up design activities.

The effects of process temperature over the range of 1025° to 1390°F was studied during this quarter. The data show that the yield of oils and tars decreases with increasing temperature, whereas the gas yield increases. The observed oils/tars yields ranged from 12.4% to 19.8% by weight of dry coal, and gas yields ranged from 6.3% to 13.9% on the same basis. Compositional effects on the oils and tars observed with increased temperature are increased light oil content, decreased pitch content, decreased oxygen content, increased nitrogen and sulfur content, and increasing aromaticity. The content of low molecular weight phenols also increases at higher temperature. Gas composition data show increased H₂ and CO content along with decreased CO₂ and C₁-C₃ hydrocarbon content as the temperature increases. The H₂S content of the gas also decreased significantly with temperature. Further data analysis to validate these general observations is now in progress.

Char upgrading studies continued during the quarter. Briquettes made in a laboratory press, using either a pitch binder or Illinois No. 6 coal to provide an in-situ binder, were calcined and tested for diametral compression strength. Preliminary results show that the strength of briquettes made at briquetting pressures of 1,273 to 19,000 psi can approach the strength of commercial metallurgical cokes as reported in the literature. For smokeless fuel applications, briquettes made with mild gasification char, pitch binder, and limestone were combusted to determine sulfur retention. These tests

showed that at least 82% sulfur retention in the ash was attained, with about 90% of the retained sulfur in the form of sulfate. Char was also subjected to steam activation at a variety of conditions to determine the potential for use as a low-cost adsorbent for water treatment. The preliminary results are encouraging, with adsorbent power as measured by an Iodine Number Test approaching or exceeding values reported in the literature for commercially available active carbons.

System integration studies have progressed to include the evaluation of the PRU test data and incorporation of these data in a process simulation model for heat and material balances. Several flow schemes have been outlined for a projected 24-ton/day process development unit (PDU) to be located at Southern Illinois University's Illinois Coal Development Park in Carterville, Illinois.

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INTRODUCTION

The U.S. Department of Energy (DOE) is supporting the development of mild gasification technology to produce coal-derived fuels and chemical feedstocks. Mild gasification may be the most affordable route to increase coal utilization in the present economic climate. Mild gasification uses operating conditions of 1000° to 1500°F, near-atmospheric pressure, and inexpensive reactants to convert coal to a slate of co-products. In contrast, gasification and hydrogasification processes operate at temperatures of 1800°F or higher, and liquefaction processes use pressures of 1500 psig or higher and require a hydrogen supply.

Mild gasification could be considered as an advanced low-temperature carbonization of coal. Low-temperature carbonization was popular in the U.S. until natural gas became abundantly available, and it is still used on a commercial scale in some foreign countries; however, the old technology has been improved to produce value-added co-products through the application of technical and scientific knowledge about coal conversion that has been developed over the past twenty years. Improvements in reactor and process design are being applied to significantly enhance the yield and quality of co-products as well as the overall economics of the technology. Because of the mild operating conditions and process simplicity, mild gasification is anticipated to use available materials of construction and well-known engineering design and construction practices. As a result, the capital and operating costs are expected to be low. In this context, by successfully developing and marketing the co-products to derive the value-added benefits, it should be possible to commercialize the technology within the next 10 years.

With support from the U.S. DOE, a project team consisting of the Institute of Gas Technology, Peabody Holding Company, Inc., and Bechtel National, Inc., is developing a mild gasification process which uses a fluidized/entrained-bed reactor. This reactor is designed to process caking bituminous coals over a wide range of particle sizes without oxidative pretreatment, and also without the use of oxygen or air as reactants. Process heat, in the conceptual commercial reactor, would be provided by recycled high-temperature fuel gases or flue gases derived from burning a portion of the process-derived fuel gases. The addition of an in-bed sulfur-capture agent such as calcium oxide, to capture the hydrogen sulfide released during

coal conversion, is an option which is being explored. The co-product streams, consisting of char, fuel gas, water, and condensables, would be separated by conventional means such as cyclones, staged condensers, and recycle-oil scrubbers.

A process research unit (PRU) has been built at IGT, consisting of an 8-inch-ID, 8-foot-long fluidized-bed section and a 4-inch-ID, 13-foot-long entrained section, externally heated by electrical resistance heaters. The coal feed capacity is 100 lb/h, and the coal can be fed either to the fluidized bed or the freeboard region above the fluidized bed and below the entrained section. The stainless steel reactor vessel is designed for maximum temperature and pressure of 1500°F and 50 psig, respectively. Figures 1 and 2 show the block flow diagram and isometric layout of the PRU.

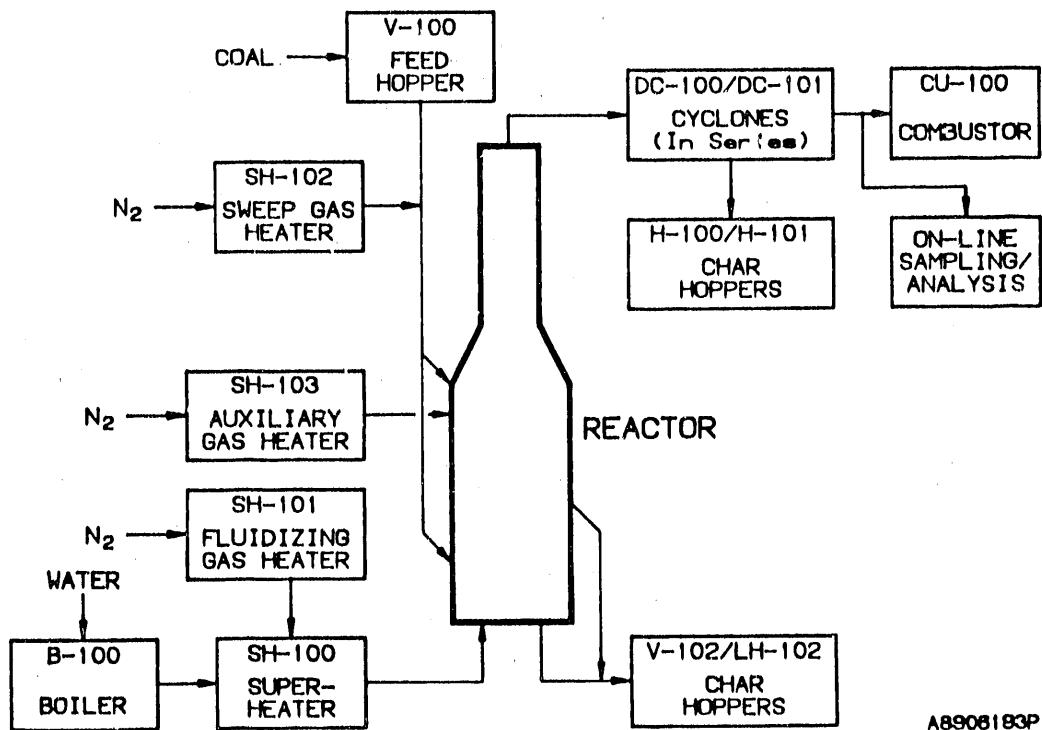


Figure 1. BLOCK FLOW DIAGRAM OF MILD GASIFICATION PRU

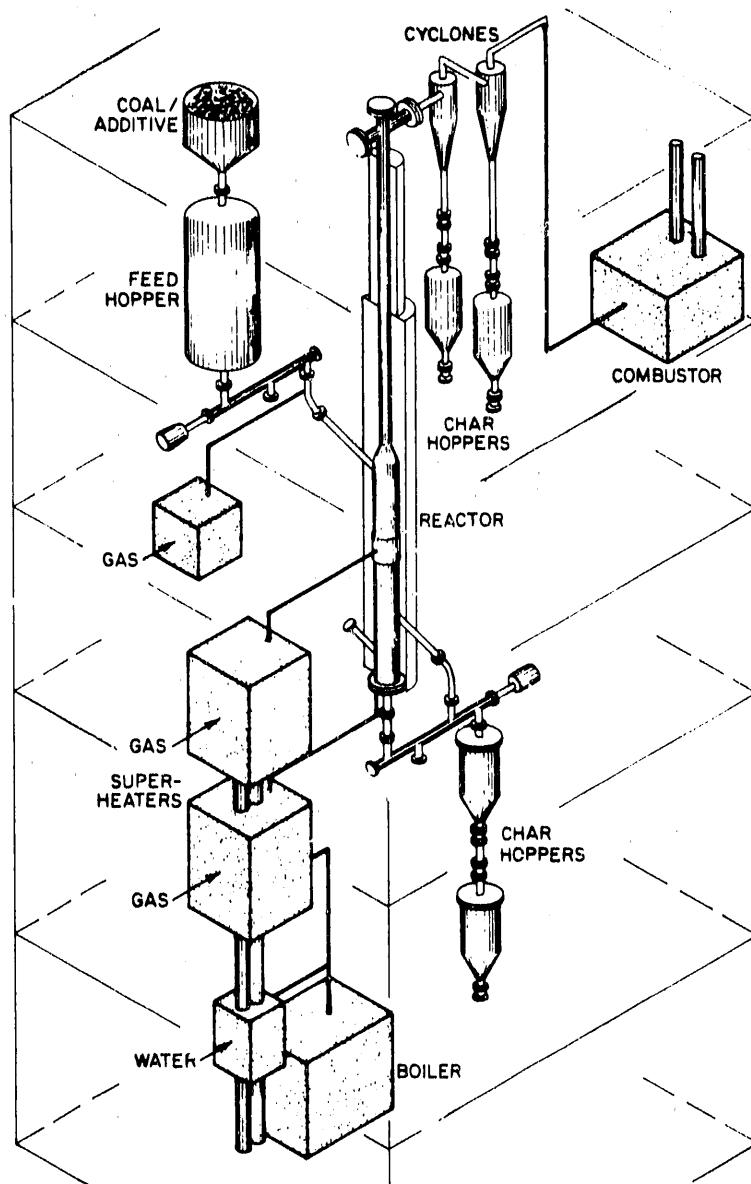


Figure 2. ISOMETRIC LAYOUT OF MILD GASIFICATION PRU

TECHNICAL DISCUSSION

Task 2. Bench-Scale Mild Gasification Study

During the quarterly reporting period, 10 mild gasification tests were conducted in the 8-inch-I.D. PRU. The coal used in all of these tests consisted of -12 mesh Illinois No. 6 coal preparation plant fines supplied by Peabody's Randolph Plant in Bladwin, Illinois. The process conditions used in these tests are summarized in Table 1, and the tests are described in detail in the following text. Detailed analytical data from tests up to and including Test MG-15 are given in Appendix A.

Based on the operating experience from previous tests, a modification, described in the previous quarterly report, was made to the PRU reactor. The fluidized-bed gas distributor was relocated to improve solids mixing and a center jet of fluidizing gas was added to the distributor design. With this modification, illustrated in Figure 3, the coal feeder port is located 12 inches below the char overflow port, and it is located 3 inches above the outer edge of the inverted-cone gas distributor, rather than 12 inches as in the previous configuration. As shown, the feed solids enter the fluidized bed at a point near the central jet, where the solids-circulation rate and turbulence may be greater than in the previous design, to promote better mixing. This allows the softening coal particles to disperse more rapidly in the char bed, reducing the frequency of collisions between sticky coal particles and improving the stability of the bed.

The feed location in all but one of these tests was the fluidized bed. Freeboard feeding was used in Test MG-17.

Summary of Mild Gasification Tests

Test MG-8 was conducted to test the operation of the reactor with the relocated gas distributor. The coal feed was screened to -40 mesh to increase the particle heat-up rate and consequently reduce the time during which the particles remain sticky and are subject to agglomeration. The feed used in this test was 100% coal. During the test, system-pressure upsets eventually led to bed instability and early shutdown. The cause of the upsets was found to be an excessive carry-over of char fines that led to plugging of an equalizing line between the reactor and feed hopper, together with the malfunction of a pressure-letdown valve-actuator mechanism. Ultimately, the

Table 1. SUMMARY OF MILD GASIFICATION TESTS DURING THE QUARTER

Test No.	Purpose of Test	Feed Material	Feed Rate, lb/h	Temp., °F	Pressure, psig	Feed Location	Fluid Bed Height, ft	Superficial Gas Velocity, ft/s	Solids Residence Time, min	Steady-State Period, h	Remarks
SG-8	Test relocated gas distributor and center jet; test -40 mesh coal for rapid particle heat-up	Coal ^a	29.4	1350	10	Fluid Bed	1.25	1.3	—	—	High fines carry-over plugged equalizing line and pressure-jetdown valve-actuator mechanism stuck causing pressure swings; cyclone inlet velocity too low for best efficient.../
MG-9	Test -40 mesh coal feed with coke breeze as starter fluidized bed	Coal	22.1	1320	10	Fluid Bed	1.25	1.5	—	—	Lost significant amount of start bed before coal fed; high fines carry-over to sampling system due to low cyclone velocity.
MG-10	Test -12 mesh feed to fluidized bed, higher temp; test increased jet diameter; maintain F/W parameter ^b at about 2.0 h ⁻¹	1:1 coal:coke	23.9	1320	10	Fluid Bed	1.25	3.8	—	—	Reactor heating element short with reactor pressure tap, loss of temperature control; feed hopper load cells gave false reading because of uneven thermal expansion
MG-11	Repeat MG-10 at ~1400°F	1:1 coal:coke	20.5	1390	10	Fluid Bed	1.25	4.0	32	3.25	Successful test operation
MG-12	Repeat at MG-11 at ~1200°F	1:1 coal:coke	22.7	1250	10	Fluid Bed	1.25	3.5	29	3.25	Successful test operation
MG-13	Test higher coal:coke ratio ^c	2:1 coal:coke	14.0	1260	10	Fluid Bed	1.25	3.5	—	—	Cake buildup on reactor wall covered pressure taps
MG-14	Repeat MG-12 at ~1100°F with relocated pressure tap	1:1 coal:coke	23.2	1100	10	Fluid Bed	1.25	3.6	28	2.25	Successful test operation
MG-15	Repeat MG-12 at 1000°F	1:1 coal:coke	19.5	1025	10	Fluid Bed	1.25	3.4	33	3.00	Successful test operation
MG-16	Test deeper fluidized bed with increased feed rate, maintain F/W parameter at ~2	1:1 coal:coke ^c	32.4	1180	10	Fluid Bed	2.00	3.5	20	3.00	Successful test operation;
MG-17	Test -40 mesh feed to entrained bed only, no fluidized bed	coal:coke	1:1	20.0	1200	10	Freeboard	—	13.8	0.02	Successful operation; part of feed collected in lower reactor section due to insufficient entrainment

^aTest coal is a -12 mesh Illinois No. 6 coal obtained from Peabody Coal Company's Randolph Preparation Plant in Baldwin, Illinois.
^bF/W is coal feed rate is lb/h divided by total weight of bed material in lb.
^cFeed coal screened to remove -40 mesh coal fines.

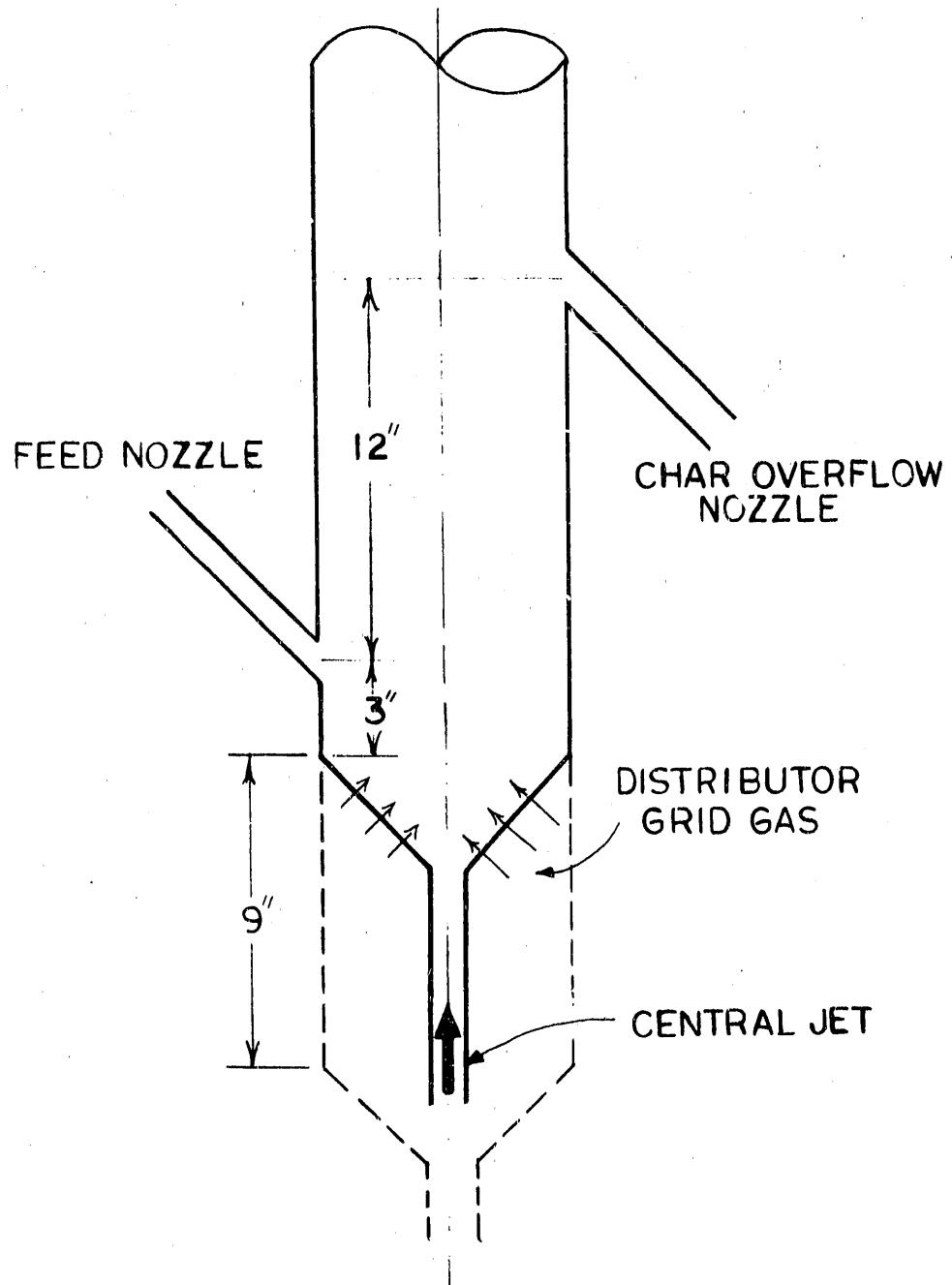


Figure 3. MODIFICATION TO PRU GAS DISTRIBUTOR

excessive fines carry-over was attributed to the fact that the lower superficial gas velocity required for fluidization of the -40 mesh coal caused the cyclones to operate at low efficiency.

Test MG-9 was also operated with -40 mesh coal, but with a start-up bed of -40 mesh coke breeze. Plugging of the sampling system with carry-over fines and the loss of most of the starter bed by entrainment caused premature shutdown.

In Test MG-10, the feed size was returned to -12 mesh, and a 1:1 coal to coke mixture was used. Also, the diameter of the central fluidizing jet was increased from 0.5 inch to 1 inch. The test was aborted when a reactor heating element grounded to a reactor pressure-tap fitting, causing a loss of temperature control in the fluidized bed. Also, the feed hopper load cells gave a false reading of feed weight, indicating an unexpectedly high feed rate. It was later determined that both problems were a result of thermal expansion stresses between the feed system and reactor. This was successfully corrected by welding additional steel supports to the feed screw housing and adding a guide to the vertical feed line to prevent lateral movement.

Following repair of the heater and the support modifications, Test MG-11 was operated at conditions similar to Test MG-10, at a temperature of 1390°F. Based on the operating experience in the U-GAS pilot plant, the importance of a parameter relating feed rate to total bed weight was recognized. This parameter is defined as --

$$F/W, h^{-1} = \frac{\text{feed rate, lb/h}}{\text{total bed weight, lb}}$$

An F/W value of 2.0 h^{-1} or less was shown to prevent caking in successful U-GAS tests, and was maintained in Test MG-11. This test was operationally successful, and a steady-state period of 3.25 hours was attained with satisfactory bed stability. Samples of gas, oils and tars, and solids collected over 1 hour of steady state were analyzed, and material balances were calculated. The Test MG-11 material and elemental balances are shown for the coal/coke mixture in Table 2 and for the coal feed on a coke-free basis in Table 3.

Three adjustments were made to the measured data in preparing these balances. First, the recovered fluidized-bed char weight was adjusted to

Table 2. MATERIAL BALANCE FOR TEST MG-11 (Coal/Diluent Mixture)

Test Temperature: 1390°F
Basis: 100 lbs dry coal

	<u>C</u>	<u>H</u>	<u>O</u>	<u>N</u>	<u>S</u>	<u>Ash</u>	<u>Total</u>
INPUT							
Coal	64.98	4.38	9.59	1.51	3.95	15.59	100.00
Moisture	NA*	0.38	3.03	NA	NA	NA	3.41
Coke	86.81	0.40	1.17	1.24	0.75	11.88	102.25
Moisture	NA	0.13	1.03	NA	NA	NA	1.16
Steam	NA	5.74	45.58	NA	NA	NA	51.33
Total	151.79	11.04	60.40	2.75	4.70	27.47	258.15
OUTPUT							
Fluid Bed Char	81.67	0.32	0.23	1.33	1.16	16.03	100.74
Cyclone Char	49.13	0.44	0.65	0.88	0.97	9.82	61.89
Entrained Filter							
Char	1.67	0.03	0.00	0.04	0.22	1.56	3.52
Moisture	NA	0.07	0.54	NA	NA	NA	0.61
Gases (dry)	8.20	2.25	6.12	0.00	0.82	NA	17.40
Oils/Tars	10.30	0.78	0.74	0.14	0.36	0.06	12.38
Aqueous Condensate	ND**	6.96	55.28	ND	ND	NA	62.24
Total	150.97	10.85	63.56	2.39	3.53	27.47	258.78
Out/In	0.99	0.98	1.05	0.87	0.75	1.00	1.00

* Not applicable.

** No data available.

Table 3. MATERIAL BALANCE FOR TEST MG-11 (Diluent-Free Basis)

Test Temperature: 1390°F
Basis: 100 lbs dry coal, diluent-free

	<u>C</u>	<u>H</u>	<u>O</u>	<u>N</u>	<u>S</u>	<u>Ash</u>	<u>Total</u>
INPUT							
Coal	64.98	4.38	9.59	1.51	3.95	15.59	100.00
Steam + Moisture	NA*	5.97	47.41	NA	NA	NA	53.39
Total	64.98	10.35	57.00	1.51	3.95	15.59	153.39
OUTPUT							
Char	46.86	0.37	0.00	1.01	1.60	15.53	65.07
Gases (dry)	7.00	2.17	3.89	0.00	0.82	NA	13.88
Oils/Tars	10.30	0.78	0.74	0.14	0.36	0.06	12.38
Aqueous Condensate							
+ Moisture	ND**	7.03	55.83	ND	ND	NA	62.86
Total	64.16	10.35	60.16	1.15	2.78	15.59	154.19
Out/In	0.99	1.00	1.06	0.76	0.70	1.00	1.01

* Not applicable.

** No data available.

force an ash balance, assuming that the accumulation of some char on the reactor walls introduces a non-reproducible error into the steady-state char collection measurement. Second, comparison of the collected aqueous condensate with the steam input reported by instruments showed a large discrepancy, which was consistent over several tests. A conclusion was reached that steam input rate was subject to a systematic error due to incorrect flowmeter orifice size and a leak at the boiler; consequently, the steam input was adjusted to give a 100% hydrogen balance on a diluent-free basis. Third, an adjustment was made for gas produced by the diluent coke in the fluidized bed, as determined by the on-line gas chromatograph. This gas, consisting primarily of H_2 , CO_2 , and a small amount of CH_4 , was subtracted from the total gas made in determining the diluent-free material balance reported in Table 3, and corollary adjustments were made to the char carbon and steam in order to determine the portion of recovered char originating from coal. As shown in Tables 2 and 3, the aqueous condensate was not analyzed for dissolved carbon, nitrogen, and sulfur. It is probable that significant portions of nitrogen and sulfur were present as dissolved ammonia and hydrogen sulfide in the aqueous condensate. These components, which would improve nitrogen and sulfur balances, are accounted for in later tests.

These three adjustments to the raw data were also used for the calculation of material balances for Tests MG-12, MG-14, and MG-15. As additional test results are acquired, the procedure described above for adjusting the data will be critically reviewed and modified if necessary.

The material balance data from Test MG-11 shows the following co-product yields on a diluent-free dry coal basis:

Char	65.1	wt % dry coal
Oils and Tars	12.4	wt % dry coal
Gas	13.9	wt % dry coal
Water	<u>9.5</u>	wt % dry coal
Total	100.9	wt % dry coal

Test MG-12 was conducted at conditions similar to MG-11, but at a lower temperature of 1250°F. This test also operated steadily for 3.25 hours, and the resulting material balance data are shown in Tables 4 and 5. The co-product yields from Test MG-12 are as follows:

Table 4. MATERIAL BALANCE FOR TEST MG-12 (Coal/Diluent Mixture)

Test Temperature: 1250°F
Basis: 100 lbs dry coal

	<u>C</u>	<u>H</u>	<u>O</u>	<u>N</u>	<u>S</u>	<u>Ash</u>	<u>Total</u>
INPUT							
Coal	65.43	4.43	10.49	1.28	3.81	14.56	100.00
Moisture	NA*	0.38	3.05	NA	NA	NA	3.43
Coke	86.83	0.40	1.18	1.24	0.75	11.87	102.27
Moisture	NA	0.13	1.03	NA	NA	NA	1.16
Steam	NA	5.74	45.48	NA	NA	NA	51.22
Total	152.26	11.08	61.23	2.52	4.56	26.43	258.08
OUTPUT							
Fluid Bed Char	78.33	0.38	0.02	1.25	1.23	15.97	97.18
Cyclone Char	43.78	0.17	1.32	0.79	0.89	8.05	1.48
Entrained Filter Char	2.70	0.00	0.00	0.00	0.00	2.34	5.04
Moisture	NA	0.17	1.32	NA	NA	NA	1.48
Gases (dry)	5.97	1.58	3.68	0.00	0.73	NA	11.96
Oils/Tars	15.06	1.24	2.73	0.24	0.39	0.07	19.73
Aqueous Condensate	ND**	7.13	56.58	ND	ND	NA	63.72
Total	145.84	10.98	65.06	2.28	3.24	26.43	253.83
Out/In	0.96	0.99	1.06	0.91	0.71	1.00	0.98

* Not applicable.

** No data available.

Table 5. MATERIAL BALANCE FOR TEST MG-12 (Diluent-Free Basis)

Test Temperature: 1250°F
Basis: 100 lbs dry coal, diluent-free

	<u>C</u>	<u>H</u>	<u>O</u>	<u>N</u>	<u>S</u>	<u>Ash</u>	<u>Total</u>
INPUT							
Coal	65.43	4.43	10.49	1.28	3.81	14.56	100.00
Steam + Moisture	NA*	5.99	47.56	NA	NA	NA	53.55
Total	65.43	10.42	58.05	1.28	3.81	14.56	153.55
OUTPUT							
Char	39.29	0.45	0.00	0.80	1.38	14.49	56.41
Gases (dry)	4.65	1.43	1.68	0.00	0.73	NA	8.49
Oils/Tars	15.07	1.25	2.73	0.24	0.39	0.07	19.75
Aqueous Condensate + Moisture	ND**	7.29	57.89	ND	ND	NA	65.18
Total	59.01	10.42	61.87	1.04	2.50	14.56	149.83
Out/In	0.90	1.00	1.07	0.81	0.65	1.00	0.98

* Not applicable.

** No data available.

Char	56.0 wt % dry coal
Oils and Tars	19.7 wt % dry coal
Gas	8.5 wt % dry coal
Water	<u>11.0</u> wt % dry coal
Total	95.8 wt % dry coal

Test MG-13 was conducted with a feed mixture containing a 2:1 ratio of coal to coke breeze as a means to increase the coal feed rate. The test was interrupted prior to steady state because of a buildup of caked coal on the reactor walls covering the pressure taps and blocking the feed and overflow discharge ports. The pressure taps were relocated away from the feed inlet for subsequent tests.

Test MG-14 was conducted with the 1:1 coal to coke mixture at 1100°F. Test operation was successful, with a 2.25-hour steady-state period during which samples were collected. The material balances for Test MG-14 are shown in Tables 6 and 7, and the measured co-product yields were as follows:

Char	67.0 wt % dry coal
Oils and Tars	12.5 wt % dry coal
Gas	6.3 wt % dry coal
Water	<u>14.7</u> wt % dry coal
Total	100.5 wt % dry coal

Test MG-15 was a repeat of the same test operation at a lower temperature of 1025°F. The test ran satisfactorily, and the available analytical data are given in Appendix A. Complete material balances for Test MG-15 are given in Tables 8 and 9 for the coal/coke mixture and the coke-free coal, respectively. The measured co-product yields from Test MG-15, on a moisture- and ash-free basis, are as follows:

Char	65.9 wt % dry coal
Oils and Tars	19.3 wt % dry coal
Gas	8 wt % dry coal
Water	<u>7.8</u> wt % dry coal
Total	101.8 wt % dry coal

Test MG-16 was operated with a higher feed rate by using a deeper fluidized bed of 24 inches and maintaining the F/W parameter at about 2.0. The bed height was controlled by reducing the speed of the char discharge screw, allowing the bed to build up higher than the discharge port. Steady-state operation at a test temperature of 1180°F was maintained for 3.0 hours with a one-hour sample collection period. It should be noted that the coal used in this test was screened to remove -40 mesh fines. This was done to

Table 6. MATERIAL BALANCE FOR TEST MG-14 (Coal/Diluent Mixture)

Test Temperature: 1100°F
Basis: 100 lbs dry coal

	<u>C</u>	<u>H</u>	<u>O</u>	<u>N</u>	<u>S</u>	<u>Ash</u>	<u>Total</u>
INPUT							
Coal	63.72	4.33	10.97	1.13	3.80	16.05	100.00
Moisture	NA*	0.42	3.32	NA	NA	NA	3.74
Diluent	87.03	0.41	1.20	1.24	0.76	11.93	102.57
Moisture	NA	0.13	1.03	NA	NA	NA	1.16
Steam	NA	6.15	48.79	NA	NA	NA	54.94
Total	150.75	11.44	65.31	2.37	4.56	27.98	262.41
OUTPUT							
Fluid Bed Char	96.53	0.82	2.80	1.49	1.81	21.42	124.87
Cyclone Char	34.26	0.46	1.22	0.57	0.75	6.16	43.42
Entrained Filter							
Char	0.36	0.00	0.00	0.00	0.00	0.36	0.72
Moisture	NA	0.18	1.43	NA	NA	NA	1.61
Gases (dry)	3.95	1.01	2.11	0.00	0.72	NA	7.79
Oils/Tars	9.83	0.85	1.49	0.12	0.22	0.03	12.54
Aqueous Condensate	ND**	8.06	63.90	ND	ND	NA	71.96
Total	144.93	11.38	72.95	2.18	3.50	27.97	262.91
Out/In	0.96	1.00	1.12	0.92	0.77	1.00	1.00

* Not applicable.

** No data available.

Table 7. MATERIAL BALANCE FOR TEST MG-14 (Diluent-Free Basis)

Test Temperature: 1100°F
Basis: 100 lbs dry coal, diluent-free

	<u>C</u>	<u>H</u>	<u>O</u>	<u>N</u>	<u>S</u>	<u>Ash</u>	<u>Total</u>
INPUT							
Coal	63.72	4.33	10.97	1.13	3.80	16.05	100.00
Steam + Moisture	NA*	6.58	52.26	NA	NA	NA	58.84
Total	63.72	10.91	63.23	1.13	3.80	16.05	158.84
OUTPUT							
Char	44.66	0.87	2.81	0.82	1.80	16.02	66.98
Gases (dry)	3.40	0.95	1.23	0.00	0.72	NA	6.30
Oils/Tars	9.83	0.85	1.49	0.12	0.22	0.03	12.54
Aqueous Condensate							
+ Moisture	ND**	8.24	65.33	ND	ND	NA	73.57
Total	57.89	10.91	70.86	0.94	2.74	16.05	159.39
Out/In	0.91	1.00	1.12	0.83	0.72	1.00	1.00

* Not applicable.

** No data available.

Table 8. MATERIAL BALANCE FOR TEST MG-15 (Coal/Diluent Mixture)

Test Temperature: 1025°F Basis: 100 lbs dry coal							
	C	H	O	N	S	Ash	Total
INPUT							
Coal	67.99	4.40	9.41	1.46	3.28	13.46	100.00
Moisture	NA*	0.33	2.60	NA	NA	NA	2.93
Diluent	86.48	0.39	1.14	1.23	0.74	11.79	101.77
Moisture	NA	0.13	1.02	NA	NA	NA	1.15
Steam	NA	10.06	79.89	NA	NA	NA	89.95
Total	154.47	15.31	94.06	2.69	4.02	25.25	295.80
OUTPUT							
Fluid Bed Char	88.72	0.71	1.83	1.47	1.45	16.34	110.52
Cyclone Char	43.48	0.66	1.33	0.83	0.91	7.29	54.50
Entrained Filter							
Char	0.72	0.00	0.00	0.00	0.00	1.51	2.23
Moisture	NA	0.11	0.90	NA	NA	NA	1.01
Gases (dry)	4.92	1.28	2.92	0.00	0.85	NA	9.97
Oils/Tars	14.31	1.33	3.09	0.15	0.30	0.12	19.30
Aqueous Condensate	0.27	11.16	88.60	0.04	0.01	NA	100.08
Total	152.42	15.25	98.67	2.49	3.52	25.26	297.61
Out/In	0.99	1.00	1.05	0.92	0.88	1.00	1.01

* Not applicable.

Table 9. MATERIAL BALANCE FOR TEST MG-15 (Diluent-Free Basis)

Test Temperature: 1025°F Basis: 100 lbs dry coal, diluent-free							
	C	H	O	N	S	Ash	Total
INPUT							
Coal	67.99	4.40	9.41	1.46	3.28	13.46	100.00
Steam + Moisture	NA*	10.44	82.85	NA	NA	NA	93.29
Total	67.99	14.84	92.26	1.46	3.28	13.46	193.29
OUTPUT							
Char	46.88	0.97	2.01	1.07	1.62	13.34	65.89
Gases (dry)	4.48	1.22	2.27	0.00	0.85	NA	8.82
Oils/Tars	14.31	1.33	3.09	0.15	0.30	0.12	19.30
Aqueous Condensate + Moisture	0.27	11.28	89.48	0.04	0.01	NA	101.08
Total	65.94	14.80	96.85	1.26	2.78	13.46	195.09
Out/In	0.97	1.00	1.05	0.86	0.85	1.00	1.01

* Not applicable.

restrict the pyrolysis to the fluidized bed by minimizing the entrainment of coal. Analytical data are not yet available for this test.

Test MG-17 was conducted with only -40 mesh feed to investigate the performance of the entrained reaction zone separately. The -40 mesh fraction represents about one-third of the -12 mesh size coal stream from the Peabody coal preparation plant. The 1:1 mixture of -40 mesh coal and coke was fed to the freeboard at 20 lb/h, and the test temperature was 1200°F. The test achieved steady operation for 3 hours and was voluntarily terminated. Following shutdown, it was found that about 36 lbs, approximately 60% of the total feed, had collected in the fluidized-bed region of the PRU. Analytical data are not yet available for this test.

Discussion of PRU Test Results

With the available data on Tests MG-11, MG-12, MG-14, and MG-15, along with previously reported data from Test SD-6, an evaluation was made of the effect of temperature on co-product yields, gas composition, and selected properties of oils and tars.

Figure 4 shows the effect of temperature on the yields of gas and oils/tars observed from Tests SD-6, MG-11, MG-12, MG-14, and MG-15. As shown, the gas yield increases with temperature, whereas the maximum condensables yield appears to lie somewhere in the 1000° to 1300°F range, declining as temperature increases.

Figure 5 shows the influence of process temperature on the boiling-range distribution of the oils and tars. It appears from these data that the principal change in the volatility of the condensables with increasing mild gasification temperature consists of an increase in light oils accompanied by a decrease in the fraction of material with a boiling point above 750°F, which is defined as pitch.

Figures 6 and 7 show the manner in which the heteroatom content of the condensables boiling at 360°F or higher temperatures changes with process temperature. Nitrogen and sulfur levels increase slightly, whereas oxygen content decreases sharply with increasing mild gasification temperature. The decrease in oxygen content signals the loss of oxygen functionalities, such as phenolic -OH, and may partially explain the decrease in pitch content at higher temperatures whereby the removal of oxygen functional groups reduces

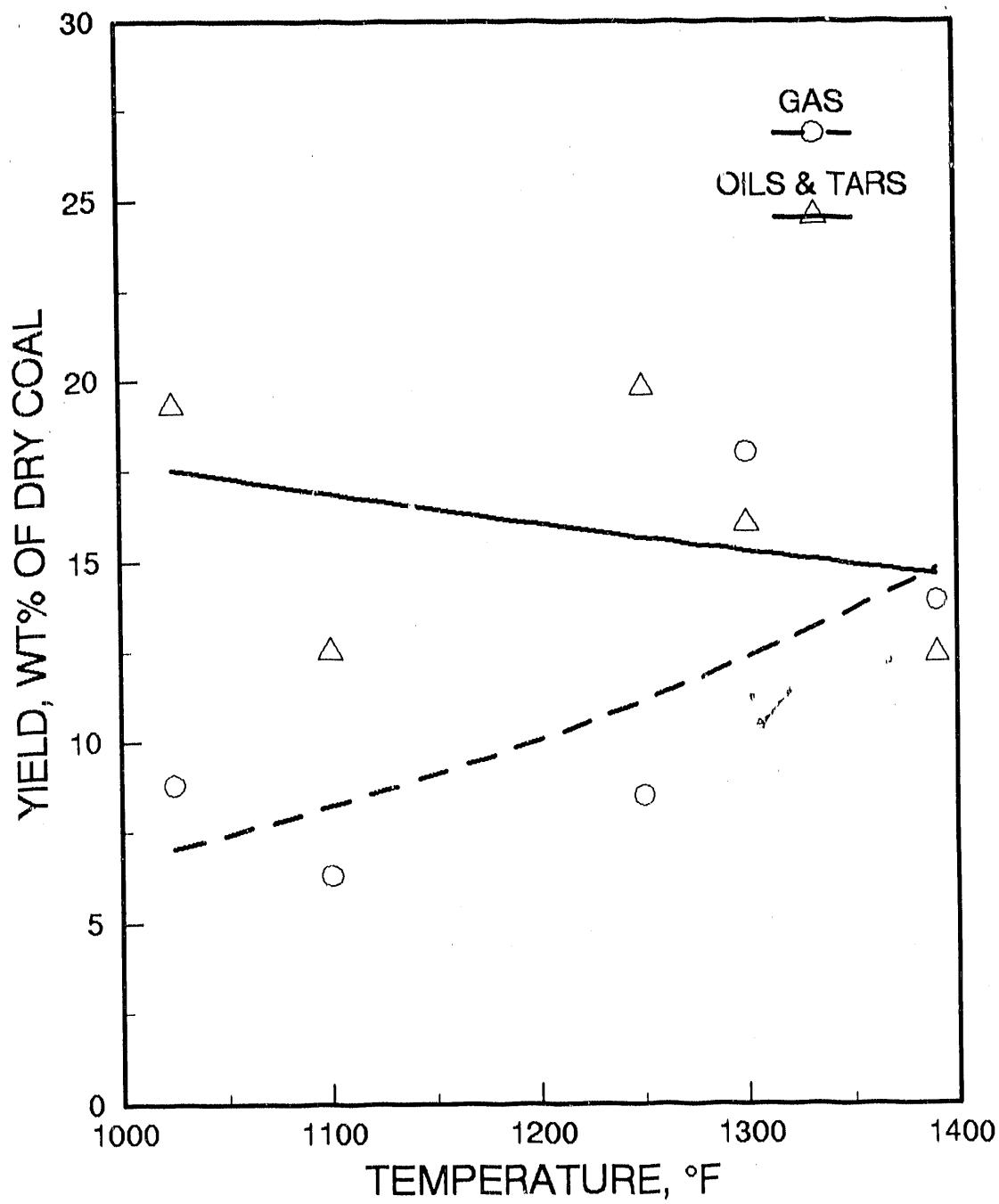


Figure 4. CO-PRODUCT YIELDS AS A FUNCTION OF PROCESS TEMPERATURE

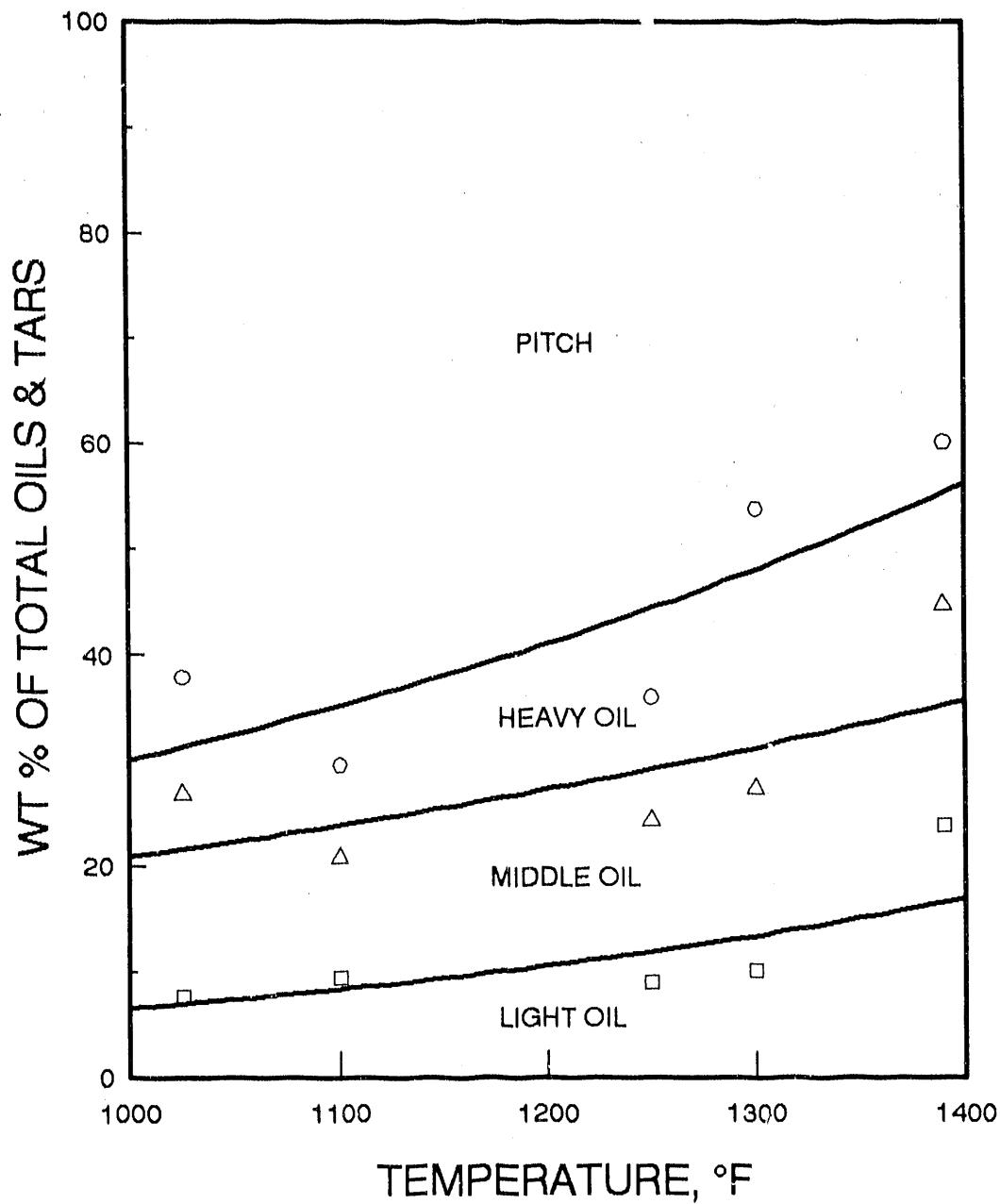


Figure 5. CONDENSABLES BOILING-RANGE FRACTIONS AS A FUNCTION OF PROCESS TEMPERATURE

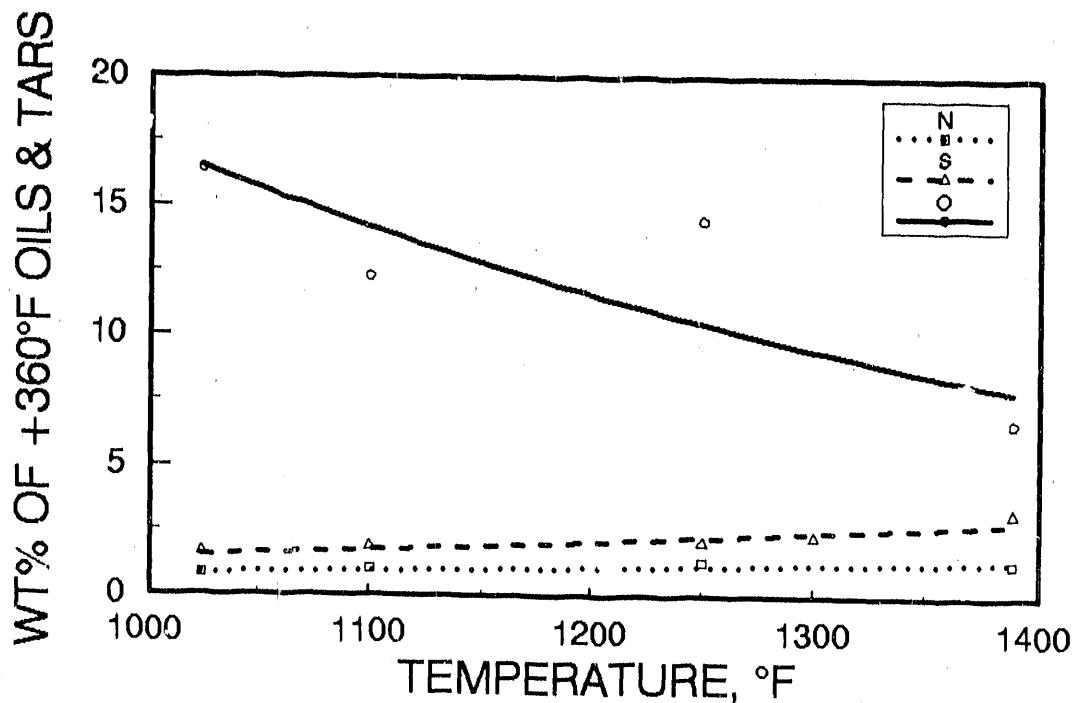


Figure 6. HETEROATOM DISTRIBUTION IN CONDENSABLES AS A FUNCTION OF PROCESS TEMPERATURE

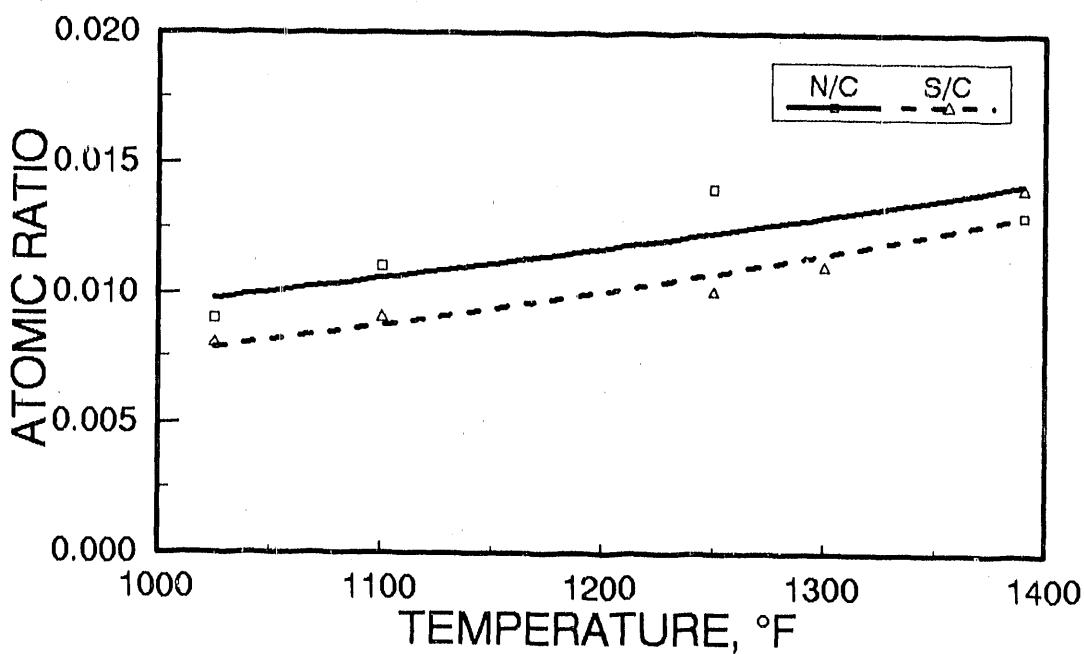


Figure 7. NITROGEN/CARBON AND SULFUR/CARBON RATIOS IN CONDENSABLES AS A FUNCTION OF PROCESS TEMPERATURE

the boiling point and shifts some pitch components into lower-boiling fractions. The slight increases in N and S content with process temperature may indicate increasing incorporation of these heteroatoms into polycondensed aromatic rings.

The H/C ratio of the condensables also changes with process temperature, as shown in Figure 8. The illustrated decrease in H/C ratio is consistent with an increase in tar aromaticity with temperature.

In addition to product gas yield, gas composition is also affected by process temperature. Figure 9 shows that the dry, nitrogen-free concentrations of H_2 and CO increase with temperature, whereas CO_2 and hydrocarbon gases decrease. These trends are consistent with well-known devolatilization and gas-phase equilibrium data. The H_2S content of the gas also decreases with temperature, and it appears that, even when the increased gas yield at higher temperature is taken into account, the amount of sulfur released as H_2S decreases with temperature.

Total Quench System

The specific system components have been specified for the design of the total quench system, and multiple fabricator and vendor quotes are being obtained. Figure 10 shows the design, which consists of a water-spray tower to condense and coalesce oils and tars prior to combustion of the product gas. The sampling system will still be used during PRU tests to verify light oils and gas compositions, and to measure the aqueous condensate upstream of the quench vessel.

Task 3. Bench-Scale Char Upgrading Study

Single briquettes were made in a hot-mold press with char materials from various PRU tests and with char from a separate laboratory coal carbonizer unit. The test briquettes were cylinders of 1.0-inch diameter by approximately 0.5-inch thickness. A 175°F-softening-point pitch obtained from Reilly Industries was used as a binder for some of the briquettes. The binder content used was 12% by weight of the char. Briquettes were also made without the pitch binder, using Illinois No. 6 coal in a 1:1 weight ratio with the chars to provide an in-situ binder.

Briquettes were prepared under various compression pressures and, depending upon the targeted application, were subjected to various post-

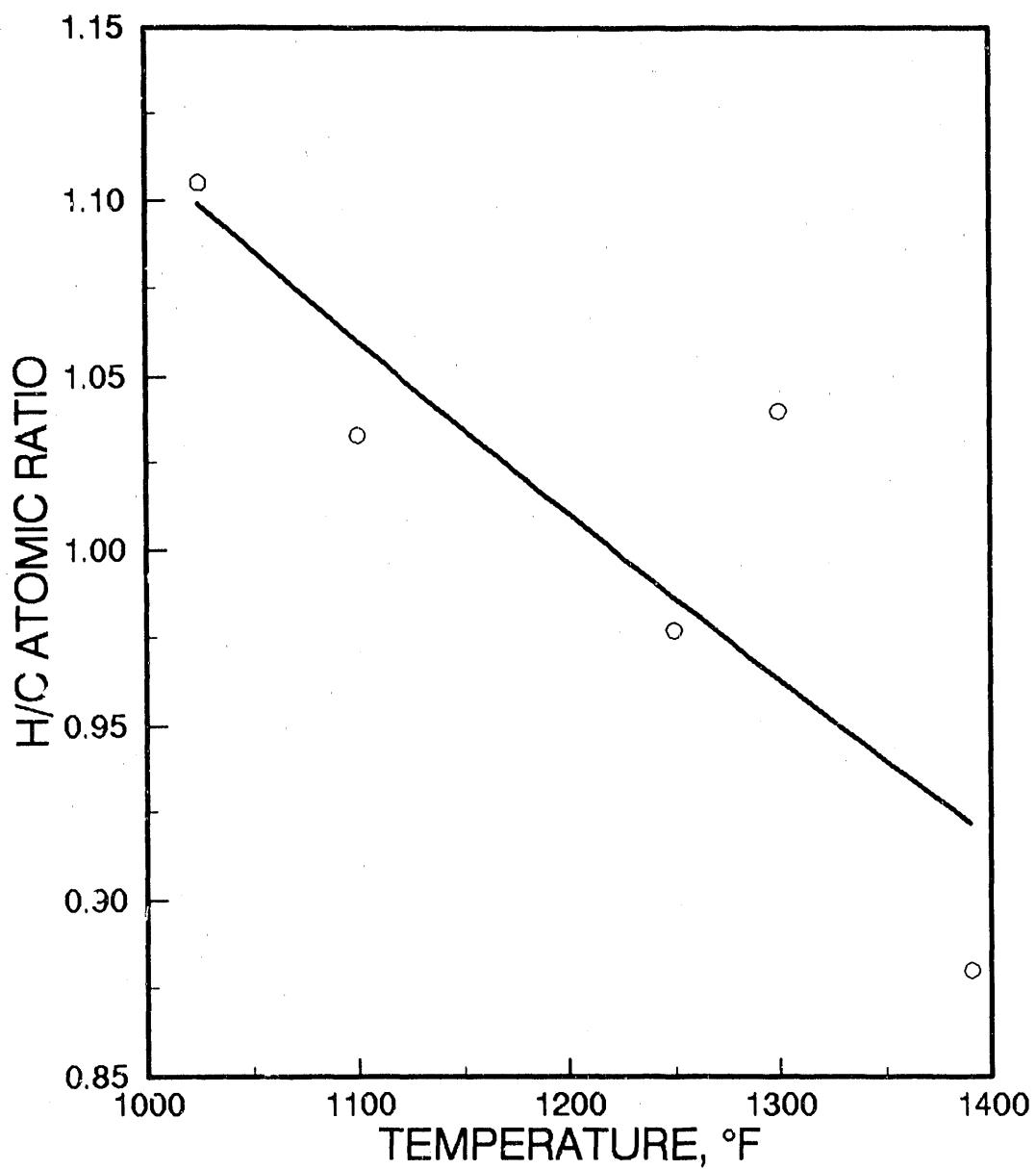


Figure 8. HYDROGEN/CARBON ATOMIC RATIO IN CONDENSABLES AS A FUNCTION OF PROCESS TEMPERATURE

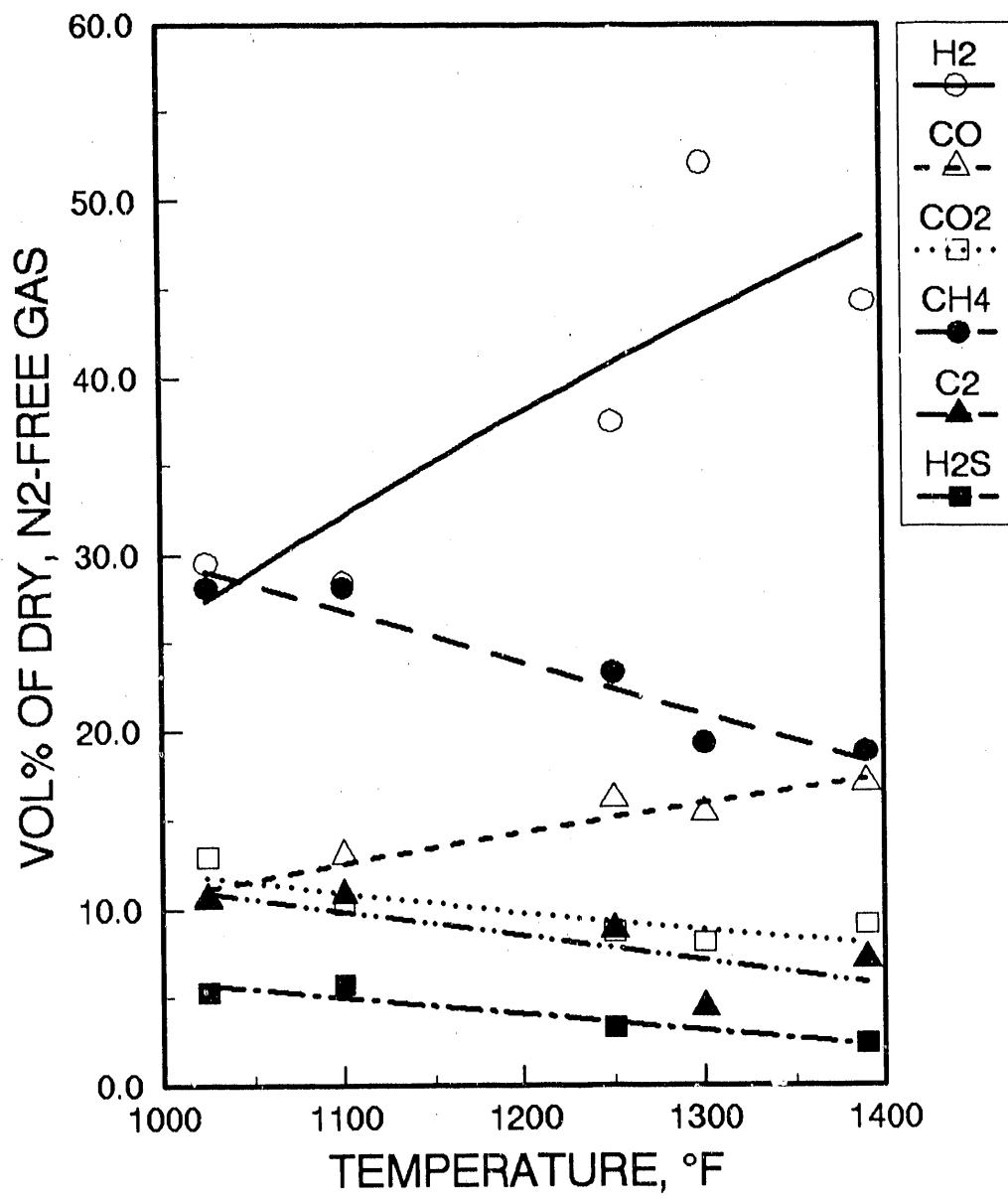


Figure 9. EFFECTS OF MILD GASIFICATION TEMPERATURE ON PRODUCT GAS COMPOSITION

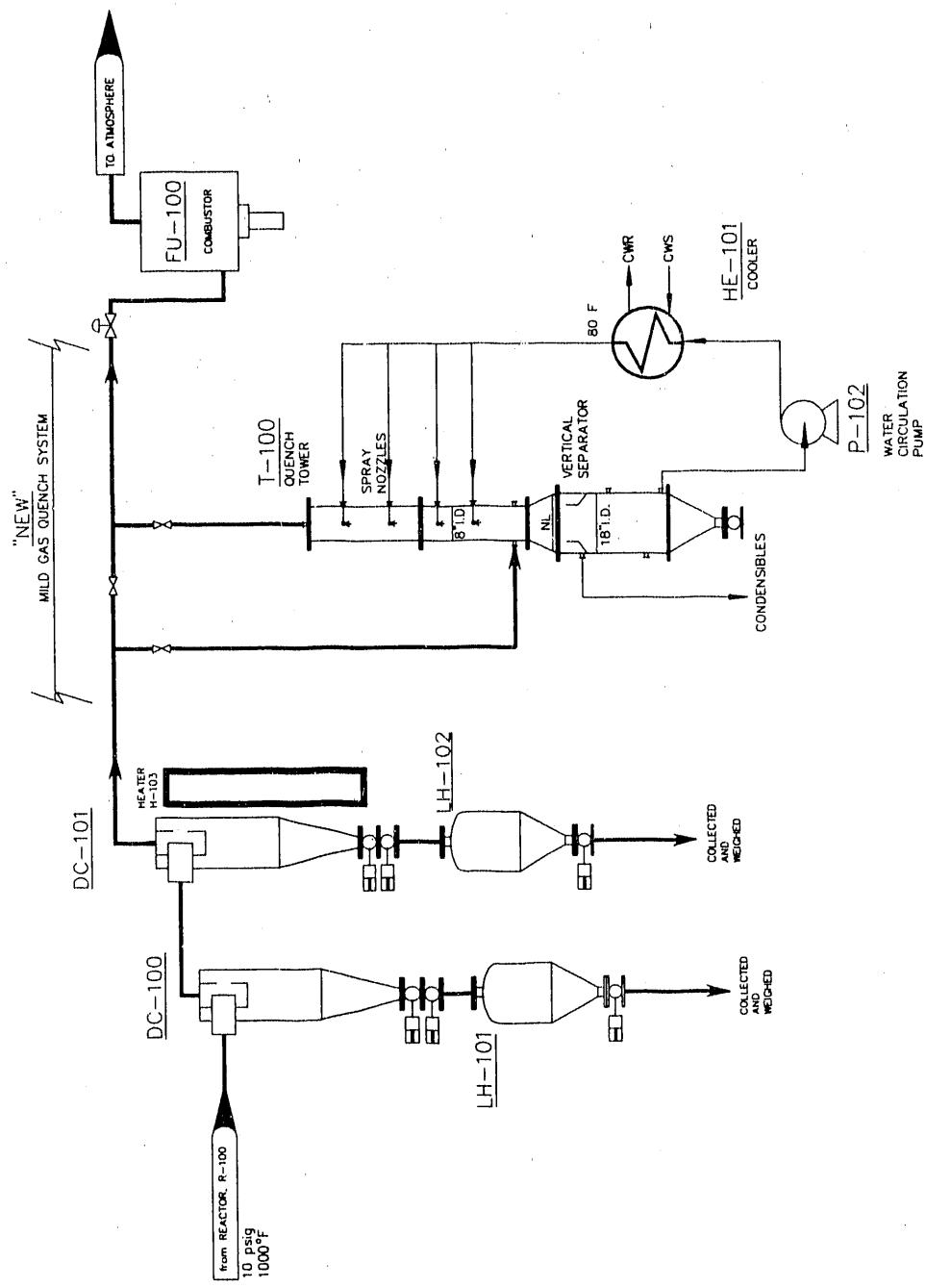


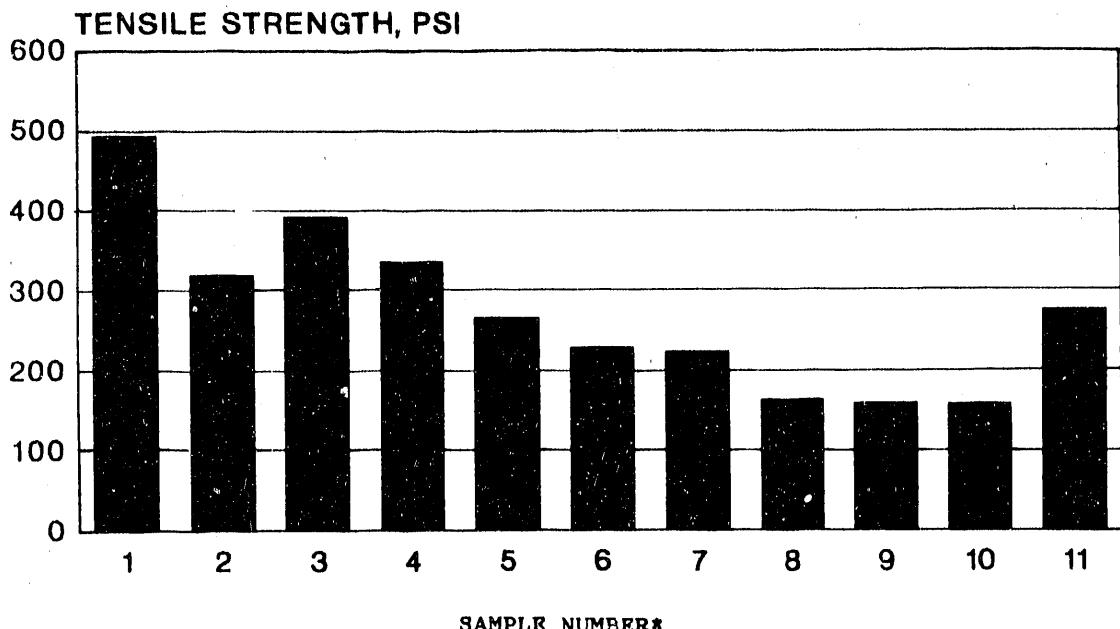
Figure 10. SCHEMATIC DIAGRAM OF TOTAL QUENCH SYSTEM

treatments to increase briquette strength. Briquettes to be used as smokeless fuel were cured at 400°F in air to polymerize and harden the binder; form coke briquettes using Illinois No. 6 coal as a binder were carbonized under a nitrogen atmosphere at 1800°F; and form coke briquettes using pitch as a binder were first cured at 400°F in air and then carbonized under nitrogen at 1800°F.

This work with lab-scale production and testing of various briquettes is useful for preliminary evaluation and screening of alternative briquetting methods, because different briquette properties are required for different applications, such as smokeless fuel or form coke.

A diametral compression test (ASTM No. B485-76) was used to evaluate the tensile strength of each briquette. The tensile strength test results of several initial briquettes produced in the hot mold are illustrated in Figure 11. These results are compared with values for two metallurgical cokes and one foundry coke described in the literature.² The hot briquettes made at 4,000 and 10,000 psi compression show strengths comparable with the commercial products. The pitch-bound briquettes formed under similar conditions have lower strength values. A possible reason for the lower strength of these briquettes could be that they were made with char used as-received from the PRU tests. The large particle size and the lack of particle-size control may have reduced the efficiency of the binder penetration, resulting in weaker briquettes. Briquettes with pitch binder can be made stronger by using -25 mesh char. Curing with air to polymerize the binder is also being examined for its effect on briquette strength.

Another potential application for mild gasification char could be as a low-cost adsorbent char material suitable for industrial water treatment use. Chars from PRU Tests MG-6 and MG-9 with a minimum of coke-breeze contamination and a char prepared in the laboratory were steam-activated at about 1560° to 1600°F in a 2-inch-I.D. reactor. These samples were then tested for their Iodine Numbers, a test designed to characterize the potential for adsorbing contaminants of large molecular size. The results are shown in Table 10 and in Figure 12. The Iodine Number values are comparable with those of commercial adsorbents produced from lignite and wood, but are generally lower than values for bituminous coal-based adsorbent carbon. One sample, T-1-50A, which was made from a char produced in the laboratory using a



<u>SAMPLE NO.</u>	<u>DESCRIPTION</u>	<u>BRIQUETTING PRESSURE</u>
1	Commercial Coke A*	
2	Commercial Coke B*	
3	Foundry Coke*	
4	Hot briq. 1:1 ratio of MG-9 char (-6 mesh) & raw coal**.	4000 psi
5	Hot briq. 1:1 ratio of MG-9 char (-6 mesh) & raw coal**.	10000 psi
6	12% pitch with MG-17 char (20x60 mesh).	10000 psi
7	12% pitch with MG-17 char (20x60 mesh).	19000 psi
8	12% pitch with MG-17 char (20x60 mesh).	4000 psi
9	Hot briq. 1:1 ratio of MG-17 char (20x60 mesh) and raw coal**.	4000 psi
10	Hot briq. 1:1 ratio of MG-17 char (20x60 mesh) and raw coal**.	10000 psi
11	Hot briq. 1:1 ratio of MG-9 char (-20 mesh) and raw coal**.	4000 psi

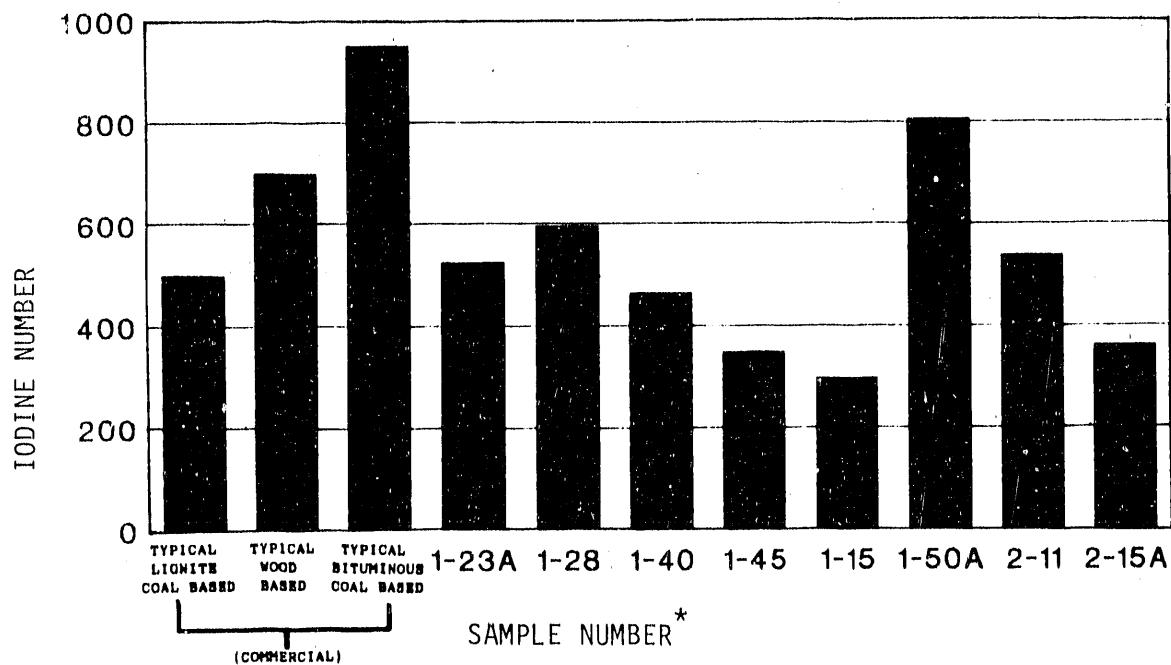
* These results were taken from Fuel, January 1972, Vol 51.

** Raw coal is IL No. 6.

Figure 11. DIAMETRAL COMPRESSION STRENGTH OF BRIQUETTES FROM MILD GASIFICATION CHAR COMPARED WITH COMMERCIAL COKES

Table 10. ADSORBENT CHAR PREPARATION DATA AND RESULTS

ACTIVATION CONDITIONS		TESTS				GAS				TECHNOLOGY	
Test Number	Char Source	1-23A	1-28	1-40	1-45	1-15	1-49	1-50A	2-11	2-15	
	Feed Char Composition (dry), wt %	MG-6	MG-6	MG-9	MG-9	MG-5	MG-5	1-50B	1-50B	MG-17	
Carbon	68.30	68.30	62.93	62.93	75.87	75.87	75.87	75.87	75.87	—	
Sulfur	2.67	2.67	3.53	3.53	2.12	2.12	—	—	—	—	
Ash	21.64	21.64	28.46	28.46	15.07	15.07	—	—	—	—	
Activation Temperature, °F	1607	1562	1562	1562	1607	1562	1562	1562	1562	1562	
Duration of Activation, min	60	100	150	127	124	120	120	120	120	240	
Activation Gas Composition, vol %											
N ₂	71.7	71.8	79.7	79.7	54.8	74.2	76.5	72.5	72.5	72.5	
H ₂ O	28.3	28.2	20.3	20.3	45.2	25.8	23.5	27.5	27.5	27.5	
% Carbon Burnoff	43	40	47	30	75	58	41	—	—	—	
Iodine Number	524.5	596.6	464.3	348.0	296.0	804.0	537.1	361.1	361.1	—	
Ball Pan Hardness Number	52.2	51.1	46.1	—	—	88.1	45.6	—	—	—	
Apparent Density, (g/ml)	0.27	0.27	0.32	0.29	—	0.43	0.25	—	—	—	
Sieve Analysis						—	—	—	—	—	
+6 mesh	—	—	—	—	—	—	—	—	—	—	
6 x 12 mesh	13.3	—	22.84	—	—	2.06	—	—	—	—	
12 x 20 mesh	29.4	—	15.72	—	—	10.89	36.57	—	—	—	
20 x 40 mesh	32.8	—	14.28	—	—	38.94	36.87	—	—	—	
40 x 60 mesh	19.8	—	18.74	—	—	35.80	19.63	—	—	—	
60 x 80 mesh	3.2	—	18.61	—	—	8.19	4.66	—	—	—	
-80 mesh	1.5	—	9.81	—	—	4.12	2.27	—	—	—	



* Sample number description in Table 10

Figure 12. IODINE NUMBER TESTS OF ACTIVATED CHAR FROM MILD GASIFICATION CHAR COMPARED WITH COMMERCIAL ADSORBENTS

preoxidized Illinois No. 6 coal, performed nearly as well as the commercial bituminous-based adsorbent.

For smokeless fuel applications, briquettes were formed in the laboratory press with New Enterprise Limestone Company dolomitic limestone and tested for post-combustion sulfur retention. The briquettes were prepared with 12% pitch binder, using a briquetting pressure of 4000 psi, and were cured at 400°F. The initial attempt at preparing and combusting limestone-containing briquettes showed that about 59% of the sulfur was retained as CaSO_4 . However, the amount of limestone used in these briquettes represented only a calcium-to-sulfur mole ratio of 1.4:1.0. The tests were repeated using briquettes made with a 2:1 calcium-to-sulfur mole ratio and burning a quantity of briquettes in a pile, which is more representative of the way smokeless fuel would be used. The sulfur retention increased substantially, with 82% sulfur retention reported for the middle of the pile, and 71% sulfur retention for a sample from the top of the pile, as shown in Table 11. In a commercial combustor or furnace, it is expected that more sulfur would be retained because of the increased contact of sulfur-containing gases with calcined limestone.

The formation and the initial testing of the briquettes show promise for a smokeless fuel application in terms of briquette strength and combustion with sulfur retention. Also, industrial adsorbent chars are another promising application of the activated mild gasification chars.

Work in the next quarter will concentrate on briquettes made for the metallurgical form coke application. Strength tests and reactivity tests will be examined for briquettes made with the mild gasification char from West Virginia metallurgical-grade coal.

Task 4. System Integration Studies

The prior scope of Task 4 was revised to a conceptual design and cost estimate of a 24-ton/day-capacity Process Development Unit (PDU). The PDU design is to be site-specific for installation at the Southern Illinois University, Carbondale, Illinois Coal Development Park in Carterville, Illinois. Progress to date has included evaluation of the data from the PRU tests and incorporation of these data in a process simulation model (ChemCAD II) of heat and material balances. Several flow schemes were outlined. The

Table 11. SMOKELESS FUEL WITH LIMESTONE COMBUSTION RESULTS

	<u>Uncombusted Briquette</u>	<u>Top Briquette</u>	<u>Center Briquette</u>
Ca:S mole ratio	2.21	2.70	2.47
Ash, wt% (ultimate)	24.71	84.00	83.60
Calcium, wt%	5.52	19.50	20.40
Total Sulfur, wt%	2.00	5.77	6.62
Sulfide S	0.79	0.081	0.15
Sulfate S	0.027	5.28	5.96
Pyritic S	0.12	0.012	0.014
Organic S	1.06	0.40	0.50
Sulfur Retention*, wt%	--	82	89

* Based on Ca:S ratios.

modeling has shown that several alternate schemes are possible for the heat input to the mild gasification process. The choices for process heat input are hot gases, hot char, and a combination of the two methods. The hot gas input could be from heated recycled product gas or a combusted flue gas. The combination of hot gas and hot char recycle offers the most advantage to the PDU operation and the design information it will develop.

Figure 13 shows the feed preparation, mild gasification, and heat supply design for the PDU using indirect recycled product gas heating. Figure 14 shows the same plant sections using heat supply with flue gas from direct burning of recycled product gas. Figure 15 shows the PDU condensate recovery scheme.

The system model is also being revised to analyze the heat effects of a separate fines feed to the entrained flow gasifier section for comparison with that of a single coal feed to the bubbling fluidized bed. A proposed configuration and design basis for the PDU will be completed in the next reporting period. This will also serve as the basis for the final conceptual design and preliminary capital cost estimate.

OTHER WORK

A paper entitled "Potential for Transportation Fuels Produced by Mild Gasification" was presented at the Sixth Annual International Pittsburgh Coal Conference in Pittsburgh, Pennsylvania on September 26, 1989.¹ In this paper,

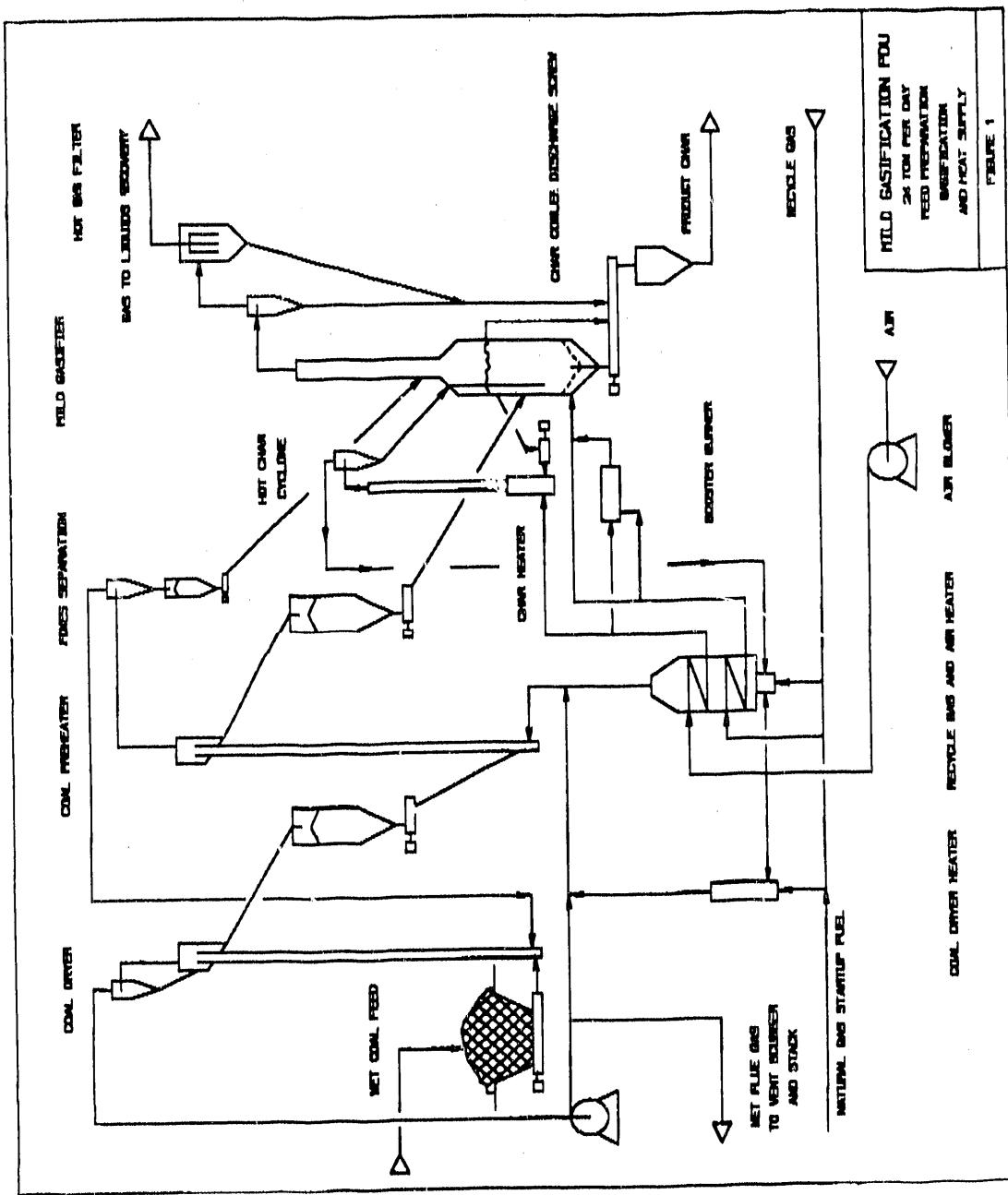


Figure 13. PDU FEED PREPARATION, GASIFICATION, AND HEAT SUPPLY
(Indirect Recycle Gas Heating)

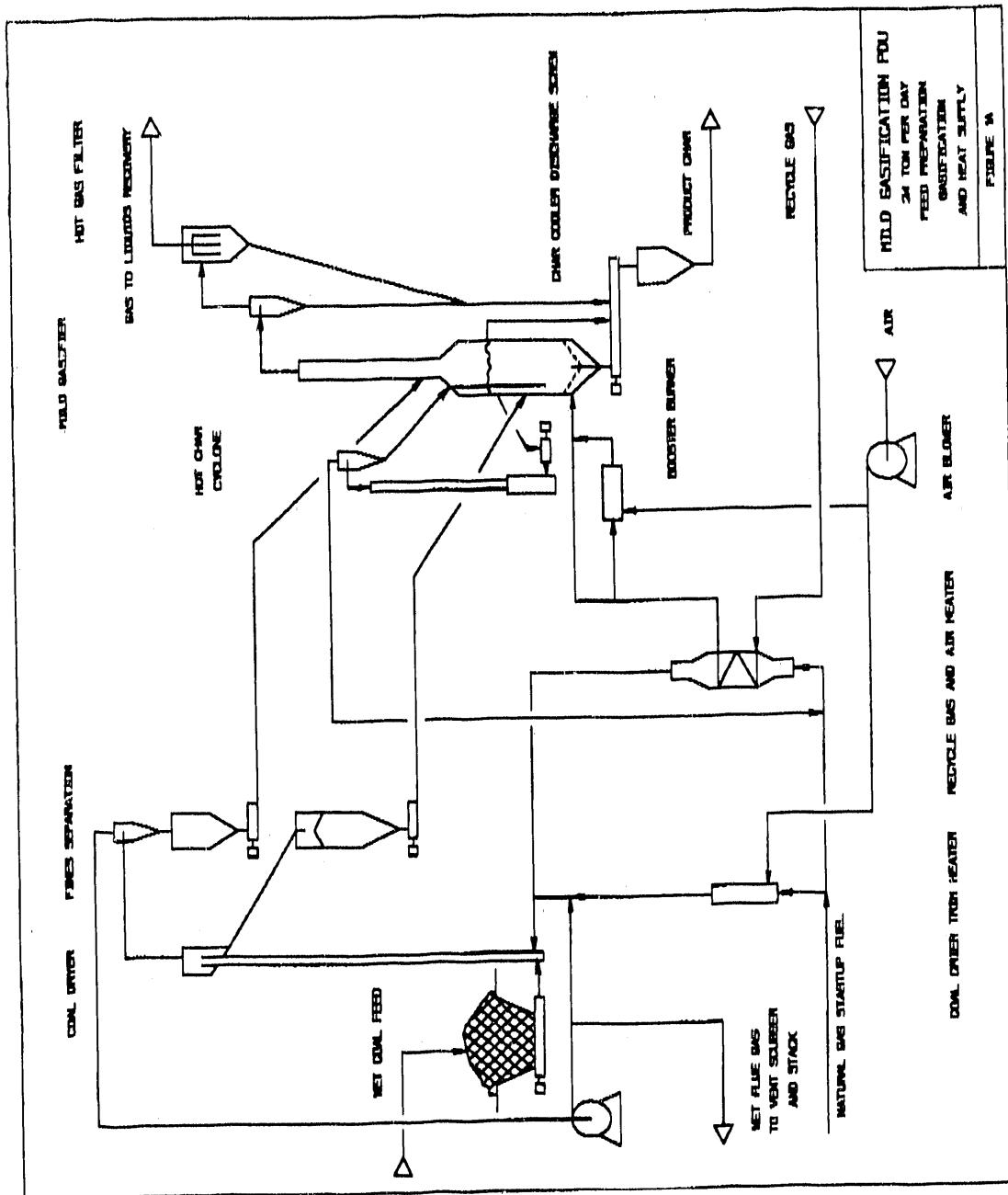


Figure 14. PDU FEED PREPARATION, GASIFICATION, AND HEAT SUPPLY
(Direct Recycle Gas Heating)

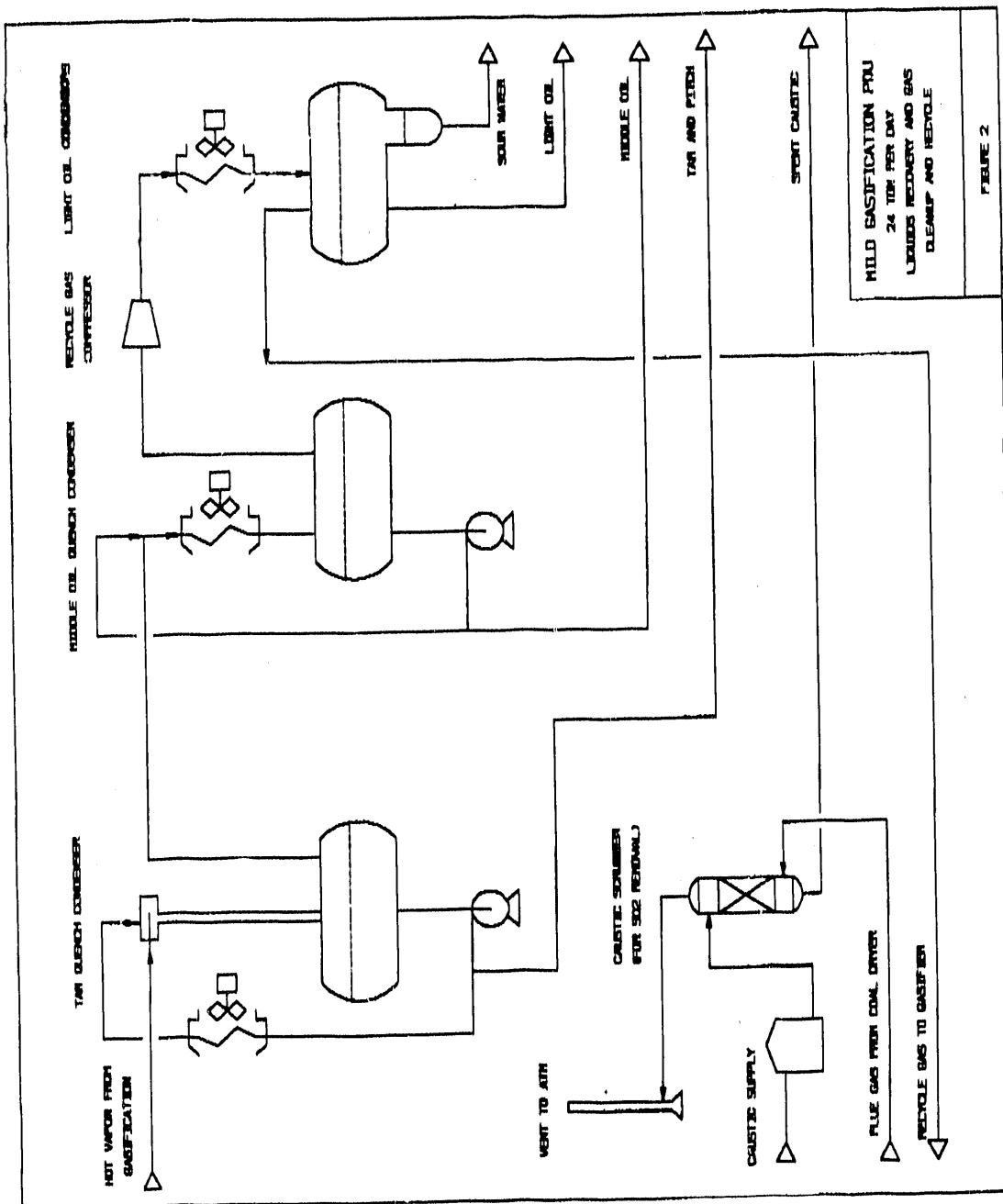


Figure 15. PDU CONDENSATE RECOVERY

the potential for producing diesel fuel or a diesel blending stock from the IGT mild gasification condensables was discussed, based on the preliminary PRU data from Tests SD-6 and MG-12. The possibility of deriving high-density fuel precursors from mild gasification liquids was also presented, and some potential candidate compounds were identified. It was concluded that high-temperature particulate removal and hydrotreatment and/or chemical upgrading of mild gasification liquids would be necessary to produce fuel-grade products.

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2. Patrick, J. W. and Stacey, A. E., "The Strength of Industrial Cokes: Part I. Variability of Tensile Strength in Relation to Fissure Formation", Fuel 51:1, 81-87, 1972.

17WP/RPP/61089qr

APPENDIX A.
Analytical Data From PRU Tests
Performed During Quarter

A-1

Table A-1. ANALYSES OF SOLIDS FROM TEST MG-11
Test Temperature = 1390°F

	Feed Mixture	Coal	Diluent	Fluid- Bed Char	1st Cyclone Char	2nd Cyclone Char	Carry-over Char
<u>Proximate Analysis, wt %</u>							
Moisture	2.21	3.20	1.12	0.24	0.41	1.57	2.95
Volatile matter	19.09	34.73	3.44	3.82	5.62	8.09	13.91
Ash	13.28	15.12	11.44	15.87	15.80	16.55	44.28
Fixed carbon	65.42	46.85	84.00	80.07	78.17	73.79	38.86
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00
<u>Ultimate Analysis, dry wt %</u>							
Carbon	75.05	64.98	85.12	81.08	79.41	76.54	47.47
Hydrogen	2.37	4.38	0.35	0.31	0.70	1.24	0.73
Sulfur	2.33	3.95	0.70	1.15	1.57	1.58	6.28
Nitrogen	1.36	1.51	1.21	1.32	1.43	1.64	1.18
Oxygen (by diff.)	5.31	9.59	1.05	0.23	1.03	2.19	0.00
Ash	13.58	15.59	11.57	15.91	15.86	16.81	44.35
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Heating Value, Btu/lb	--*	--	--	--	--	--	--
Particle Density, g/cm ³	--	1.46	1.82	--	--	--	--
<u>Particle-Size Distribution, wt % retained on screen (mesh)</u>							
6	--	0.00	0.00	0.30	0.00	0.00	--
12	--	1.69	21.30	35.10	0.00	0.00	--
20	--	6.58	26.90	37.50	0.22	0.00	--
30	--	--	--	--	--	--	--
40	--	31.00	20.80	22.35	5.64	0.83	--
60	--	35.70	11.90	3.75	23.95	3.31	--
70	--	--	--	--	--	--	--
80	--	16.90	6.26	0.42	16.65	9.09	--
100	--	4.51	2.68	0.12	8.92	1.65	--
120	--	--	--	--	--	--	--
140	--	2.26	3.58	0.12	12.50	5.79	--
170	--	--	--	--	--	--	--
200	--	0.38	2.46	0.00	10.50	5.79	--
230	--	0.19	0.67	0.00	3.75	0.83	--
270	--	0.19	0.89	0.00	4.27	1.65	0.00
325	--	0.19	0.67	0.12	3.56	1.65	25.00
PAN	--	0.41	1.89	0.22	10.04	69.41	75.00
	--	100.00	100.00	100.00	100.00	100.00	100.00

* Not determined.

Table A-2. ANALYSES OF SOLIDS FROM TEST MG-12
Test Temperature = 1250°F

	<u>Feed Mixture</u>	<u>Coal</u>	<u>Diluent</u>	<u>Fluid- Bed Char</u>	<u>1st Cyclone Char</u>	<u>2nd Cyclone Char</u>	<u>Carry-over Char</u>
<u>Proximate Analysis, wt %</u>							
Moisture	2.22	3.32	1.12	0.58	0.91	2.14	7.54
Volatile matter	19.34	35.23	3.44	5.20	6.51	12.41	31.28
Ash	12.78	14.11	11.44	16.33	14.54	18.97	42.96
Fixed carbon	65.66	47.34	84.00	77.89	78.04	66.48	18.22
	100.00	100.00	100.00	100.00	100.00	100.00	100.00
<u>Ultimate Analysis, dry wt %</u>							
Carbon	75.28	65.43	85.12	80.60	80.06	74.44	53.54
Hydrogen	2.39	4.43	0.35	0.39	0.87	1.35	NA
Sulfur	2.26	3.81	0.70	1.27	1.64	1.49	NA
Nitrogen	1.25	1.28	1.21	1.29	1.44	1.41	NA
Oxygen (by diff.)	5.75	10.49	1.05	0.02	1.32	1.93	NA
Ash	13.07	14.56	11.57	16.43	14.67	19.38	46.46
	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Heating Value, Btu/lb	--*	--	--	--	--	--	--
Particle Density, g/cm ³	--	1.46	1.82	--	--	--	--
<u>Particle-Size Distribution, wt % retained on screen (mesh)</u>							
6	--	0.00	0.00	0.00	0.00	0.00	--
12	--	1.69	21.30	15.70	0.00	0.00	--
20	--	6.58	26.90	43.40	0.19	0.00	--
30	--	--	--	--	--	--	--
40	--	31.00	20.80	31.60	2.64	0.00	--
60	--	35.70	11.90	6.82	19.20	1.59	--
70	--	--	--	--	--	--	--
80	--	16.90	6.26	1.14	17.50	1.59	--
100	--	4.51	2.68	0.45	9.62	0.00	--
120	--	--	--	--	--	--	--
140	--	2.26	3.58	0.45	14.20	1.59	--
170	--	--	--	--	--	--	--
200	--	0.38	2.46	0.23	12.10	1.59	--
230	--	0.19	0.67	0.00	4.42	0.00	--
270	--	0.19	0.89	0.00	5.19	1.59	--
325	--	0.19	0.67	0.00	3.65	0.00	--
PAN	--	0.41	1.89	0.21	11.29	92.05	--
	--	100.00	100.00	100.00	100.00	100.00	--

* Not determined.

Table A-3. ANALYSES OF SOLIDS FROM TEST MG-14
Test Temperature = 1100°F

<u>Feed</u>	<u>Coal</u>	<u>Diluent</u>	<u>Fluid-Bed</u>	<u>1st Cyclone</u>	<u>2nd Cyclone</u>	<u>Carry-over</u>
<u>Mixture</u>			<u>Char</u>	<u>Char</u>	<u>Char</u>	<u>Char</u>
<u>Proximate Analysis, wt %</u>						
Moisture	2.36	3.60	1.12	0.91	0.86	2.37
Volatile matter	19.62	35.80	3.44	6.34	7.42	10.30
Ash	13.48	15.52	11.44	16.99	13.89	18.22
Fixed carbon	64.54	45.08	84.00	75.76	77.83	69.11
	100.00	100.00	100.00	100.00	100.00	100.00
<u>Ultimate Analysis, dry wt %</u>						
Carbon	74.42	63.72	85.12	77.31	79.13	73.53
Hydrogen	2.34	4.33	0.35	0.66	1.05	1.19
Sulfur	2.25	3.80	0.70	1.45	1.74	1.42
Nitrogen	1.17	1.13	1.21	1.19	1.33	1.16
Oxygen (by diff.)	6.01	10.97	1.05	2.24	2.74	4.04
Ash	13.81	16.05	11.57	17.15	14.01	18.66
	100.00	100.00	100.00	100.00	100.00	100.00
Heating Value, Btu/lb	--*	--	--	--	--	--
Particle Density, g/cm ³	--	1.46	1.82	--	--	--
<u>Particle-Size Distribution, wt % retained on screen (mesh)</u>						
6	--	0.00	0.00	0.98	0.00	0.00
12	--	1.69	21.30	34.60	0.00	0.00
20	--	6.58	26.90	34.80	0.07	0.00
30	--	--	--	--	--	--
40	--	31.00	20.80	20.60	1.85	0.30
60	--	35.70	11.90	5.39	18.05	1.56
70	--	--	--	--	--	--
80	--	16.90	6.26	1.23	19.10	1.56
100	--	4.51	2.68	0.49	10.75	0.97
120	--	--	--	--	--	--
140	--	2.26	3.58	0.74	14.85	2.32
170	--	--	--	--	--	--
200	--	0.38	2.46	0.25	11.80	3.00
230	--	0.19	0.67	0.25	4.20	0.97
270	--	0.19	0.89	0.25	4.72	1.94
325	--	0.19	0.67	0.25	3.53	1.94
PAN	--	0.41	1.89	0.17	11.08	85.44
	--	100.00	100.00	100.00	100.00	100.00

* Not determined.

Table A-4. ANALYSES OF SOLIDS FROM TEST MG-15
Test Temperature = 1025°F

	<u>Feed Mixture</u>	<u>Coal</u>	<u>Diluent</u>	<u>Fluid- Bed Char</u>	<u>1st Cyclone Char</u>	<u>2nd Cyclone Char</u>	<u>Carry-over Char</u>
<u>Proximate Analysis, wt %</u>							
Moisture	1.98	2.84	1.12	0.46	0.70	1.59	4.54
Volatile matter	17.85	32.26	3.44	5.31	7.98	11.18	26.68
Ash	12.27	13.09	11.44	14.71	13.17	19.08	64.57
Fixed carbon	67.91	51.81	84.00	79.52	78.15	68.15	4.21
	100.00	100.00	100.00	100.00	100.00	100.00	100.00
<u>Ultimate Analysis, dry wt %</u>							
Carbon	76.56	67.99	85.12	80.28	79.91	73.09	--
Hydrogen	2.38	4.40	0.35	0.65	1.21	1.24	--
Sulfur	1.99	3.28	0.70	1.32	1.68	1.21	--
Nitrogen	1.34	1.46	1.21	1.34	1.52	1.37	--
Oxygen (by diff.)	5.23	9.41	1.05	1.65	2.42	3.70	--
Ash	12.52	13.46	11.57	14.78	13.26	19.39	67.64
	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Heating Value, Btu/lb	--*	--	--	--	--	--	--
Particle Density, g/cm ³	--	1.46	1.82	--	--	--	--
<u>Particle-Size Distribution, wt % retained on screen (mesh)</u>							
6	--	0.00	0.00	0.00	0.00	0.00	--
12	--	1.69	21.30	18.90	0.00	0.00	--
20	--	6.58	26.90	50.76	0.14	0.00	--
30	--	--	--	--	--	--	--
40	--	31.00	20.80	23.00	2.63	0.00	--
60	--	35.70	11.90	4.61	14.42	0.88	--
70	--	--	--	--	--	--	--
80	--	16.90	6.26	1.03	15.62	0.88	--
100	--	4.51	2.68	0.43	10.21	0.88	--
120	--	--	--	--	--	--	--
140	--	2.26	3.58	0.51	16.32	0.88	--
170	--	--	--	--	--	--	--
200	--	0.38	2.46	0.26	13.42	0.88	--
230	--	0.19	0.67	0.08	4.85	0.00	--
270	--	0.19	0.89	0.18	5.40	0.88	--
325	--	0.19	0.67	0.08	3.88	0.88	--
PAN	--	0.41	1.89	0.18	13.12	93.84	--
	--	100.00	100.00	100.00	100.00	100.00	--

* Not determined.

Table A-5. OILS AND TARS ANALYSES FROM TESTS MG-11, MG-12, MG-14, AND MG-15

Test Number	MG-11	MG-12	MG-14	MG-15
Test Temperature, °F	1390	1250	1100	1025
Elemental Analysis of Oils and Tars^a				
Ash	0.53	0.36 ^b	0.25 ^b	0.65 ^b
Carbon	82.29	75.74	77.97	73.86
Hydrogen	6.08	6.21	6.76	6.85
Nitrogen	1.26	1.27	0.98	0.79
Sulfur	3.21	2.03	1.83	1.57
Oxygen (by diff.)	6.63	14.38	12.21	16.28
Total	100.00	100.00	100.00	100.00
H/C Atomic Ratio	0.88	0.98	1.03	1.11
Simulate Distillation by Gas Chromatography^c				
Cumulative Wt% Recovered	Boiling Point, °F			
5	340	353	388	351
10	390	433	487	408
15	437	506	576	466
20	482	573	661	525
30	568	705	840	652
40	652	844	--	810
50	739	1023	--	--
60	831	--	--	--
70	939	--	--	--
EP (end point) ^d	1093	1093	1093	1093
% Residue at EP	20.3	47.6	60.6	53.4

^a Determined by evaporation at 100°F, 15-20 mm Hg; light oils boiling below approximately 300°F are not included.

^b Corrected for THF-soluble FeCl_3 which was determined to be a contaminant from corrosion of stainless steel exposed to the hot methylene chloride quench solvent.

^c Correction applied for heteroatom content of coal liquids, which is not accounted for in standard simulated distillation method for petroleum-based liquids.

^d Characteristic of chromatographic column and method, not necessarily true end point of distillation.

Table A-6. COMPONENT ANALYSES OF FULL-RANGE OILS AND TARS FROM TESTS MG-11, MG-12, MG-14, AND MG-15

Test Number	MG-11	MG-12	MG-14	MG-15
Test Temperature, °F	1390	1250	1100	1025
<u>Component, wt % of total oils and tars^a</u>				
Benzene	8.7	1.2	0.9	0.5
Toluene	5.5	1.3	1.5	0.8
Xylenes	2.8	0.8	0.5	0.5
Ethylbenzene	2.2	0.6	0.1	0.1
Indene	2.0	0.4	0.2	0.1
Styrene	1.2	0.3	0.2	0.2
Other light oils	1.4	4.3	6.0	5.4
Total light oil ^b	23.8	8.9	9.4	7.6
Phenol	2.8	1.0	1.2	0.3
Cresols	2.0	1.4	1.7	1.7
Xylenols	0.6	0.6	1.4	1.3
Naphthalene	2.0	0.5	0.1	0.1
Other middle oils	13.6	11.8	6.9	15.7
Total middle oil ^c	21.0	15.3	11.3	19.1
Heavy oil ^d	15.3	11.7	8.8	11.1
Pitch ^e	39.9	64.1	70.5	62.2
Total oils and tars	100.0	100.0	100.0	100.0

^a Includes light oils which are not included in the oils and tars of Table A-5.

^b Atmospheric boiling point < 360°F; estimated from simulated distillation data.

^c Atmospheric boiling point 360° to 590°F.

^d Atmospheric boiling point 590° to 750°F.

^e Atmospheric boiling point >750°F.

Table A-7. GAS COMPOSITIONS FROM PRU TESTS MG-11, MG-12, MG-14, AND MG-15

Test Number	MG-11	MG-12	MG-14	MG-15
Test Temperature, °F	1390	1250	1100	1025
<u>Component</u>	<u>Mol % in gas, nitrogen-free</u>			
H ₂	44.3	37.5	28.4	29.5
CO	17.0	16.1	13.0	10.6
CO ₂	9.1	8.7	10.3	12.9
CH ₄	18.8	23.3	28.1	28.1
C ₂ H ₄	6.0	6.3	6.5	5.8
C ₂ H ₆	1.1	2.5	4.3	4.7
C ₃ H ₆	1.2	2.1	2.7	2.5
C ₃ H ₈	0.1	0.2	1.0	0.6
H ₂ S	2.4	3.3	5.7	5.3
Total	100.0	100.0	100.0	100.0
Molecular weight	16.0	17.5	19.9	19.9
Higher heating value, Btu/lb	546	626	719	694

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