

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

Quarterly Technical Progress Report
for the Period July - September 1990

by

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U.S. Department of Energy
Morgantown Energy Technology Center
P.O. Box 880
Morgantown, West Virginia

October 1990

Work Performed Under DOE METC Contract No. DE-AC21-87MC24267

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

1.0 INTRODUCTION

The primary objective of the EERC/AMAX Mild Gasification project is to commercialize a process that will produce several value-added products from a high-sulfur midwestern bituminous or a low-sulfur subbituminous coal. Indiana No. 3 and Wyodak coals are being tested in a 100-lb/hr process development unit (PDU) that has undergone shakedown testing and is undergoing parametric testing. This unit will provide the engineering data for use in determining the technical and economic data required for the design of a commercial-scale mild gasification plant.

The design and construction of the 100-lb/hr PDU is complete. Several key capabilities/features incorporated in the design philosophy that will be crucial for scaleup facilities planning include:

- The ability to determine the effect of steam on equipment operation and yield structures.
- The use of mechanical mixing to reduce particle agglomeration.
- The use of process-derived condensables as cooling liquor.
- The use of process-derived water or water addition as cooling liquor.
- The ability to determine optimum residence times.
- The ability to test sorbents for sulfur reduction.
- The capability to produce sufficient product for end-user evaluation.

Figure 1 shows a schematic of the two integrated reactors with the associated carbonizer/condensation train, char hopper collection vessel, steam superheater, and coal feed hopper. The first reactor operates in a fast fluidized-bed mode at a temperature that will produce the optimum condensable product in terms of both quality and quantity. The char from this reactor may have a sufficiently reduced volatile content for use in carbon black production or Pellet Technology Corporation (PTC) pellet production. The second reactor will be used for optimizing char volatile content for these or other processes, such as activated carbon or formcoke production. The second reactor will operate in a bubbling bed mode.

In a commercial-scale process, system heat and carbonizing gas would be provided by an atmospheric fluidized-bed combustor (AFBC). For economic reasons, carbonizing gas for the PDU will be stoichiometrically combusted natural gas and steam. The coal is pneumatically transported into the bottom of the reactor, and char is withdrawn from the top of the bed.

The quench system contains two options: recycled condensable scrubbing or water scrubbing. In the first option, a high-temperature tar venturi scrubber is used to remove any remaining particulates from the gas stream and to condense boiling point fractions of liquid products ranging in temperature from 350° to 700°F (175° to 380°C). Further cooling takes place in a sieve

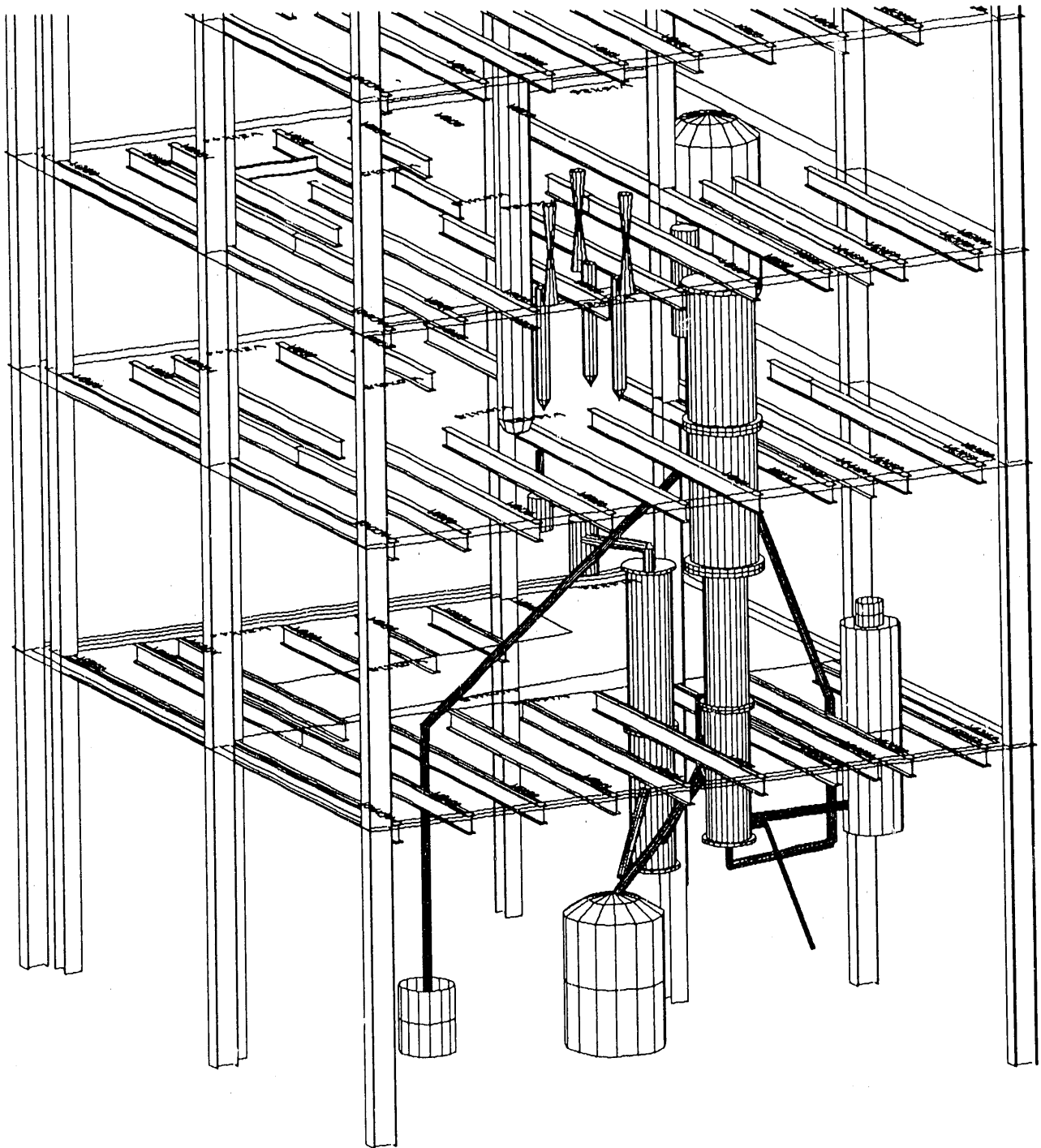


Figure 1. EERC mild gasification process development unit (PDU).

tower, again using recycled product liquor. This unit operates at an exit temperature just above the dew point of the product gas, approximately 160° to 180°F (70° to 80°C). The product gas then passes through a water scrubber and a demister to ensure that organic material does not escape and pass through the flare system. The second quench system option uses two water scrubbers to condense all of the tar, oil, and water from the system before the product gas is flared.

An on-line mass spectrometer is used to analyze the product gas stream to determine whether it contains a sufficient quantity of condensable organics to warrant further processing (such as a Rectisol process) for their removal. This experimentation is being done on a bench-scale basis.

Because wastewater treatment processes and solvent extraction of coal products in water streams are well documented, the present phase of the EERC mild gasification program only encompasses bulk treatment of the condensables for safe disposal of all liquid products. An activated sludge unit/char filter is being used for wastewater treatment while liquid-liquid extraction using di-isopropyl ether is being used for phenol recovery.

Work during the quarter included the shakedown of the carbonizer tar/oil quench system, the performance of tests using Wyodak and Indiana No. 3 coals, and the initiation of parametric testing. Testing of the Wyodak product chars as carbon black and metallurgical coke substitute feedstocks was also performed.

2.0 CARBONIZER TESTS

2.1 Carbonizer Tests

To date, nine tests have been conducted on the carbonizer reactor using both the water and tar/oil scrubbing systems (see Table 1). One test was conducted with hot gas, one with petroleum coke, six with Wyodak coal, and one with Indiana No. 3 coal. Operating parameters for the reactor included temperatures from 750°F to 1200°F (400° to 650°C), fluidization velocities of 2.5 to 8 ft/sec, and pneumatic coal transport velocities of 16 to 28 ft/sec. Fines carryover to the cyclones has varied from below 10% to as much as 30% of the -1/4-inch feed material, depending on fluidization velocity. Typical exit temperatures from the reactor were within 100°F (40°C) of the bed temperature when devolatilizing the coal.

2.2 Product Yields

One set of material balances was attempted, but the results from these tests are not available. Product yields have been estimated measuring solids flow and using ash and nitrogen as tie elements. The condensables and water yields are then estimated based on difference. Char and gas yields are very comparable to those obtained using the 4-lb/hr continuous fluidized-bed reactor (CFBR); therefore, condensable yields should also be very similar. PDU and CFBR results are compared in Table 2. The apparent increase in gas production with the associated loss of char is actually due to the small amount of excess air that was not combusted in the natural gas burner.

TABLE 1
CARBONIZER TESTS

Run Number	Coal	Temperature (°F/°C)	Quench System	Parameter
P001	Refractory Cure	2200/1200	Water	Shakedown Gas System
P002	Petroleum Coke	1100/600	Water	Shakedown Solids Handling
P003	Wyodak	1100/600	Water	Shakedown Water Quench
P004	Wyodak	1100/600	Water	
P005	Wyodak			
P006	Wyodak	1020/550	Tar/Oil	Shakedown Tar/Oil
P007	Indiana	1020/550	Tar/Oil	Shakedown Indiana Coal
P008	Wyodak	820/440	Tar/Oil	Quench Liquid Entrainment
P009	Wyodak	820/440	Tar/Oil	Quench Liquid Entrainment

TABLE 2
MILD GASIFICATION PRODUCT YIELDS (wt% maf coal feed)

Reactor	Wyodak		Indiana	
	CFBR	PDU	CFBR	PDU
Gas	30.0	33.0	12.0	16.0
Water	--	--	5.0	5.0 ¹
Condensables	10.0	8.5 ¹	18.0	18.0 ¹
Char	60.7	58.5	65.0	61.0

¹ Estimated by difference and comparison to 4-lb/hr CFBR data.

2.3 Char Analysis

Table 3 shows proximate analyses for char products from a Wyodak test (P005), which are also comparable to CFBR test results. Analysis of the primary and secondary cyclone fines indicated that there was little difference between the product char and the fines. The excess blowover in this test (>30%) was due to several instances when the fluidization velocity approached 6 ft/sec. Figures 2 and 3 show the particle-size distributions for the individual solid products and the combined (total) product, respectively.

TABLE 3
WYODAK SOLIDS PROXIMATE ANALYSES AT 1100°F (wt%)

	Feed Coal	CFBR Char	PDU Products			
			Total PDU Char Product	Product Char	Primary Cyclone Char	Secondary Cyclone Char
Moisture	23.9	0	1.9	1.5	2.5	4.2
Volatiles	33.8	12.6	15.4	15.7	15.0	16.0
Fixed Carbon	37.9	76.2	72.1	73.9	69.2	65.5
Ash	4.4	11.2	10.6	8.9	13.4	14.4

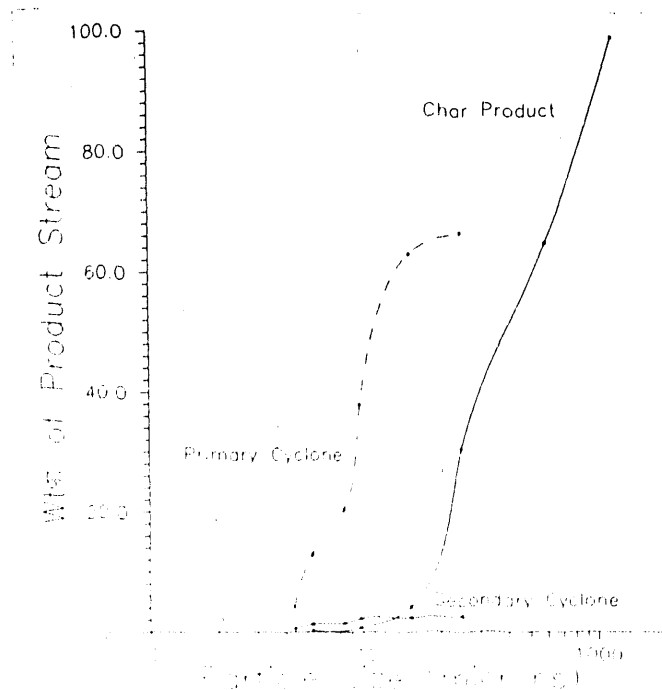


Figure 2. Wyodak char and cyclone fines particle-size distribution.

Tables 4, 5, and 6 show proximate and sulfur analyses of the char products obtained from Tests P006, P007, and P009, respectively. Table 4 shows that the volatile contents of the Wyodak primary and secondary cyclone fines do not differ significantly from the volatile contents of the product char, while the Indiana cyclone fines (Table 5) contain significantly more volatile material than the Indiana product char. This was due to the high fluidization velocities utilized during Test P006, which blew bed material into the cyclones. The cyclone fines from Test P007 are more representative of material that became entrained in the fluidization gas stream and had insufficient residence time to devolatilize. In Table 5, "Char From Plenum" refers to char that was removed from the plenum following completion of the test and a cooldown period. No char was recovered for proximate analysis from the secondary cyclone during Test P009, as shown in Table 6.

2.4 Condensable Liquid Analysis

2.4.1 Analytical Methods Development

Since the tar scrubber and the sieve tower start-up fluids are petroleum-derived, it is important to establish when, during operation of the reactor, the recycle cooling liquids are essentially composed of only coal-derived tars and oils. Two analytical methods will be used to determine when this occurs: aliphatic-to-aromatic ratio determination and simulated distillation. Because the start-up fluids are petroleum-derived, their aliphatic-to-aromatic ratios will be higher than those of the more aromatic coal-derived liquids. During operation, as the system approaches steady-state

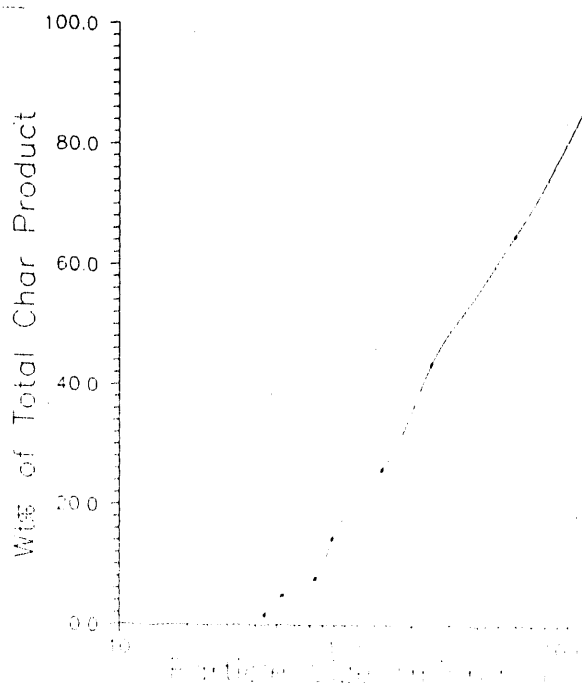


Figure 3. Wyodak char and cyclone fines combined particle-size distribution.

TABLE 4
 PROXIMATE AND SULFUR ANALYSES (wt%): TEST P006

	Wyodak Feed Coal (1/4 x 0)	Product Char	Primary Cyclone Char	Secondary Cyclone Char
Moisture	23.88	1.39	1.99	6.11
Volatiles	33.82	23.79	25.24	18.80
Fixed Carbon	37.90	66.62	61.57	62.22
Ash	4.41	8.21	11.21	12.85
Sulfur, mf	0.63	0.27	0.59	0.51

TABLE 5
 PROXIMATE AND SULFUR ANALYSES (wt%): TEST P007

	Indiana Feed Coal (1/4 x 0)	Product Char	Primary Cyclone Char	Secondary Cyclone Char	Char From Plenum
Moisture	8.08	1.45	1.81	4.69	1.21
Volatiles	40.20	11.53	19.91	19.51	9.28
Fixed Carbon	45.00	69.14	65.72	63.23	69.73
Ash	6.72	17.88	12.57	12.56	19.78
Sulfur, mf	4.07	3.60	0.67	0.61	3.31

operating conditions, the aliphatic-to aromatic ratio of a liquid sample will approach that of a pure coal-derived liquid. This ratio will steadily fall until the system is at equilibrium and operating on coal-derived liquids.

Simulated distillation of each liquid sample will be performed using capillary gas chromatography. The technique works as follows: A condensable sample (liquid or tar) is dissolved in methylene chloride at a concentration of approximately 20 milligrams per milliliter (mg/mL), internal standards are added, and the mixture is injected into a gas chromatograph (GC). The temperature in the GC oven is maintained at 122°F (50°C) for two minutes, after which it is raised to 626°F (330°C) at a rate of 14.4°F (8°C) per minute. The chromatogram obtained is compared with a chromatogram of a carefully prepared mixture of normal alkanes (C7, C8, C9 ..., C25) obtained under identical chromatographic conditions. Since the boiling point of each alkane

TABLE 6
 PROXIMATE AND SULFUR ANALYSES (wt%): TEST P009

	Wyodak Feed Coal (1/4 x 0)	Product Char	Primary Cyclone Char	Secondary Cyclone Char
Moisture	23.88	1.35	2.10	tar
Volatiles	33.82	30.16	28.06	in
Fixed Carbon	37.90	60.73	59.24	cyclone
Ash	4.41	7.76	10.59	
Sulfur, mf	0.63	0.40	0.71	

is known, the relationship of boiling point to GC retention time can be calculated. Once this relationship is determined, the boiling point of any compound in the sample can be approximated based on a comparison of the unknown compound's GC retention time to the retention times of the alkane standard compounds that "bracket" the unknown compound; i.e., if an unknown compound has a retention time midway between the retention times of C7 and C8, the compound is assigned a boiling point midway between the boiling points of C7 and C8. After assigning boiling points to all compounds detected in a sample (this operation is computerized because, in most samples, about 200 to 400 compounds are detected), a plot is generated of cumulative "area percent" of a sample distilled versus temperature. Area percent refers to how the GC detector quantitates, on a relative basis, each compound in a sample. (In the ASTM D-86 distillation method, the distillate is quantitated on the basis of volume percent distilled.)

Because simulated distillation relies on area percent values and assigned boiling points, a simulated distillation curve for a given sample may differ somewhat from the actual distillation curve for the sample. However, the technique is valuable because it can be automated to provide reproducible data for use in comparing a large number of liquid samples on the basis of volatility. Figure 4 compares simulated and ASTM D-86 distillation curves obtained for Diesel #2 (labeled "Fuel Oil" in the figure) and shows that the two techniques give comparable, but not identical distillation profiles. One of the reasons for the difference is number of data points. The simulated distillation curve is plotted using about 200 to 400 data points, which are derived from the calculated distillation temperature of each component detected in a sample, while the ASTM distillation curve is plotted using about 10 data points, which are derived from the temperatures at which specified volumes of a sample (5, 10, 20, 30% ..., etc.) are distilled.

Another reason for the difference in the distillation curves yielded by the two techniques may be the effect of flash volatilization, which occurs during the simulated distillation process when the sample is injected into the gas chromatograph. To separate the methylene chloride chromatography solvent

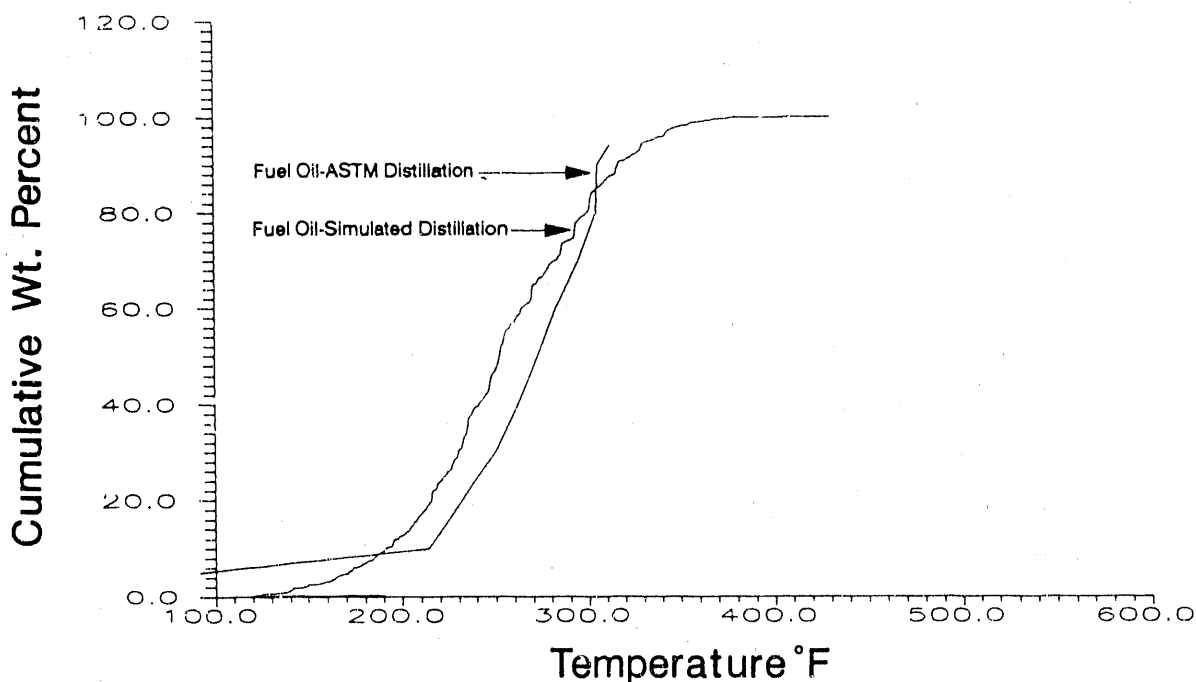


Figure 4. ASTM/simulated distillation comparison.

from the sample components, the temperature in the injection port on the gas chromatograph is maintained at 608°F (320°C). Upon injection of the sample, the solvent is immediately vaporized and begins moving through the 60-meter long GC column at a rate approximately equivalent to the carrier gas flow rate. The solvent is unreactive with the liquid phase that coats the inside of the GC column. The sample components are also volatilized, but, unlike the solvent, each component moves through the column at a rate dependent on the extent of its reactivity with the liquid phase. Since a simulated distillation sample (normally about 1 microliter in volume) is essentially separated into its constituent components by flash volatilization, the components are unable to exert attractive forces on each other. However, during an ASTM D-86 distillation, the sample (normally about 100 milliliters in volume) is slowly heated, which allows the higher-boiling components to exert attractive forces on the lower-boiling components, thereby artificially raising the boiling points of the lower-boiling components.

Figure 4 also shows the effect of sample decomposition at high temperatures, the onset of which is designated as the temperature at which the sample begins to emit black smoke. Because of decomposition, the distillation had to be terminated before 100% of the sample had distilled (the distillation was approximately 94% complete). In Figure 4, this decomposition is evident by the increased (steeper) slope of the ASTM curve at high temperatures, which is a result of less sample collected with increasing temperature (more sample is decomposed). The next quarterly report will describe a comparison of simulated and ASTM distillation of the Mandan decant oil.

2.4.2 Condensable Liquid Analytical Results

Five tests were run using the water-scrubbing system. The first scrubber entrance temperature was approximately 850°F (455°C) with an exit temperature of 180°F (80°C). The exit temperature of the second scrubber was 80°F (30°C). Figure 5 compares the condensable product boiling point distributions (obtained using simulated distillation, which is explained in an upcoming section of this report) of Wyodak chars produced in the PDU and the CFBR. The shift to higher-boiling material in the PDU was probably due to tar polymerization during the 10-day period that elapsed before the sample was analyzed. This polymerization was quite evident, since the tar was fluid when transferred from the preliminary collection barrel and solid when analyzed ten days later. Sunlight and/or air reaction with wastewater were also evident when transferring the wastewater, as the water changed from yellowish brown to red when exposed to the atmosphere. The observation of tar polymerization and wastewater reactivity is evidence that condensable product stabilization, analysis, and upgrading must be performed as soon as possible after removal of the condensables from the reactor. Figure 6 shows the boiling point distribution of the phenolic constituents of the condensables, which make up approximately 17% of the total condensable product.

Two tests were conducted using the tar/oil quench system. The entrance temperature to the tar scrubber is maintained at 850°F (455°C) with the exit temperature held between 350° and 700°F (175° and 370°C). The sieve tower exit temperature is maintained at 130° to 180°F (55° to 80°C), and the water scrubber exit temperature is 80° to 100°F (25° to 40°C). The system is started up with Diesel #2 in the sieve tower and Mandan Decant Oil (a petroleum

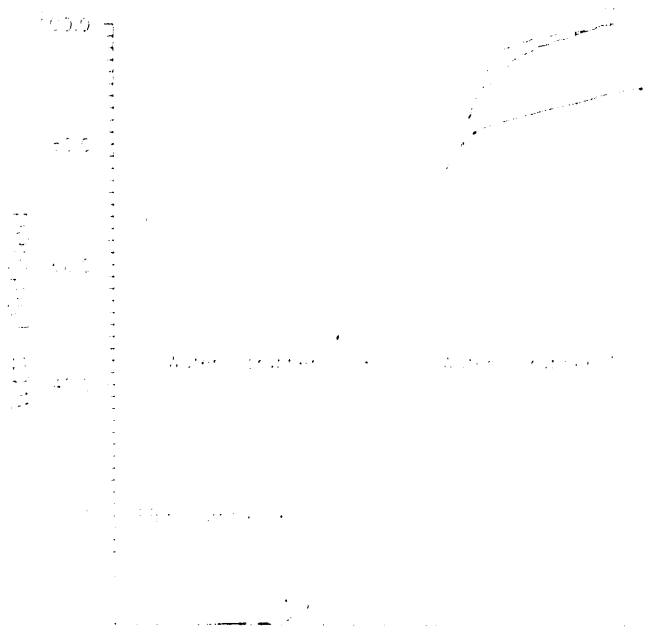


Figure 5. Wyodak condensable boiling point distribution.

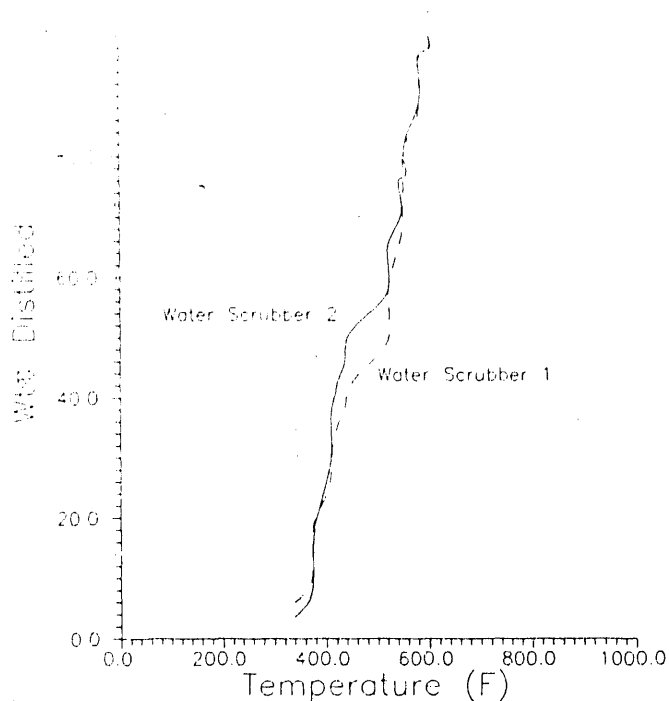


Figure 6. Wyodak condensable phenolic constituents boiling point distribution.

processing resid roughly equivalent to Fuel Oil #6) in the tar scrubber. As the amount of coal-derived tar/oil increases in the quench systems, the petroleum-derived materials are increasingly replaced with coal liquids. To date, this system has shown the greatest ease of operation and the least amount of plugging. Figure 7 shows the condensation train collection points.

Due to liquids entrainment problems, start-up fluids were continually added during Tests P006 and P007. Because of these additions, the recycle fluids did not become completely coal-derived. Figure 8 shows the simulated distillation curves for liquids from each unit operation. The heaviest products were obtained from the tar scrubber, followed by the sieve tower, demister, water scrubber and back-pressure cyclone. This progression of heaviest to lightest products follows the order in which the liquids flow through the condensation train, except for the demister, which is last, and should theoretically yield the lightest product. Figure 9 shows simulated distillation curves for samples taken from each tray in the sieve tower. No significant difference in product volatility is evident.

Figure 10 shows simulated distillation curves for P007 liquids. The boiling point distributions for these liquids are shifted toward lighter, lower boiling products than P006 liquids. This was expected, as Indiana No. 3 coal has yielded lighter liquid product slates than Wyodak coal in tests in the CFBR. The tar scrubber and sieve tower liquids provided essentially identical curves, with lighter material being removed from the water scrubber.

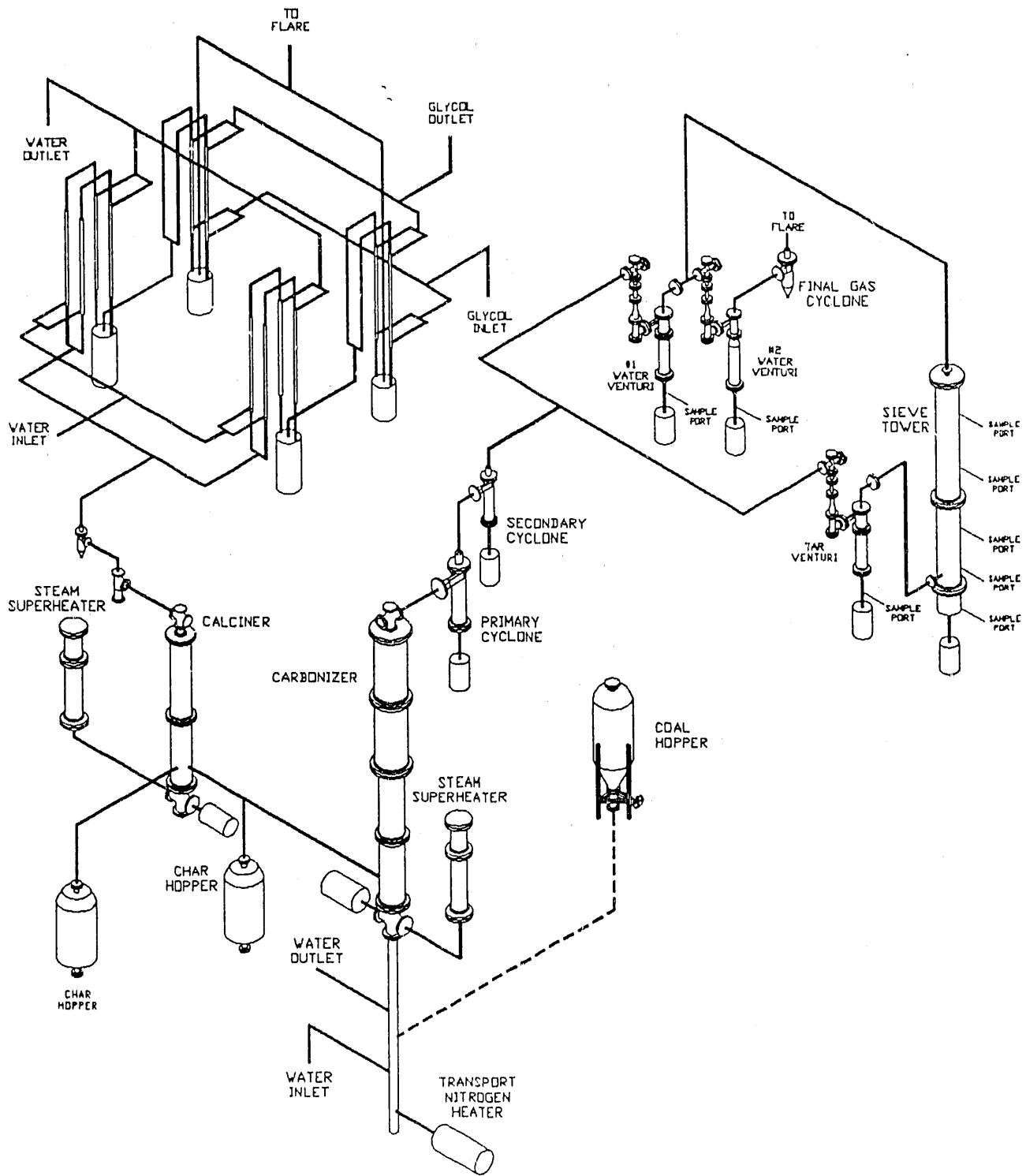


Figure 7. Condensation train collection points.

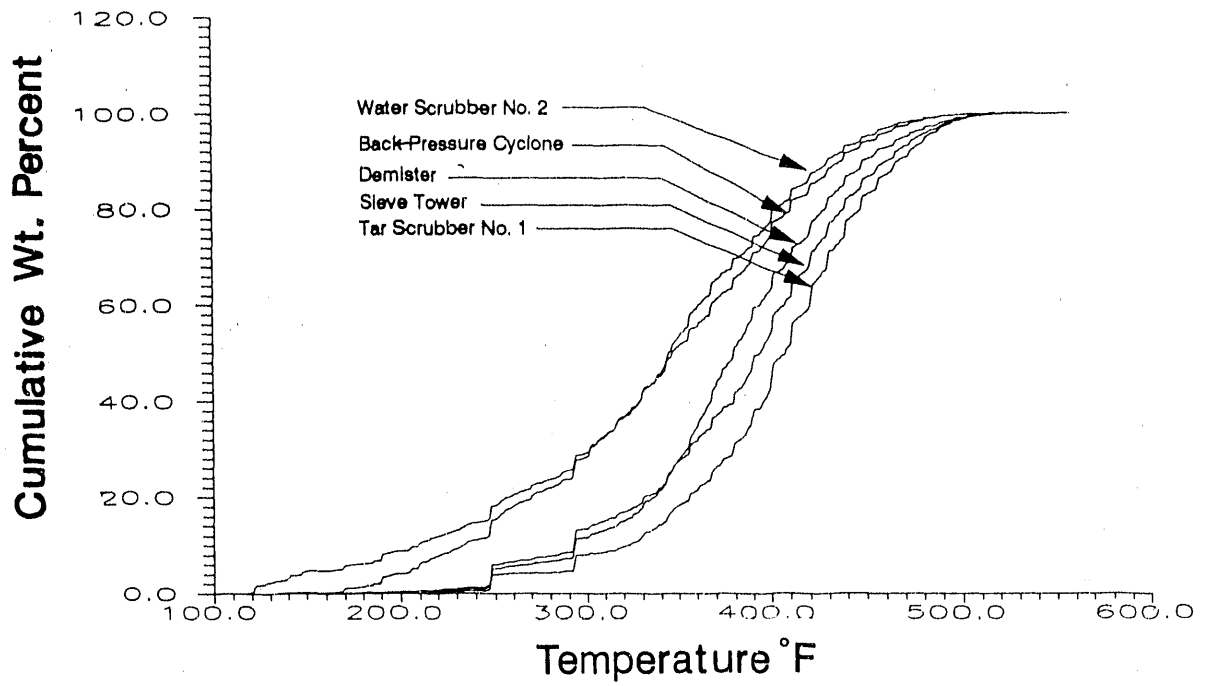


Figure 8. Simulated distillation curves for liquids from each unit operation.

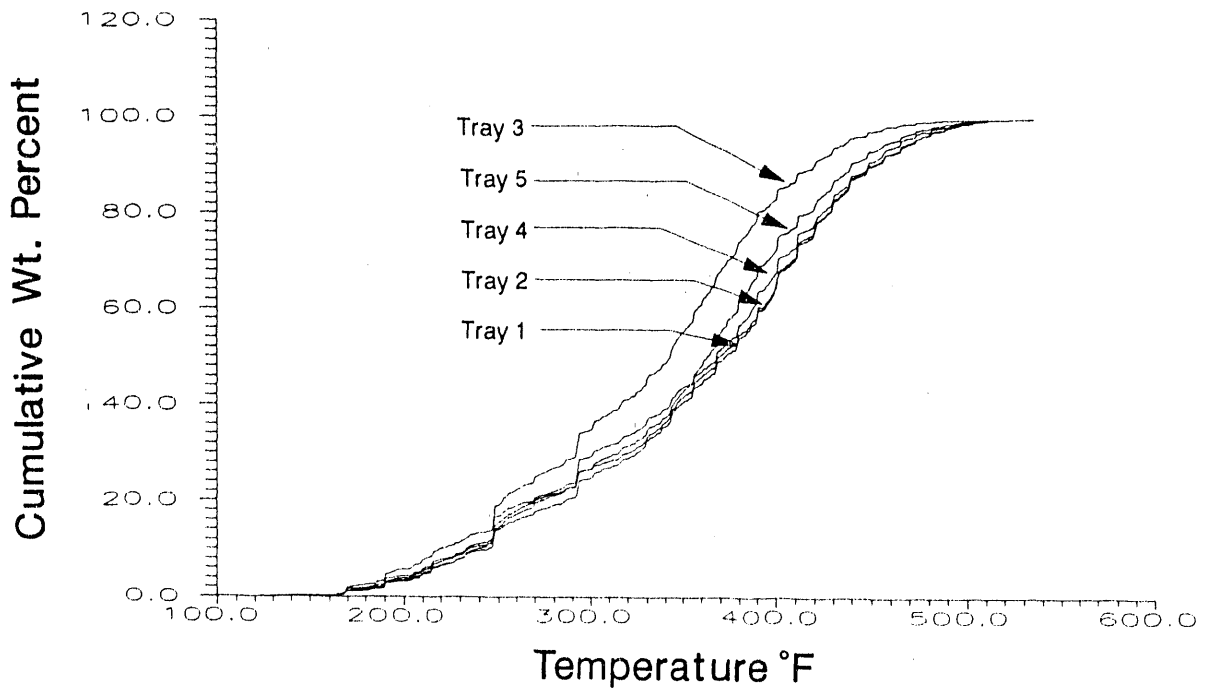


Figure 9. Simulated distillation curves for samples from the sieve tower.

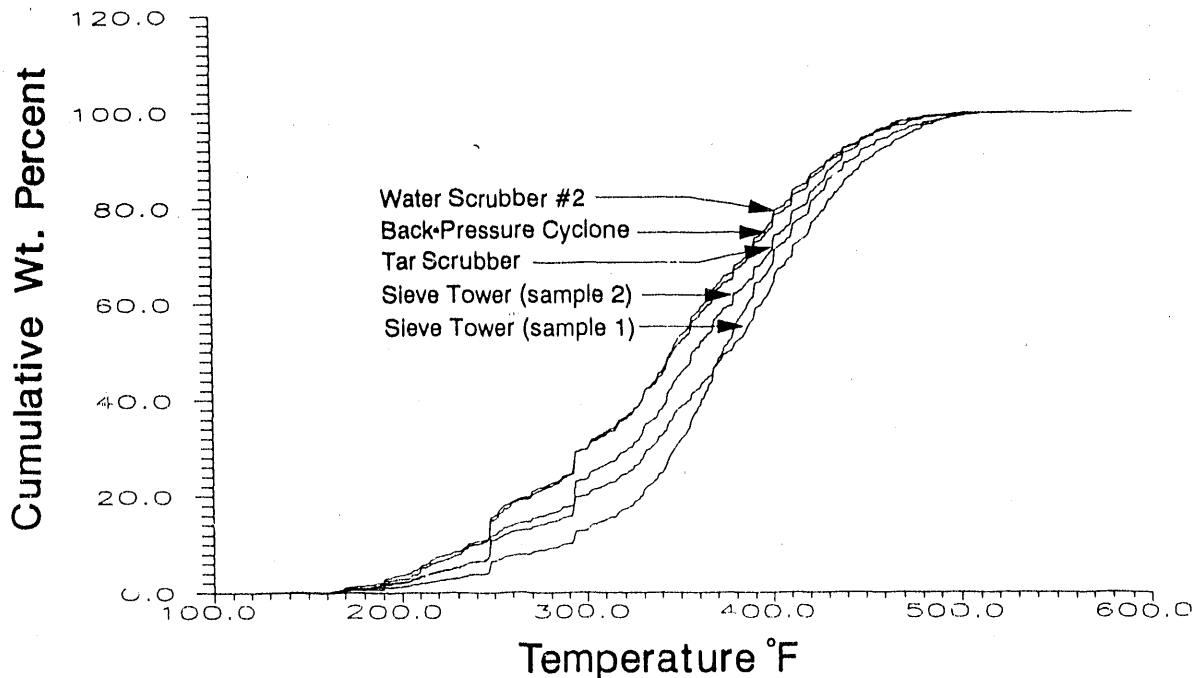


Figure 10. Simulated distillation curves - P007 liquids.

Figure 11 shows simulated distillation curves for liquids collected during Test P009, along with curves for the two start-up fluids. The P009 liquids were removed from the system via "knockout pots," which are used to collect all fluids (gases or liquids) that pass through or are blown out of the quench system. The knockout pots contained a mixture of the two start-up fluids and coal-derived liquids and tars. Figure 11 shows that the knockout pot liquids (which probably contained primarily start-up fluids) were significantly lighter than the knockout pot tars (which appeared to be primarily coal-derived, based on their greenish color and coal-tar odor). The tar was removed as semisolid material with the consistency of cold molasses from the bottom of the collection barrel, which was located outside of the reactor building. The temperature at the time the tar was sampled was about 45°F (10°C). After being warmed to room temperature, the tar took on the consistency of a viscous liquid, similar to a heavy motor oil. The tar removed from the secondary cyclone polymerized soon after exposure to ambient temperature.

2.5 Wastewater Analysis

Condensables were not quantified during these tests, but wastewater samples and water-insoluble tar samples were obtained. Table 7 shows the organic material breakdown of wastewater samples obtained from two collection barrels. Samples were obtained from both the top and bottom of the barrel using a peristaltic pump. Estimated concentrations were based on the GC/MS

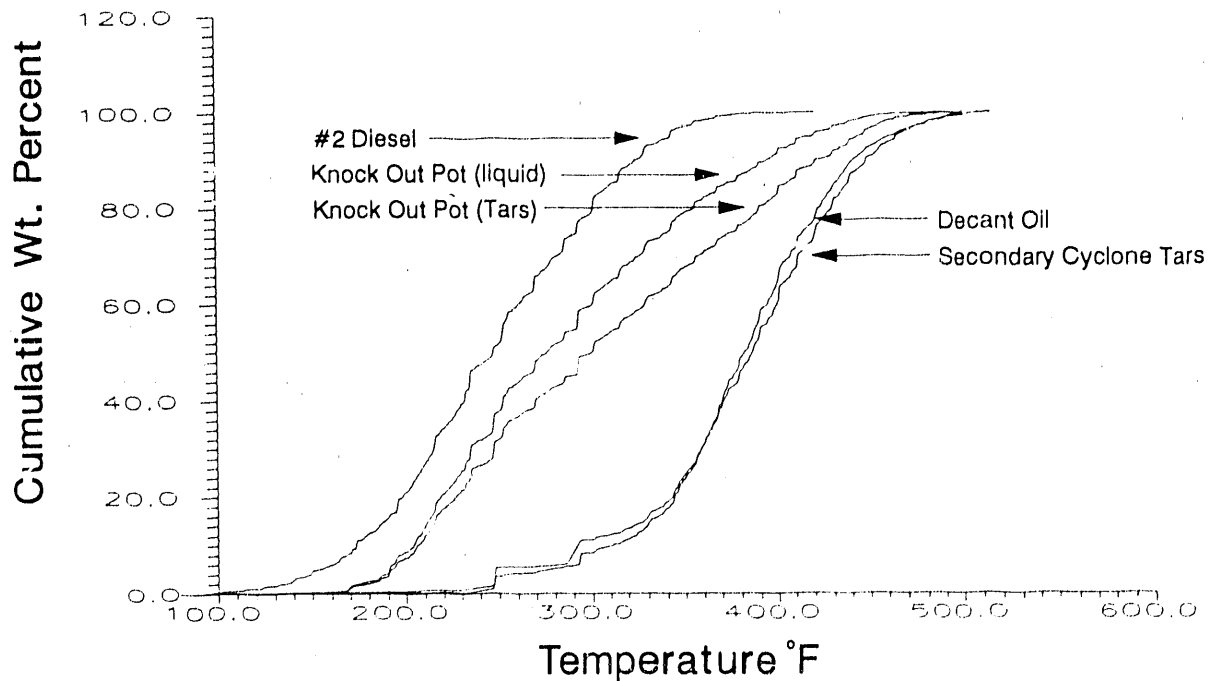


Figure 11. Simulated distillation curves - P009 liquids.

analysis of acid-base/neutral extractions of the wastewater, after it had been spiked with deuterated phenol and pyridine internal standards. The GC/MS data, which identified a large portion of the TOC, indicates that the major constituent of the wastewater organics is phenolic-based material. The analysis of the water sample from the demister, which is located downstream from the main condensation train, indicates that a small amount of organics were not condensed out of the product gas.

2.6 Quench System Entrainment Studies

During Tests P004-P007, it became evident that cooling liquid could not be kept in the venturi scrubbers due to the high gas velocities needed to maintain reactor operating temperatures of 1000°-1100°F (540°-595°C). Since the only heat source is fluidization gas, a combination of factors are involved in keeping the reactor at a specific operating temperature. These factors include the amount of combustion gas, the moisture level of the feed coal, the amount of condensables produced at the operating temperature, and the fluidization gas velocity. During the initial runs, in order to maintain the reactor at a temperature of 1100°F (540°C), 5000-7000 scfh of gas was needed. At this velocity, it was not possible to maintain liquid levels in any of the venturi scrubber cyclones. As a result, a small amount of start-up cooling liquid was constantly added to each cyclone. The blown over cooling liquid was collected downstream in the demister vessels. To determine the lower limit of the gas velocity that would cause liquid entrainment, lower temperatures were maintained in the carbonizer during Tests P008 and P009.

TABLE 7
WYODAK WASTEWATER ORGANIC CONCENTRATIONS

Species	Concentration (ppm)			
	Water Scrubber 1, Bottom ¹	Water Scrubber 1, Top ¹	Demister, Top ¹	Demister, Bottom ¹
Phenol	1100	1200	500	600
Cresols	350	200	300	400
C ₂ -Phenols	30	20	25	40
C ₃ -Phenols	4	20	3	4
Naphthols	15	10	ND ²	ND
Catechol	100	350	ND	ND
Methylcatechol	15	15	ND	ND
Pyridine	3	2	10	10
C ₁ -Pyridines	10	10	30	20
C ₂ -Pyridines	4	3	6	5
Indole	20	15	10	10
Naphthalene	5	7	3	2
Total	1656	1852	887	1091
TOC	3020	3110	1143	1218
TC	4203	4216	1726	1793

¹ Level at which sample was taken from barrel.

² Not detected.

Because these lower temperatures do not provide acceptable devolatilization, a cold flow test program was initiated that involved simulating the action of the venturi scrubber cyclones using a cyclone fabricated from plexiglas. Figure 12 shows the cyclone entrainment test stand (CETS) developed for the simulation studies. Table 8 describes the tests performed using the CETS.

During the first two tests, it was evident that the cyclones did not function adequately, as cooling liquid was entrained and removed from the system at a rate of 2 gpm. The entrainment was due to several factors, discovered during the tests, that necessitated a significantly different design for a liquid/gas cyclone as compared to a solid/gas cyclone. The design modifications required to improve the liquid/gas cyclone are currently being reviewed for patent application status and will be disclosed in an appropriate format at a later date. These modifications to the cyclones will be made at EERC and will not delay the program.

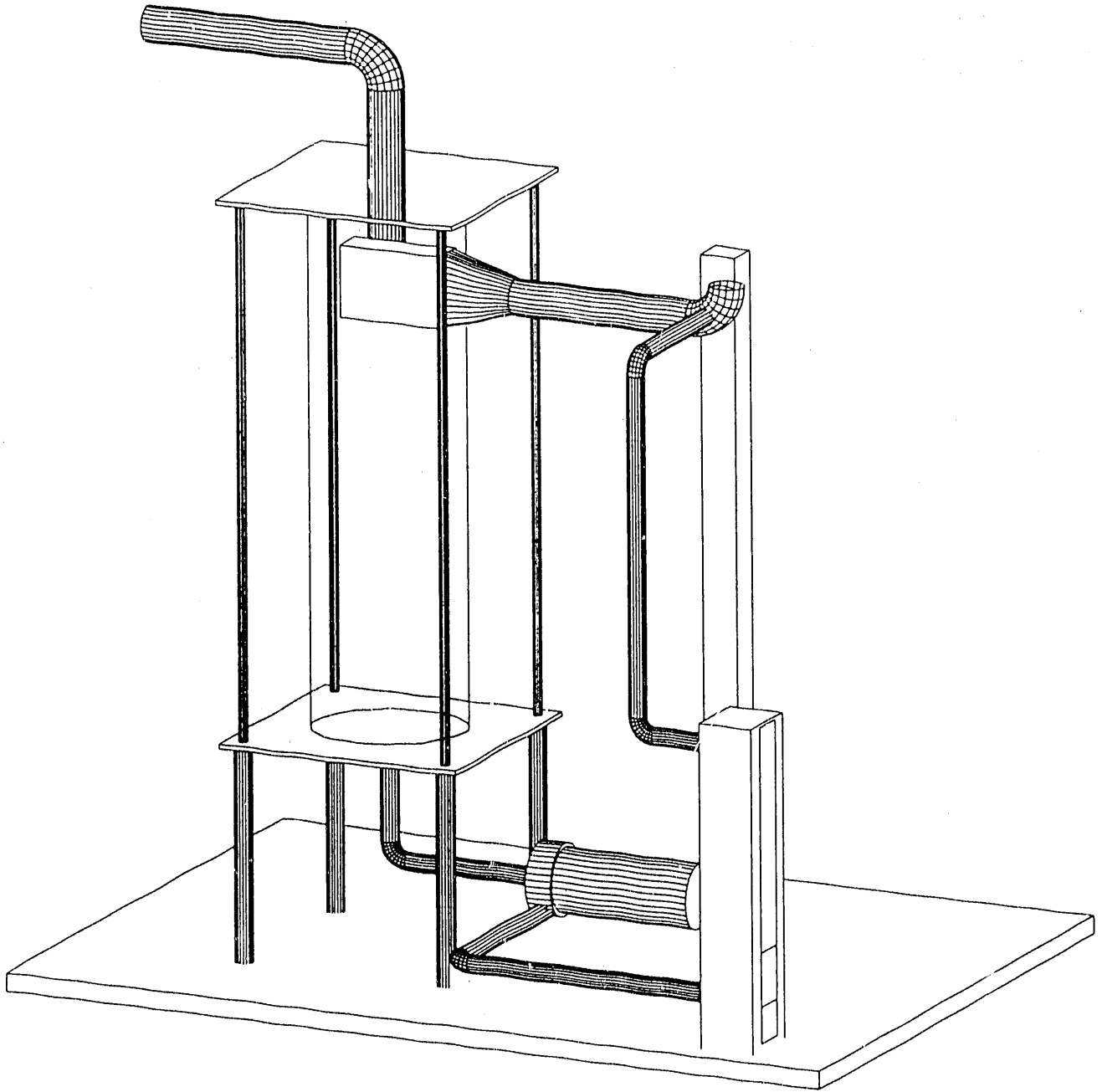


Figure 12. Cyclone entrainment test stand (CEIS).

TABLE 8
COLD FLOW ENTRAINMENT STUDIES

Test Number	Cyclone Size	Gas Flow (scfh)	Recycle Rate (gph)	Parameter
T001	4"	5000	22	Norm. Op.
T002	6"	7000	22	Norm. Op.
T003	8"	7000	22	Norm. Op.
T004	10"	7000	22	Norm. Op.
T005	12"	7000	22	Norm. Op.
T006	4"	5000	22	Modified
T007	6"	7000	22	Modified
T008	8"	7000	22	Modified
T009	10"	7000	22	Modified
T010	12"	7000	22	Modified

3.0 CHAR UPGRADING

Pellet Technology Corporation (PTC) has utilized Wyodak and Indiana No. 3 coal chars as metallurgical coke substitutes in iron ore reducing tests. The tests were performed using pellets made from EERC mild gasification char (either Wyodak char from the 4-lb/hr CFBR or Indiana char from the 100-lb/hr PDU), iron ore, lime, and silica. The steel industry has specified that, for a coal char to be suitable for use as a coke substitute, its sulfur and ash contents should be less than 1 and 10 wt%, respectively. Table 9 shows these target values, along with the sulfur and ash contents of a Wyodak char (carbon) pellet and an iron ore/Wyodak char (iron/carbon) pellet.

After pressing, the pellets were dried, hardened, and subjected to temperatures of 2500 and 2700°F (1370 and 1480°C). Figure 13 shows the iron reduction times required for the iron/carbon pellets described in Table 9. Table 10 shows the results of pelletizing tests performed using Wyodak char prepared at 1200°F (650°C). Comparison of these results with results of tests performed with Indiana char (Table 11) shows that although the moisture contents of green (undried) pellets are similar regardless of the char used, the hardened pellets made from Indiana char have a significantly higher compressive strength. Weight loss during the reduction process was slightly higher for the Wyodak pellets at both reaction temperatures.

4.0 AMAX R&D PROJECT ACTIVITY

4.1 Magnetic Separation of Indiana No. 3 Coal and Char

The effect of cleaning Indiana No. 3 coal by magnetic separation prior to mild gasification was investigated. The 14 x 50-mesh and 50 x 100-mesh

fractions of Chinook feed coal were subjected to permanent roll magnetic separation. As shown in Table 12, the nonmagnetic fractions of these feeds contained less ash but more sulfur than the magnetic fractions.

For comparison, an as-received Chinook coal sample and a nonmagnetic fraction of the Chinook feed coal were subjected to laboratory mild gasification tests at 1020°F (550°C) at AMAX R&D. The resulting chars were then separated using the permanent roll separator. Table 13 shows the results. Overall, the char produced from the nonmagnetic fraction of the feed coal contained less ash but more sulfur than the char produced from the as-received feed coal.

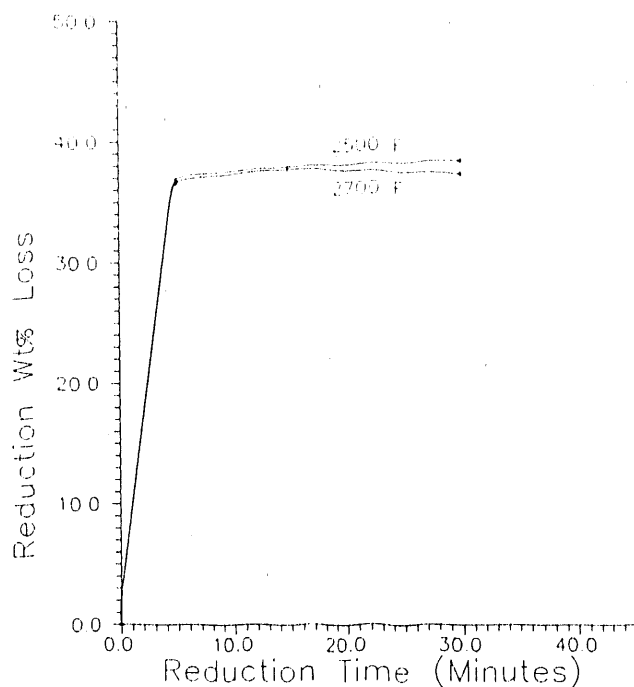


Figure 13. PTC iron/carbon pellet reduction time.

TABLE 9

WYODAK CHAR PELLETT CHARACTERISTICS

	Char Target Value	Iron/Carbon Pellet	Carbon Pellet
Sulfur Content	<1%	0.05%	0.3%
Ash Content	<10%	8.80%	18.0%

TABLE 10
RESULTS OF PELLET TECHNOLOGY CORPORATION CHAR
PELLETIZING TESTS USING WYODAK CHAR

	Test No. 22 (7/11/90)	Test No. 23 (7/16/90)	Test No. 24 (7/23/90)	Test No. 25 (7/31/90)	Test No. 26 (8/6/90)
Pellet Composition, %					
Empire Iron Ore Concentrate	75	--	--	75	--
Coal Char*	18	90	92	18	92
Burned Lime Fines	5	7	7	5	7
Silica Sand Fines	2	3	1	2	1
Pellet Diameter, inches					
	0.59	0.75	0.75	0.59	0.75
Green Pellet Characteristics					
Moisture, weight %	12.3	27.7	27.6	9.9	30.0
Compressive Strength, lb	9.9	16.7	15.7	12.8	17.4
Impact Number (18 inches)	12.3	>25	16.4	17.6	>25
Dried Pellet Characteristics					
Compressive Strength, lb	7.0	7.7	9.6	22.7	6.3
Impact Number (18 inches)	1.6	2.0	2.0	2.3	2.7
Hardened Pellet Characteristics (300 psig/1 hour)					
Compressive Strength, lb	104.0	46.2	45.4	84.0	23.2
Impact Number (18 inches)	>25	16.6	27.6	>30	14.8
Pellet Density, g/cm ³					
	2.38	1.03	1.00	2.56	0.94
Tumble Index					
Head Sample, g	500	500	501	503	462
After 200 Revolutions, %					
+3 Mesh	97.19	92.45	92.81	98.61	68.97
+14 Mesh	0.11	0.15	0.33	0.02	0.33
-14 Mesh	2.70	7.40	6.68	1.37	30.70
Reduction Weight Loss, % (2500°F)					
5 Minutes	36.91			36.99	
15 Minutes	38.02			37.76	
30 Minutes	38.53			37.91	
Reduction Weight Loss, % (2700°F)					
5 Minutes	36.63			37.80	
15 Minutes	37.77			37.90	
30 Minutes	37.44			38.20	

* Char No. M149-190 used for Tests 22-24. 78% M149-190/22% M149 tops used for Tests 25 and 26.

TABLE 11
RESULTS OF PELLET TECHNOLOGY CORPORATION CHAR PELLETIZING TESTS
USING INDIANA CHAR

	<u>Test No. 28</u>	<u>Test No. 29</u>	<u>Test No. 30</u>	<u>Test No. 31</u>
Pellet Composition, %				
Empire Iron Ore Concentrate	75	--	--	--
Na ₂ CO ₃	--	--	--	0.5
Coal Char*	18	92	92	0.5
Burned Lime Fines	5	8	6	6.0
Silica Sand Fines	2	1	2	2.0
Pellet Diameter, inches				
	0.59	0.75	0.75	0.75
Green Pellet Characteristics				
Moisture, weight %	11.33	25.27	25.24	26.69
Compressive Strength, lb	7.70	11.90	13.60	13.00
Impact Number (18 inches)	6.36	>25	>25	>25
Dried Pellet Characteristics				
Compressive Strength, lb	5.3	6.9	6.4	5.71
Impact Number (18 inches)	1.0	2.0	2.0	2.00
Hardened Pellet Characteristics (300 psig/1 hour)				
Compressive Strength, lb	146.4	18.4	20.3	13.2
Impact Number (18 inches)	>25	12.6	5.2	5.5
Pellet Density, g/cm ³				
	2.49	1.16	1.32	1.09
Tumble Index				
Head Sample, g	504	503	500	500
After 200 Revolutions, %				
+3 Mesh	98.04	82.95	68.00	66.40
+14 Mesh	0.19	0.73	2.80	22.60
-14 Mesh	1.77	16.21	29.20	11.00
Reduction Weight Loss, % (2500°F)				
5 Minutes	34.71			
15 Minutes	35.20			
30 Minutes	36.72			
Reduction Weight Loss, % (2700°F)				
5 Minutes	35.25			
15 Minutes	36.22			
30 Minutes	35.90			

* Char No. P007 used for Test 28. Char No. P008 used for Tests 29-31.

TABLE 12
 PERMANENT ROLL MAGNETIC SEPARATION OF INDIANA NO. 3 COAL

Treatment	Fraction	Weight Recovery, % (Dry Basis)	Analysis, % (Dry Basis)					
			Ash	Volatile	S (Total)	S (Pyritic)	S (SO ₂) (Organic)	
None (Feed Coal, 14 x 50-mesh)	Nonmagnetics	85.1	9.66	38.86	4.03	0.91	0.79	2.33
	Magnetics	14.9	19.13	33.21	3.53	0.87	1.41	1.25
Total		100.0	11.07	38.02	3.96	0.91	0.89	2.17
None (Feed Coal, 50 x 100-mesh)	Nonmagnetics	31.7	15.09	35.82	4.20	0.74	1.09	2.37
	Magnetics	68.3	19.28	33.17	3.71	0.63	1.55	1.54
Total		100.0	17.95	34.01	3.87	0.66	1.40	1.80

TABLE 13
 PERMANENT ROLL MAGNETIC SEPARATION OF INDIANA NO. 3 COAL CHAR

Treatment	Fraction	Weight Recovery, % (Dry Basis)	Analysis, % (Dry Basis)					
			Ash	Volatile	S (Total)	S (Pyritic)	S (SO ₂)	S (Organic)
1020°F (550°C) From Chinook Feed Coal	Nonmagnetics	22.8	13.87	12.94	1.98	0.49	0.08	1.41
	Magnetics	77.2	17.29	12.43	2.99	0.12	0.14	2.73
Total		100.0	16.51	12.55	2.76	0.20	0.13	2.43
1020°F (550°C) From non-magnetic Chinook Coal	Nonmagnetics	32.2	14.09	10.14	2.76	0.11	0.06	2.58
	Magnetics	67.8	14.25	10.39	3.07	0.15	0.08	2.84
Total		100.0	14.20	10.31	2.97	0.14	0.07	2.76

The nonmagnetic fraction of the 1020°F (550°C) char produced from the nonmagnetic feed fraction had a similar ash content but a significantly greater sulfur content compared to the nonmagnetic fraction of the char produced from the as-received feed coal. These results indicate that precleaning the Chinook coal by magnetic separation does not provide any advantage in terms of product sulfur concentration, although some reduction in ash concentration is observed. Use of the lower sulfur magnetic fraction from the as-received Chinook coal as a mild gasification feed would provide only limited overall sulfur reduction, with a concurrent significant increase in product ash content.

4.2 Product Char Characterization

A sample of Indiana No. 3 coal char produced in the UNDEERC 100-lb/hr PDU (P007, 8-9-90) was dry-screened, and the resulting size fractions were analyzed. Table 14 shows that, in general, ash content decreased and volatile matter increased as functions of decreasing particle size. Sulfur content appeared to decrease with decreasing particle size. However, this effect was not as extensive in samples treated by magnetic separation, as discussed in the following section. Methanation tests will also be performed on this char sample.

4.3 Magnetic Separation of EERC PDU Indiana No. 3 Char

The effect of cleaning Indiana No. 3 coal char by magnetic separation was investigated. The P007 char was divided into different size fractions and subjected to permanent roll magnetic separation tests. Table 15 summarizes the results. The char fed to the magnetic separation process contained 3.2% total sulfur and 19.9% ash on a dry basis. The sulfur analyses in Table 15 do not confirm the marked reduction in sulfur content as a function of decreasing particle size, which was shown in Table 14.

Magnetic separation was effective in cleaning all of the size fractions tested. No attempt was made to magnetically separate the small amount of 100-mesh material. The nonmagnetic portion of the 14 X 10-mesh char contained less than 1% sulfur. However, this cleaned fraction constituted only about 5% of the total char. All other individual, cleaned fractions contained at least 2% sulfur. Magnetic fractions contained up to 4.3% sulfur.

Ash separations were most effective when performed on the naturally occurring 14 X 100-mesh fraction of the char. The nonmagnetic portion of the 14 X 100-mesh char contained 13.8% ash. Little or no ash separation occurred during magnetic separation of the coarser fractions. Results obtained on the 14 X 100-mesh fraction suggest that improved separation could be obtained if the coarser material were crushed to pass 14 mesh prior to magnetic separation.

Also shown in Table 15 are the combined analyses of products from magnetic separation of the +100-mesh fractions. Nearly one-third of the total char was collected as nonmagnetic material containing about 2.2% total sulfur and 19.3% ash. Nearly 50% of the total char was recovered in the combined

nonmagnetic-plus-middling fractions with a combined analysis of about 2.4% sulfur and 19.2% ash. The magnetic char, which constitutes the remaining char exclusive of the -100-mesh fraction, contained about 4.0% sulfur and 20.6% ash.

4.4 Gravity Separation of Indiana No. 3 Char

Tests to determine the suitability of the P007 char for gravity separation are in progress. These tests are being performed using heavy liquids. A test column for pneumatic separation of the product char has been set up to size and separate the char by elutriation.

4.5 Methanation Testing of Wyodak Char

Samples of Wyodak char produced in the UNDEERC 4-lb/hr CFBR were subjected to methanation tests to determine the potential suitability of the char as a feedstock for production of pure carbon. Table 16 summarizes the analyses of the raw char and the spent chars resulting from the methanation reaction with hydrogen at 300 psig in a bench-scale reactor at AMAX R&D. The

TABLE 14
ANALYSIS OF INDIANA NO. 3 CHAR (P007, 8/9/90)

Analysis, % (As-Received)								
Size Fraction	Weight	H ₂ O	Ash	Volatile Matter	C	H	N	S
>4 Mesh	5.8	1.72	20.41	11.71	68.68	2.21	0.71	2.40
4 X 6 Mesh	7.5	2.10	20.65	14.58	67.08	2.38	0.69	2.34
6 X 8 Mesh	10.0	2.14	20.12	12.65	68.84	2.31	0.73	2.73
8 X 12 Mesh	17.2	1.90	21.65	14.08	67.87	2.23	0.71	3.16
12 X 16 Mesh	27.0	2.07	19.59	13.55	69.77	2.28	0.72	2.48
16 X 20 Mesh	18.2	2.12	19.31	13.71	68.91	2.35	0.73	2.44
20 X 30 Mesh	6.1	2.15	20.07	14.68	68.33	2.35	0.66	2.28
30 X 40 Mesh	2.8	2.40	18.31	15.66	59.94	2.48	0.66	2.10
40 X 50 Mesh	1.8	2.68	16.21	16.15	71.27	2.56	0.63	1.54
50 X 70 Mesh	1.7	2.69	14.69	16.39	74.33	2.43	0.57	1.12
<70 Mesh	1.9	2.75	15.99	16.62	72.08	2.40	0.57	1.22
Head	100.0	1.68	19.41	14.32	70.00	2.40	0.81	2.50
Calculated Head	100.0	2.09	19.85	13.83	68.94	2.31	0.71	2.51

TABLE 15

PERMANENT ROLL MAGNETIC SEPARATION OF INDIANA NO.3 CHAR (P007 Char, 8/9/90)

Size Fraction	Magnetic Fraction	Moisture, %	Weight Recovery, % (Dry Basis)		Analysis, % (Dry Basis)						
			Of Size Fraction	Of Total Char	Ash	Volatile	S (Total)	S (Pyritic)	S (SO ₂)	S (Organic)	
>1/4 inch Char	Nonmagnetics	1.47	69.2	1.2	14.39	12.17	2.56	0.21	0.06	2.28	
	Middlings	1.71	8.4	0.1	14.18	13.26	3.37	0.67	0.08	2.62	
	Magnetics	1.53	22.3	0.4	16.49	13.34	4.29	1.09	0.10	3.10	
	Total	1.57	100.0	1.7	14.84	12.52	3.01	0.45	0.07	2.50	
1/4 inch X 14 Mesh Char	Nonmagnetics	1.11	35.2	27.1	20.47	12.47	2.37	0.20	0.08	2.08	
	Middlings	1.12	16.7	12.8	19.34	12.67	3.00	0.32	0.15	2.53	
	Magnetics	1.09	48.1	36.9	20.72	12.80	4.05	0.58	0.18	3.29	
	Total	1.10	100.0	76.8	20.40	12.66	3.28	0.41	0.14	2.74	
14 X 100 Mesh Char	Nonmagnetics	2.10	22.4	4.7	13.83	15.16	0.94	0.07	0.05	0.82	
	Middlings	1.68	13.7	2.9	17.57	12.98	2.49	0.16	0.13	2.20	
	Magnetics	1.38	63.9	13.5	20.21	14.08	3.67	0.63	0.23	2.81	
	Total	1.56	100.0	21.0	18.42	14.17	2.90	0.44	0.18	2.28	
<100 Mesh Char	Total	1.47	100.0	0.5	19.59	17.70	2.94	0.78	0.28	1.88	
	Total Char	1.21	100.0	100.0	19.89	13.00	3.20	0.42	0.15	2.63	
	Nonmagnetics Middlings	1.26	33.1	32.9	19.31	12.84	2.17	0.18	0.08	1.91	
>100 Mesh Char	Middlings	1.23	15.9	15.8	18.97	12.73	2.91	0.30	0.14	2.47	
	Nonmagnetics +Middlings	1.25	49.0	48.7	19.20	12.81	2.41	0.22	0.10	2.09	
	Magnetics	1.17	51.0	50.8	20.55	13.14	3.95	0.60	0.19	3.16	
	Total	1.21	100.0	99.5	19.89	12.98	3.20	0.41	0.15	2.64	

TABLE 16
CHARACTERIZATION OF UNDEERC CHAR

	UNDEERC <u>Char</u>	1380°F (750°C) <u>Spent Char</u>	1470°F (800°C) <u>Spent Char</u>
Proximate Analysis (wt%)			
Moisture	3.6	2.2	2.2
Volatile	14.7	11.1	7.1
Fixed Carbon	75.2	70.8	70.3
Ash	10.1	18.2	22.7
Ultimate Analysis (wt%)			
Carbon	78.0	78.2	74.7
Hydrogen	2.1	1.3	0.9
Sulfur	0.5	0.7	0.6
Nitrogen	0.6	0.3	0.3
Oxygen (BD)	8.7	1.3	0.8
Surface Area, m ² /g	6.0	600.0	690.0

conversion (weight loss) for the Wyodak char during methanation was 45% at 1380°F (750°C) and 55% at 1470°F (800°C). The spent chars exhibited reasonable surface areas for activated carbon production. A sample of Indiana No. 3 char from Test P007 is being evaluated in the bench-scale methanation unit at AMAX R&D.

5.0 FUTURE WORK

Parametric testing with Wyodak and Indiana coals will start in September and will be followed by product end testing and process evaluation in the first half of 1991. Two subtasks were added to Task 4 by DOE: Costing of a pilot plant and costing of the PDU product and equipment disposal. These subtasks will be completed by the second quarter of 1991.

END

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