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

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by

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

1.0 INTRODUCTION

The current objective of the University of North Dakota Energy and Environmental Research Center (EERC) mild gasification project is to optimize reactive char and marketable liquids production on a 100-lb/hr scale using Wyodak subbituminous and Indiana No. 3 bituminous coals. Tests performed using the EERC 100-lb/hr process development unit (PDU) include a refractory-cure (Test P001), a test using petroleum coke (Test P002), and tests using Wyodak and Indiana coals. The reactor system used for the 11 PDU tests conducted to date consists of a spouted, fluid-bed carbonizer equipped with an on-line condensation train that yields three boiling point fractions of coal liquids ranging in volatility from about (77°-750°F) 25°-400°C. The September-December 1990 quarterly report described reaction conditions and the bulk of the analytical results for Tests P010 and P011. This report describes further P010 and P011 analytical work, including the generation of simulated distillation curves for liquid samples on the basis of sulfur content, using gas chromatography coupled with atomic emission detection (GC/AED) analysis.

Conditions of Test P010 (Wyodak coal) include a reactor temperature of 1100°F (590°C), reactor pressure of 14.7 psi, residence time of 30 minutes, and a fluidization gas mixture comprised of the products of natural gas combustion with 80% excess air. Following an 8-hour heat-up period, continuous coal feed was maintained for about 30 hours. Conditions of Test P011 (Indiana No. 3 coal) were similar to those of P010, except that the fluidization gas was comprised of the products of natural gas combustion with stoichiometric amounts of air. Test P011 was terminated ahead of schedule due to the loss of recycle tar coolant in the tar scrubber.

During Test P010, the tar venturi scrubber was used to remove particulates remaining in the gas stream (that were not removed by the cyclones) and condense boiling point fractions of liquid products ranging in temperature from 350° to 700°F (175° to 380°C) using recycled product liquor. Further cooling occurred in the sieve tower, again using recycled product liquor. The sieve tower exit temperature was just above the dew point of the product gas-- approximately 160° to 180°F (70° to 80°C). The product gas then passed through a water scrubber, which cooled the gas stream to 80° to 100°F (27° to 38°C), and a demister to ensure that organic material did not escape and pass through the flare system. One-quart samples of condensables were obtained from the tar scrubber, sieve tower, and water scrubber at roughly 5-hour intervals during the "balance period" portion of Test P010. The balance period is the portion of the test following system heat-up and stabilization during which, ideally, coal feed rate and char and liquid collection rates are constant, and steady-state conditions exist in the reactor system. The analysis of samples collected during a balance period provides the data needed for mass balance calculations.

2.0 GC/AED ANALYSIS OF TEST P010 (WYODAK) LIQUIDS

The use of GC/AED enables identifying and quantitating elements in compounds as the compounds elute from a GC column. When a compound leaves the

GC column and enters the atomic emission detector, electrons in the atoms that make up the compound are energized by a microwave-induced plasma and excited to higher energy levels. When the electrons return to their stable state, they emit light, which passes into a spectrophotometer. The light is separated by a diffraction grating into wavelengths characteristic of the element(s) selected for analysis and transmitted to a photodiode array detector, which can be tuned to monitor a specific range of wavelengths, depending on the element(s) of interest. To quantitate a specific element--sulfur, for example--the photodiode array is tuned to monitor a wavelength characteristic of energy emission from sulfur atoms. When energy of this wavelength is detected, the energy is converted into an electrical signal, the intensity of which is proportional to a specific quantity of sulfur. By calibrating the atomic emission detector response with standards of known concentration, sulfur concentration in unknown materials can be determined. It should be noted that when the atomic emission detector is monitoring wavelengths characteristic of energy emission from sulfur, it is essentially acting as a sulfur detector, not as a sulfur compound detector. However, when combined with GC/mass spectrometry (GC/MS) analysis (which can provide mass spectra of sulfur species as they elute from the GC column), GC/AED analysis is very helpful in identifying sulfur-containing compounds.

The use of GC/AED for sulfur analysis enables plotting a "sulfur content simulated distillation curve" for the chromatographable portion of a condensables sample. The chromatographable portion of a liquid sample normally corresponds to the volatile portion of the sample. Sulfur content simulated distillation curves can be plotted using GC/AED data, just as simulated distillation curves are plotted using GC/flame ionization detection (GC/FID) data, the only difference being in how the data is collected--AED instead of FID. An explanation of the GC/FID simulated distillation technique was provided in the October-December 1990 quarterly progress report, along with a comparison of simulated distillation with American Society for Testing and Materials (ASTM) D1160 vacuum distillation. Whereas a GC/FID simulated distillation curve describes the relationship of overall sample volatility with increasing temperature, a sulfur content simulated distillation curve describes the relationship of the sample's sulfur content volatility with increasing temperature. A sample's GC/FID simulated distillation curve may or may not resemble its sulfur content simulated distillation curve, depending on the type and distribution of sulfur species in the sample.

During the P010 balance period, three condensables samples were collected from each of the three condensation unit operations for a total of nine samples. Table 1 shows the sulfur content of each condensables sample, determined using GC/AED analysis. Also shown in Table 1 is the GC/AED-determined sulfur content of Diesel #2 and Mandan decant oil, which were used as start-up fluids in the sieve tower and tar scrubber, respectively. In addition to monitoring for sulfur, GC/AED analysis was used to monitor the samples for nitrogen, oxygen, carbon, and hydrogen. Nitrogen was not detected in any of the samples, and oxygen was found in two of the water scrubber samples in quantities insufficient for generation of oxygen content simulated distillation curves. (Greater concentrations of oxygen were found in the water phase of the water scrubber samples--see October-December report.) GC/AED carbon content simulated distillation curves are, ideally, similar to GC/FID simulated distillation curves, since a flame ionization detector is basically a "carbon counter." GC/AED hydrogen content simulated distillation curves are displayed in some of the figures in this report.

TABLE 1

GC/AED SULFUR CONTENTS OF CONDENSABLES

	Sulfur Content (wt%, mf)		
	Sample 1	Sample 2	Sample 3
Tar Scrubber	0.6	0.5	0.3
Sieve Tower	0.8	0.7	0.5
Water Scrubber[1]	0.5	0.5	0.3
Diesel #2	0.5	--	--
Mandan Decant Oil	2.1	--	--

[1] Samples from the water scrubber were recovered as two separate phases: an organic phase floating on an aqueous phase. The values in Table 1 refer to the organic phase.

2.1 Tar Scrubber Condensables Analysis

Figure 1 shows the sulfur content simulated distillation of condensables samples obtained from the tar scrubber during the P010 balance period, along with data for the Mandan decant oil used as start-up fluid in the tar scrubber. The higher distillation temperatures of the tar scrubber liquids compared to the decant oil indicate that the sulfur in these liquids is contained on heavier, less volatile compounds than the sulfur in the decant oil; this suggests the presence of a significant quantity of coal-derived material in the tar scrubber liquids. Figure 2 shows the overall sample simulated distillation curve, the sulfur content simulated distillation curve, and the hydrogen content simulated distillation curve (obtained using GC/AED to quantitate hydrogen in the same manner as sulfur) for Tar Scrubber Sample 1. The separation between the sulfur and FID distillation curves indicates that a greater percentage of sulfur-containing compounds will distill at any given temperature on the sulfur distillation curve (up to about 850°F, at which point the two curves begin to converge) than will nonsulfur-containing compounds; this suggests the possibility of preferentially removing sulfur from the tar scrubber liquids by distillation.

2.2 Sieve Tower Condensables Analysis

Figure 3 shows the sulfur content simulated distillation of sieve tower condensables samples, along with data for the Mandan decant oil and diesel fuel used as start-up fluids for the tar scrubber and sieve tower, respectively. Figure 4, which compares the three distillation curves (FID, sulfur content and hydrogen content) for Sieve Tower Sample 1, shows that sulfur content volatility follows GC/FID-measured overall sample volatility fairly closely.

2.3 Water Scrubber Condensables Analysis

Figure 5 shows the sulfur content simulated distillation of water scrubber condensables samples, along with data for the decant oil and diesel fuel. Figure 6 compares the three distillation curves (FID, sulfur content, and hydrogen content) for Water Scrubber Sample 1 and shows that, unlike the tar scrubber and sieve tower samples in which sulfur content is more

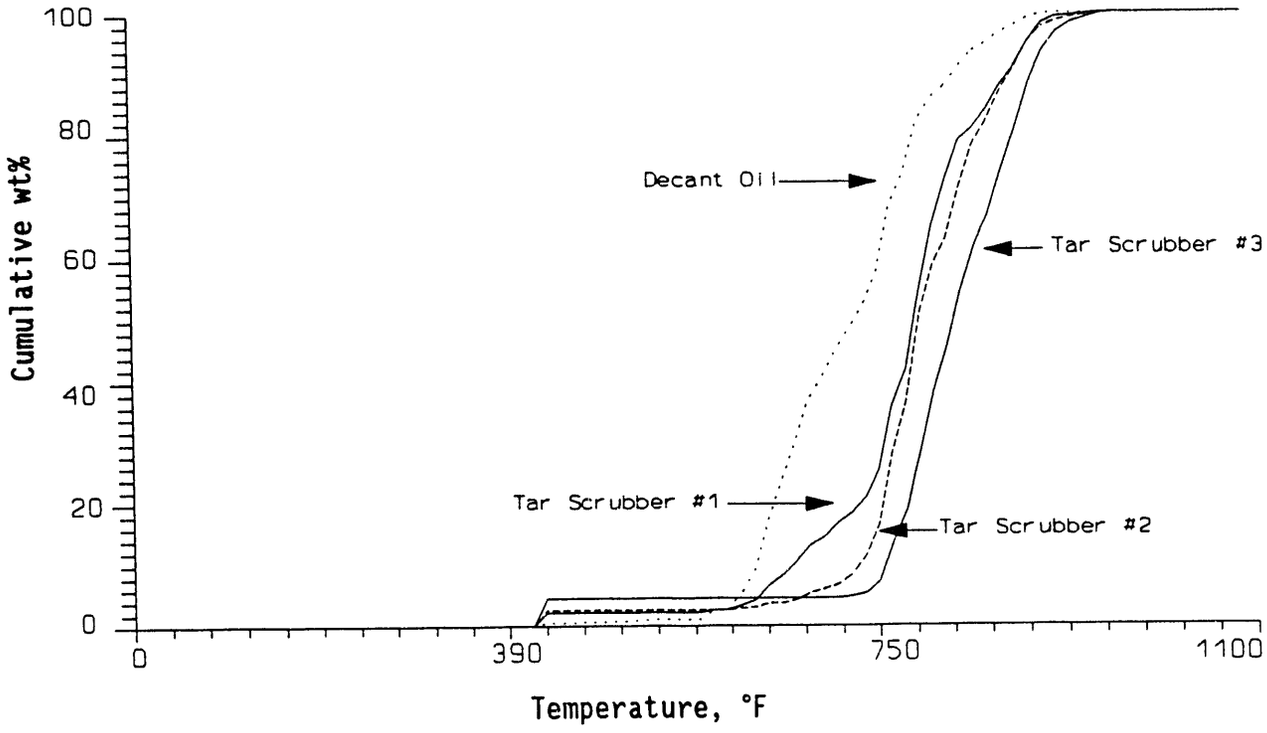

Figure 1. Sulfur content simulated distillation curves for P010 tar scrubber condensables.

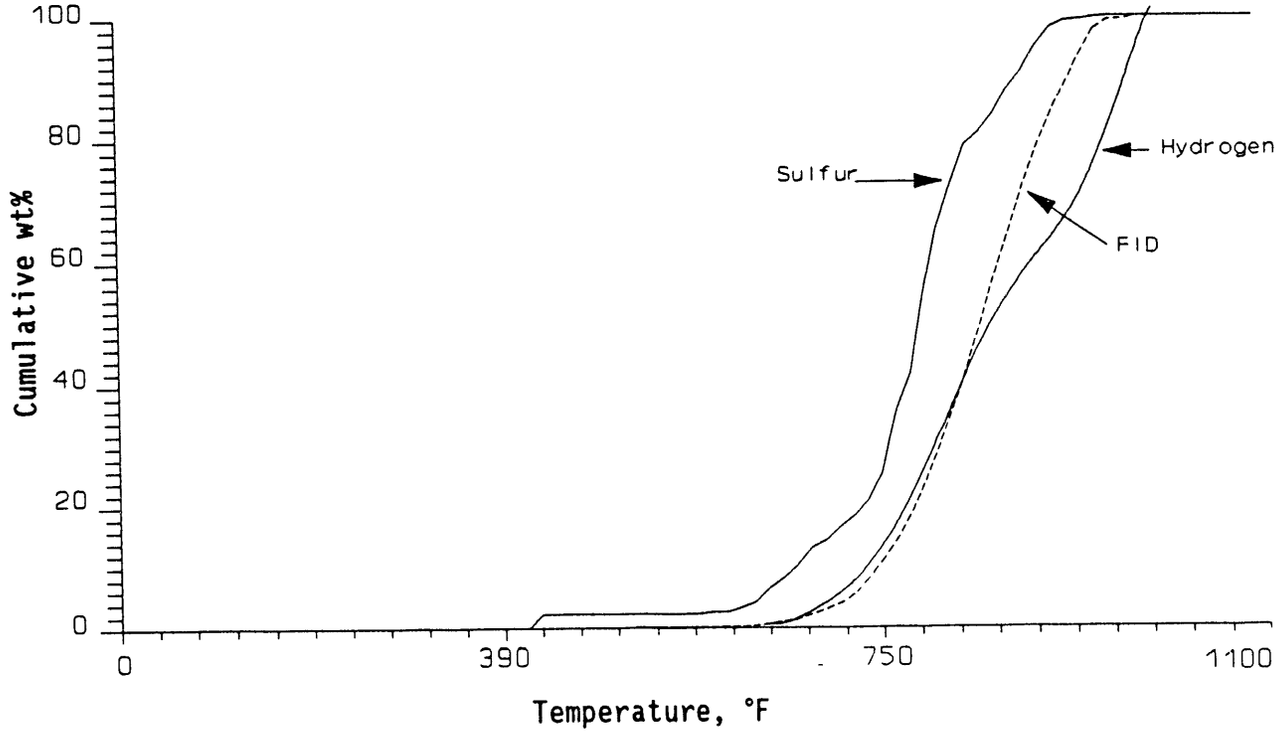

Figure 2. FID, sulfur content, and hydrogen content simulated distillation curves for P010 tar scrubber condensables--Sample 1.

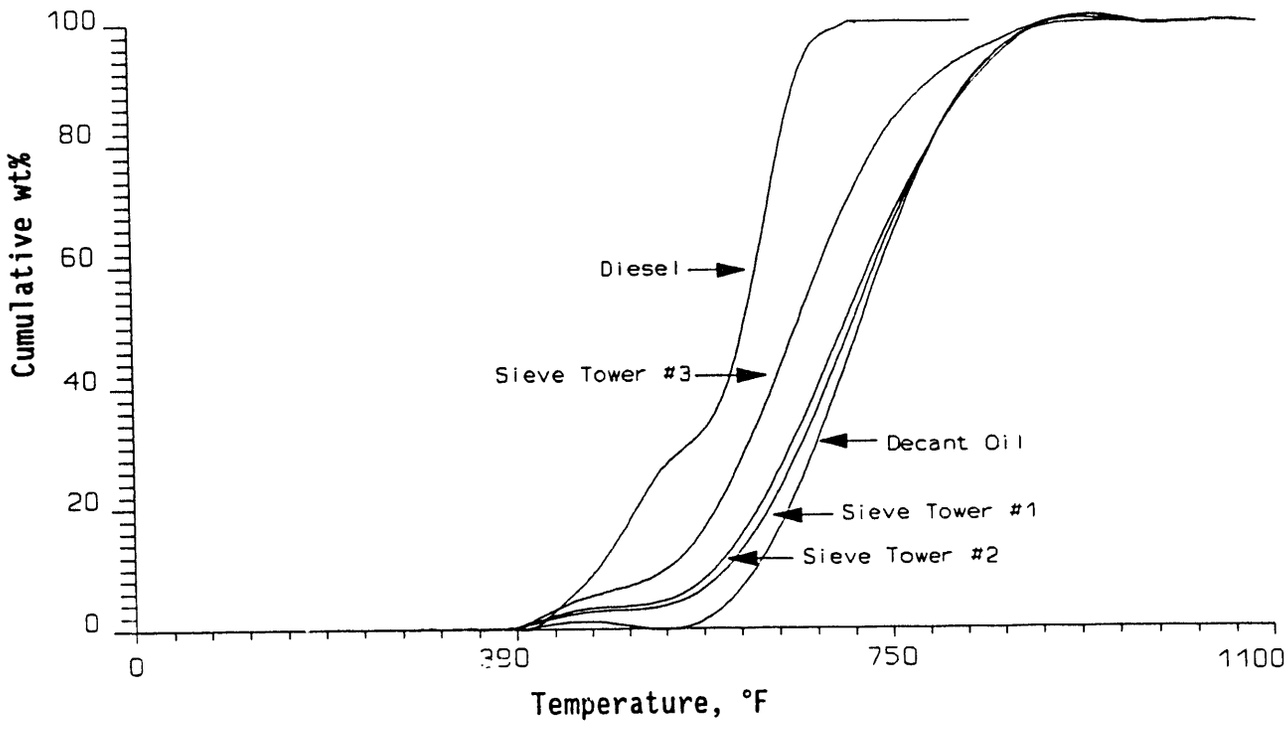

Figure 3. Sulfur content simulated distillation curves for P010 sieve tower condensables.

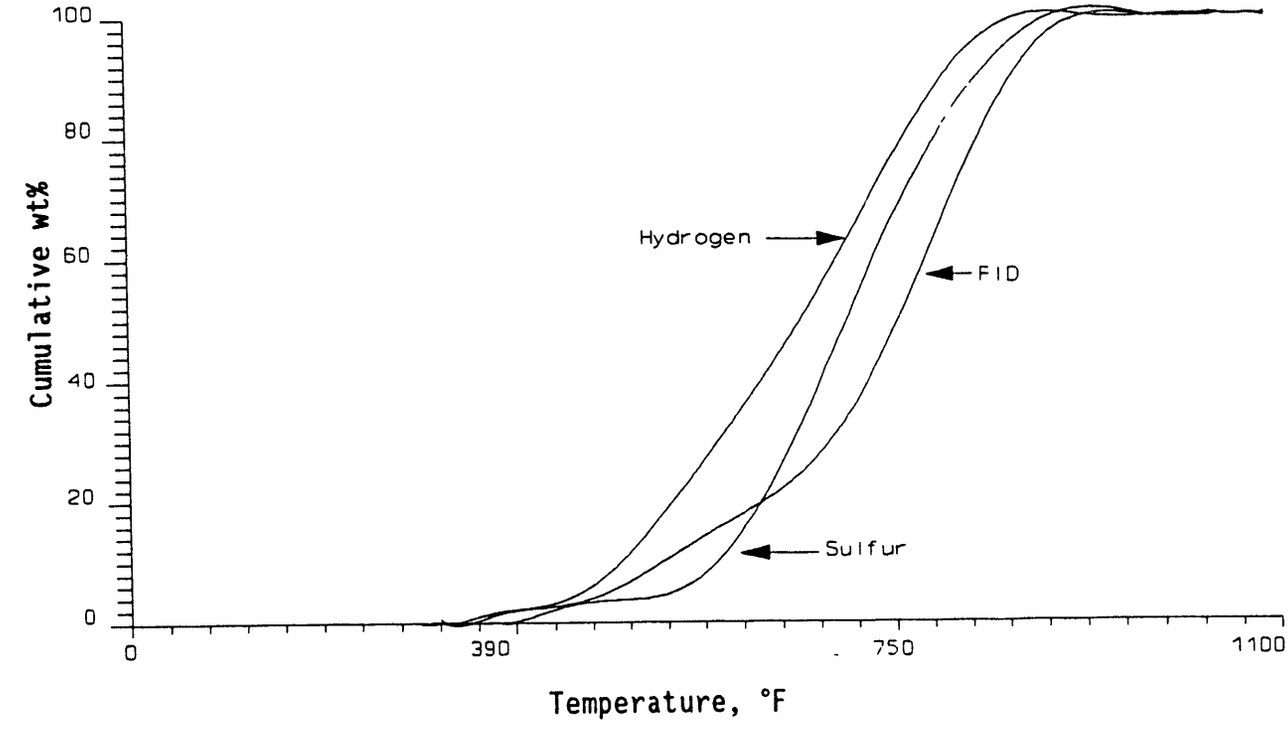

Figure 4. FID, sulfur content, and hydrogen content simulated distillation curves for P010 sieve tower condensables--Sample 1.

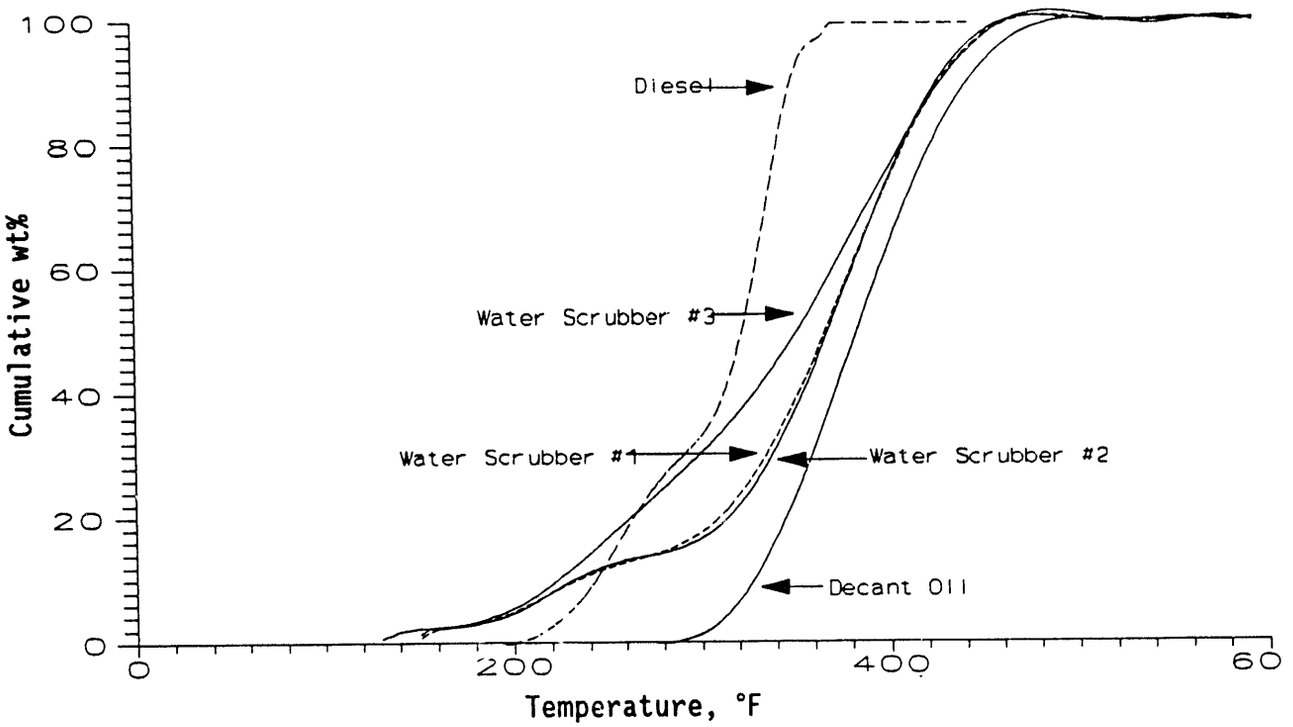

Figure 5. Sulfur content simulated distillation curves for P010 water scrubber condensables.

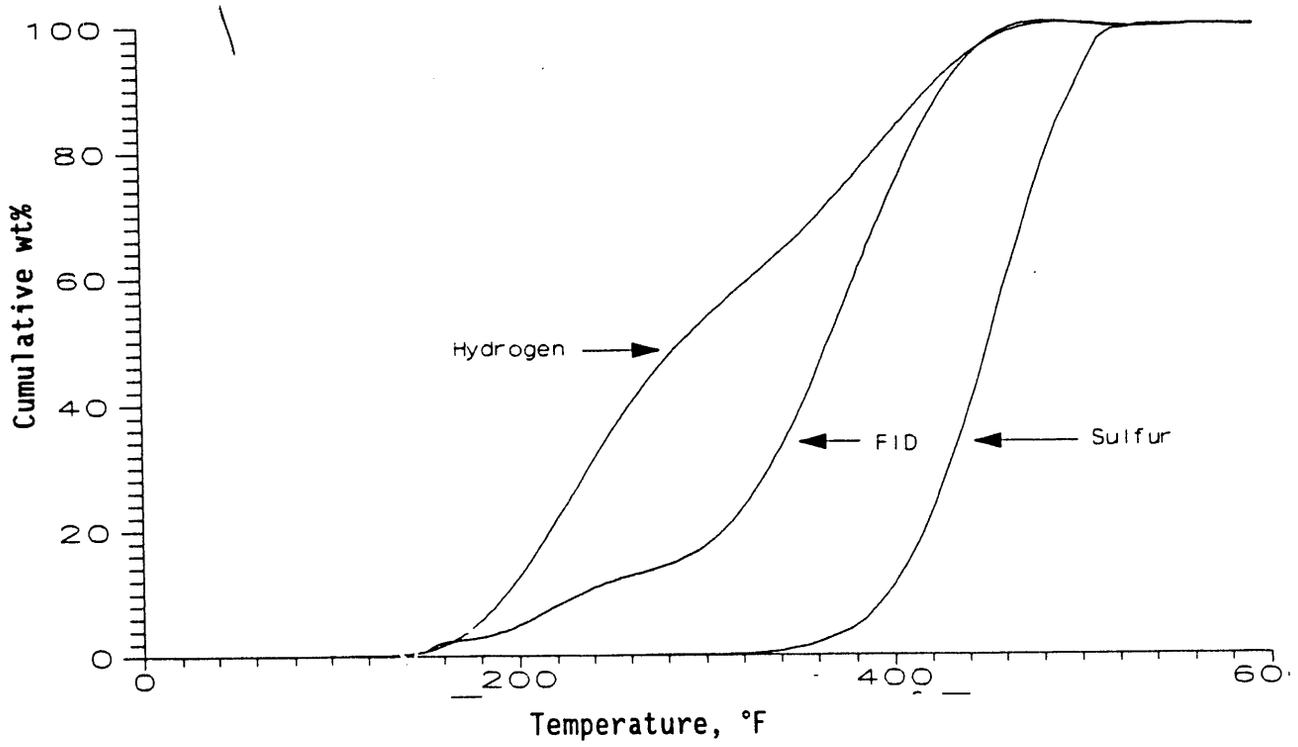

Figure 6. FID, sulfur content, and hydrogen content simulated distillation curves for P010 water scrubber condensables--Sample 1.

concentrated in the lighter, more volatile fraction of the liquids, the sulfur content in the water scrubber sample is significantly more concentrated in the heavier, less volatile fraction of the liquid.

2.4 Sulfur Content Volatility Changes with Time

Figure 7 shows sulfur content simulated distillation curves for the first samples obtained from the three condensation unit operations (tar scrubber, sieve tower, and water scrubber). Figures 8 and 9 show curves obtained from each of the unit operations following Sample Periods 2 and 3, respectively. Comparison of the three figures shows that the sulfur content volatility of the sieve tower and water scrubber liquids closely follows that of the decant oil, especially for Samples 1 and 2.

3.0 GC/AED ANALYSIS OF TEST P011 (INDIANA #3) LIQUIDS

Condensables samples collected during Test P011, which was terminated ahead of schedule because of recycle coolant loss in the tar scrubber, included a hard, glassy tar from the tar scrubber and a three-phase liquid mixture from the water scrubber. The water scrubber sample was comprised of a top oil layer, a middle aqueous layer, and a bottom tar layer. Figure 10 is a comparison of the sulfur content distillation curves for the three samples and the two start-up fluids. As with Test P010 (performed with Wyodak subbituminous coal), the Indiana (bituminous) tar scrubber liquids (labeled "glassy tar" in the figure) contain heavier, less volatile sulfur compounds than the decant oil; this indicates the presence of sulfur compounds from coal in the glassy tar. Comparison of the water scrubber oil curve with that of the diesel fuel indicates the presence of a significant quantity of coal-derived light, volatile sulfur compounds in the water scrubber oil. Also, the presence of heavy coal-derived material in the Indiana coal liquids is suggested by inspection of the hydrogen content simulated distillation curves in Figure 11, which show that the tar scrubber tar (labeled "glassy tar" in the Figure 11) contains hydrogen on heavier compounds than those found in the petroleum-derived start-up fluids.

Figures 12 and 13 show hydrogen content, sulfur content, and FID simulated distillation curves for the water scrubber tar and tar scrubber tar, respectively. Comparison of the boiling point curves shown on the two figures illustrates the applicability of GC/AED analysis in providing a relative measurement of a material's aromaticity. In Figure 12, the increased volatility of the water scrubber tar hydrogen content, compared to its FID-measured overall volatility, suggests that this material is more aliphatic than aromatic. In Figure 13, the lower volatility of the tar scrubber tar hydrogen content, compared to its overall volatility, suggests that this material is more aromatic than aliphatic.

4.0 AMAX R&D PROJECT ACTIVITY

4.1 Feed Coal and Char Characterization

Gravity separation tests were performed on samples of P007 Indiana No. 3 (Chinook) char which were earlier subjected to dry magnetic separation. A pneumatic separation was performed in a 3-inch diameter fluidized bed using

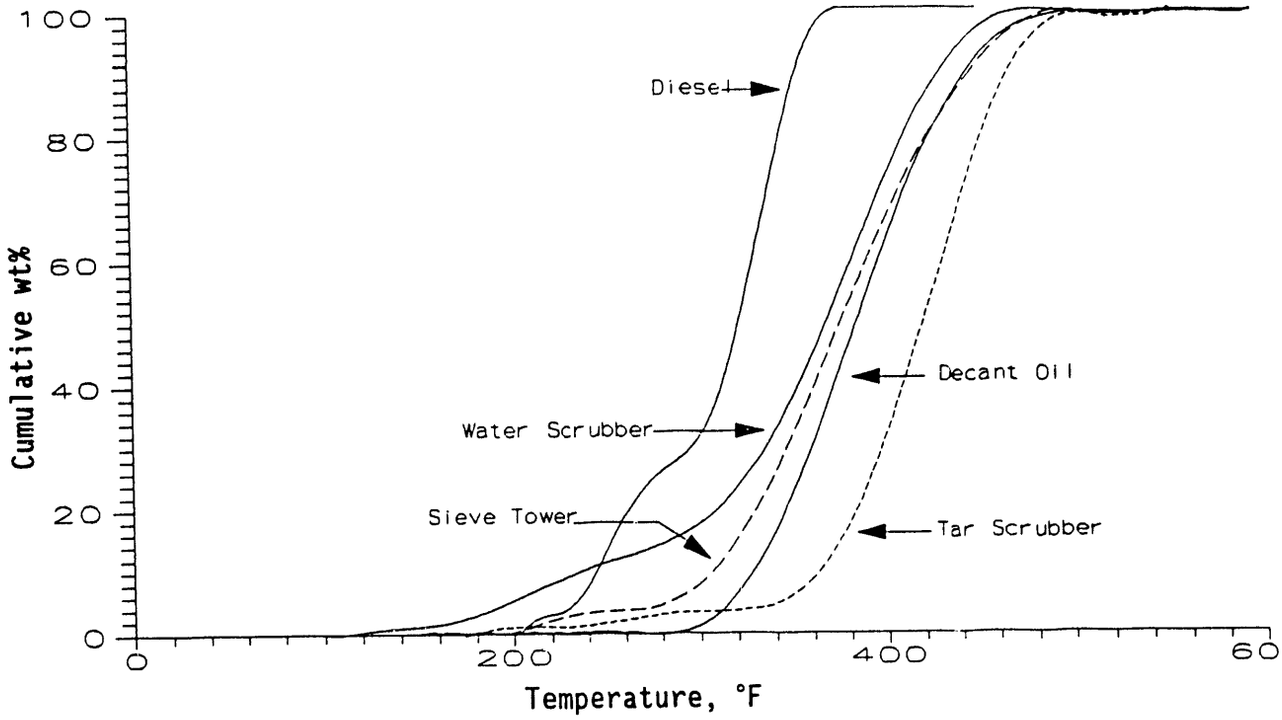

Figure 7. Sulfur content simulated distillation curves for P010 condensables from the tar scrubber, sieve tower, and water scrubber--Sample 1.

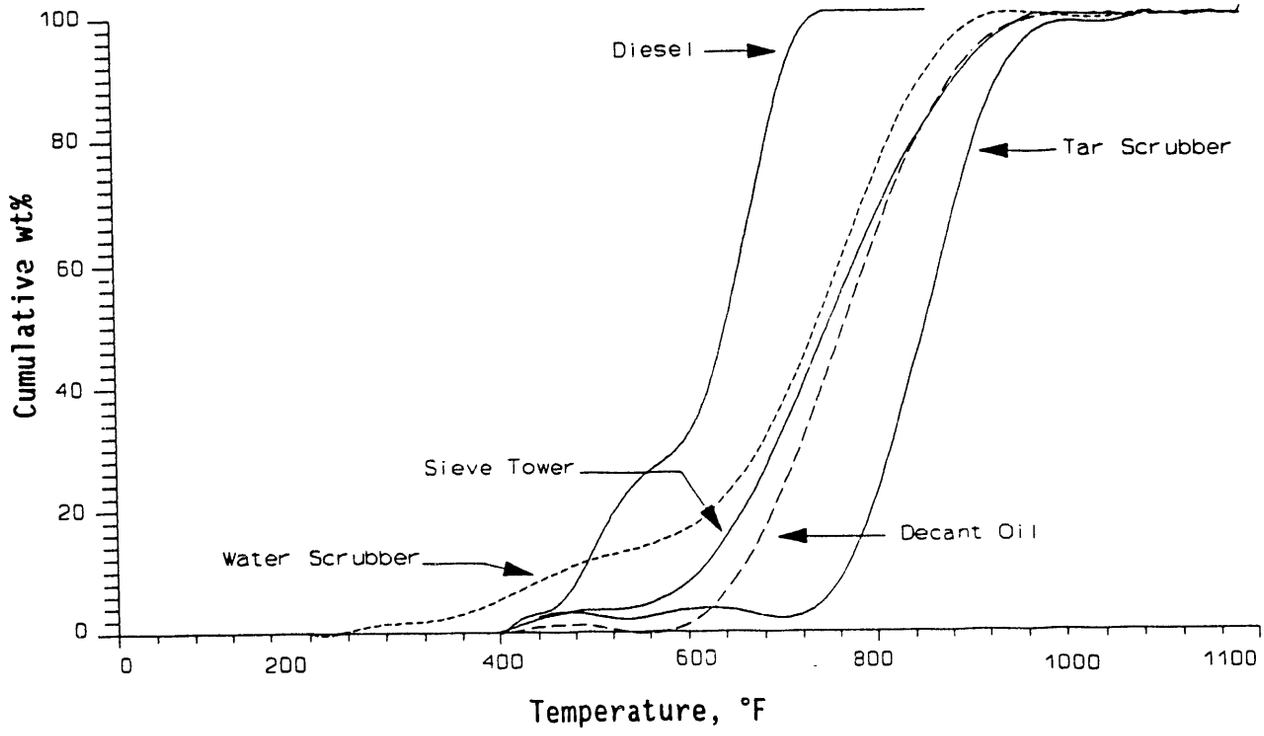

Figure 8. Sulfur content simulated distillation curves for P010 condensables from the tar scrubber, sieve tower, and water scrubber--Sample 2.

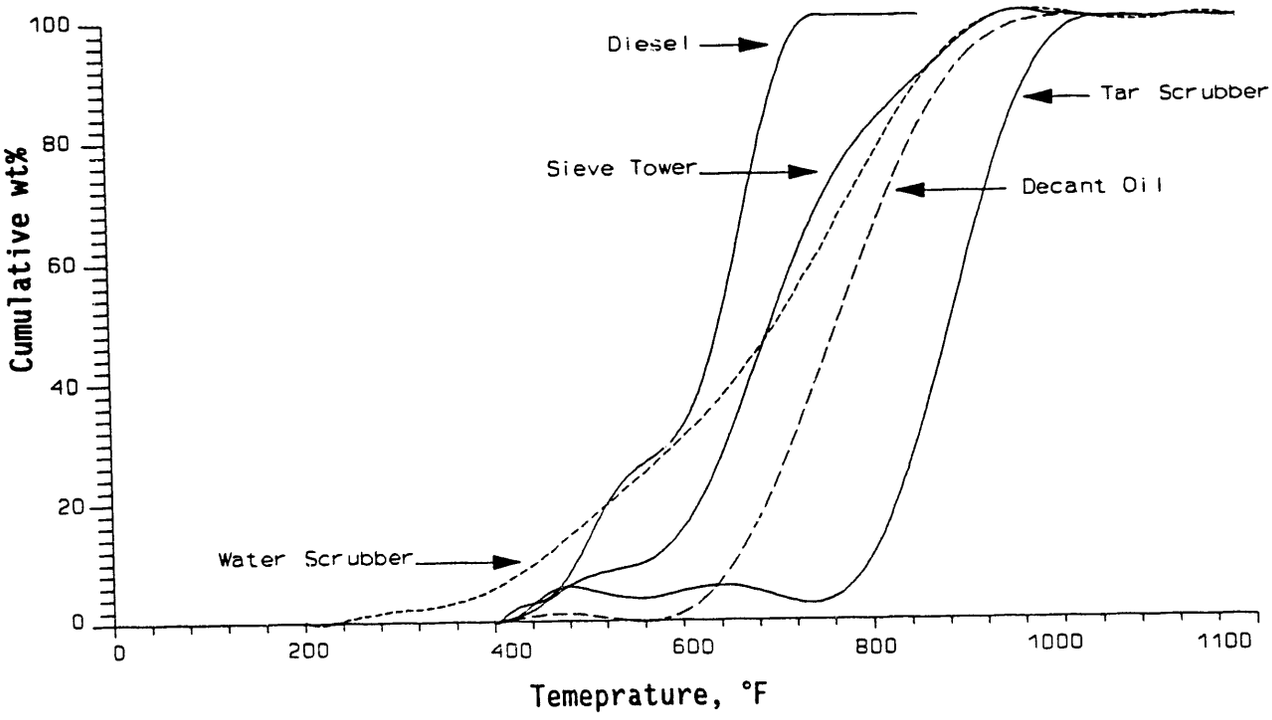

Figure 9. Sulfur content simulated distillation curves for P010 condensables from the tar scrubber, sieve tower, and water scrubber--Sample 3.

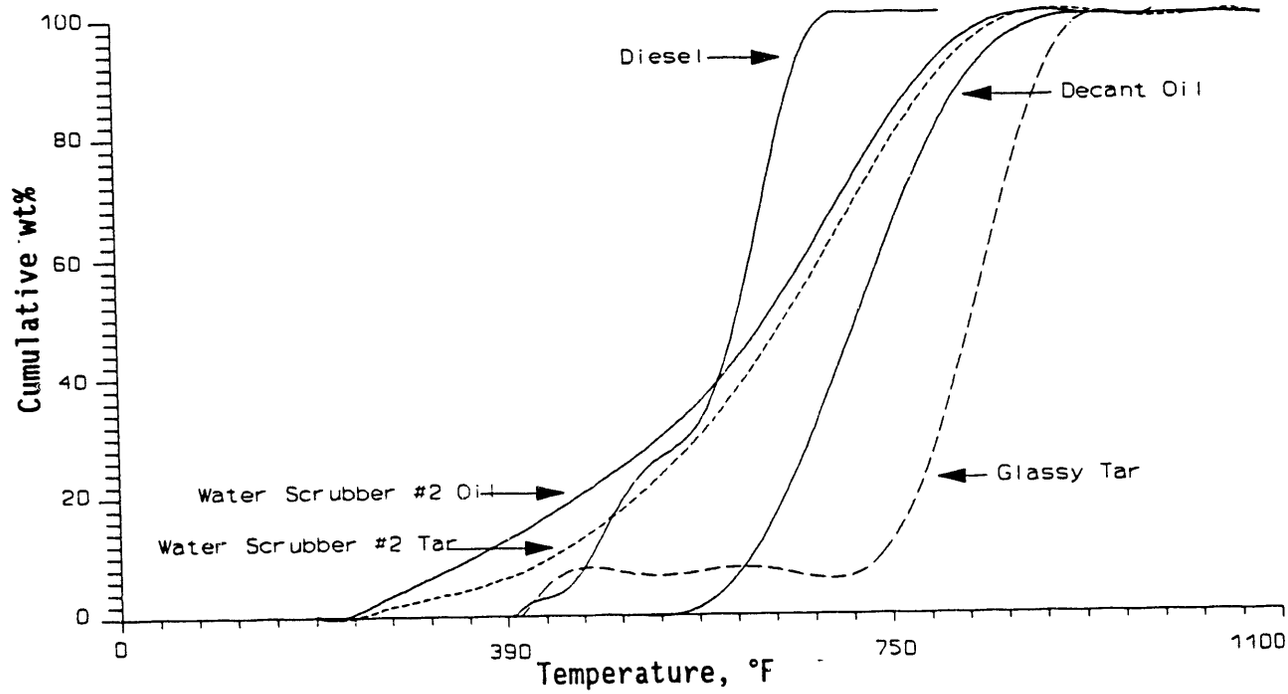

Figure 10. Sulfur content simulated distillation curves for P011 condensables: tar scrubber tar, water scrubber tar, and water scrubber oil.

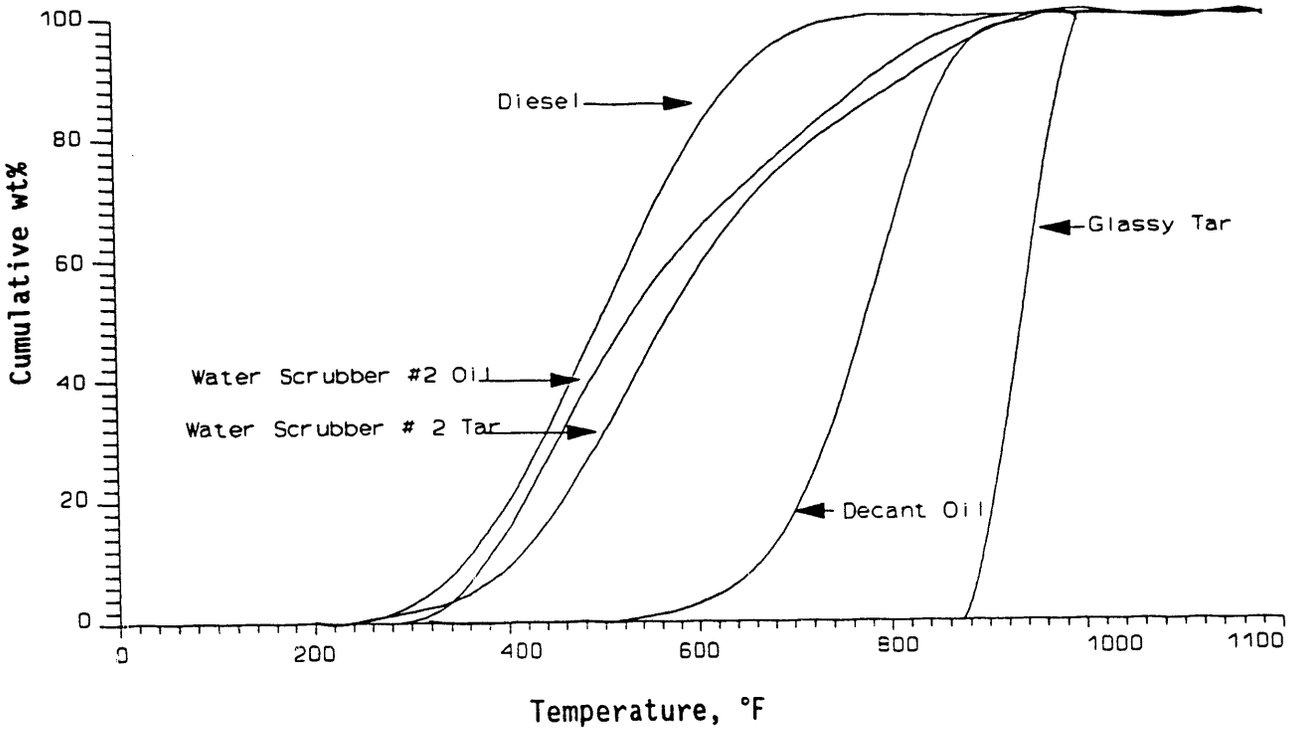

Figure 11. Hydrogen content simulated distillation curves for P011 condensables: tar scrubber tar, water scrubber tar, and water scrubber oil.

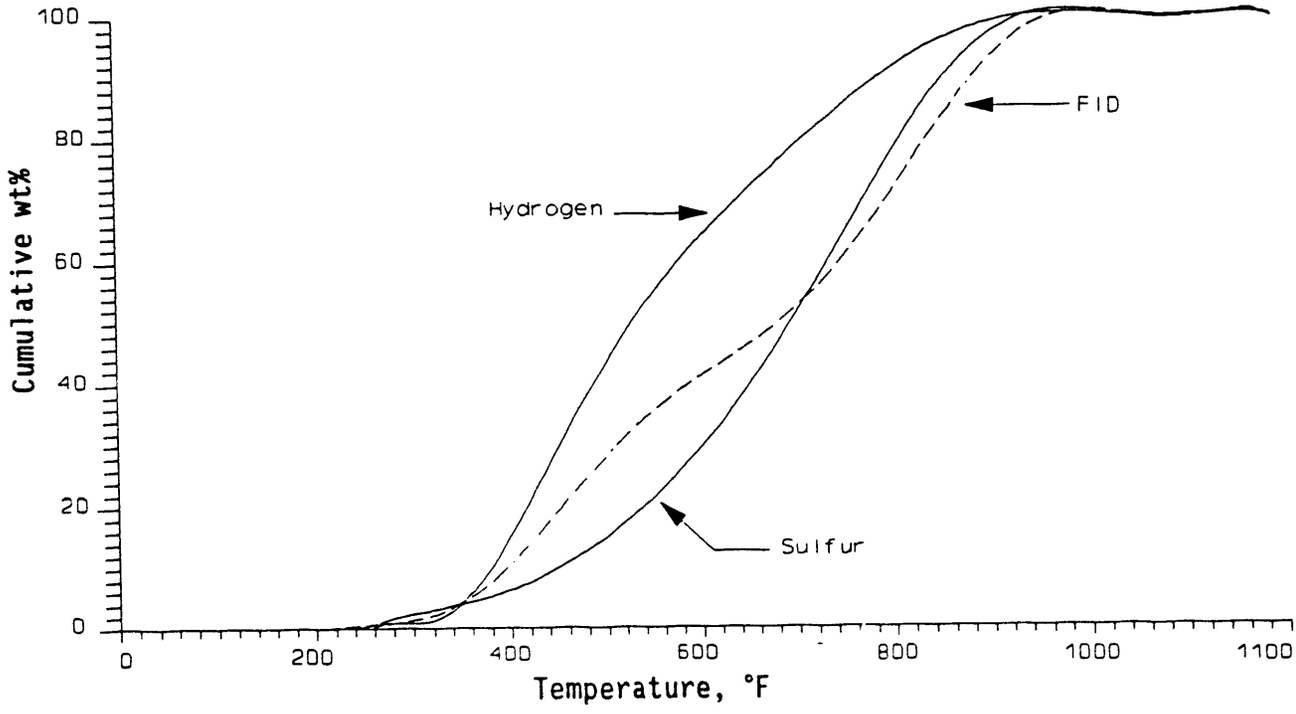

Figure 12. FID, sulfur content, and hydrogen content simulated distillation curves for P011 water scrubber tar.

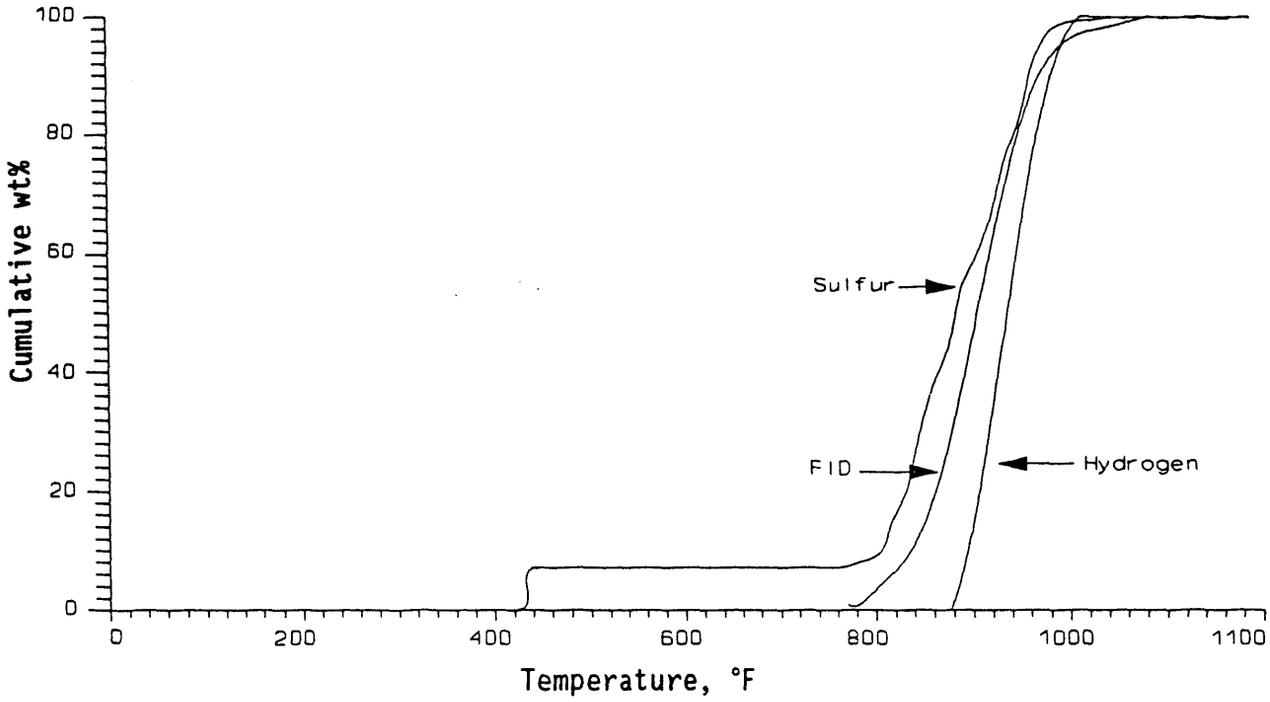

Figure 13. FID, sulfur content, and hydrogen content simulated distillation curves for P011 tar scrubber tar.

ambient air. Initial tests were performed using the 14- x 100-mesh fraction of magnetic, middling, and nonmagnetic chars.

Based on proximate analyses of the feeds and products from the fluidized-bed separation tests, reduction in ash content was achieved. The sharpest separations were made on the nonmagnetic portion of the 14- x 100-mesh char. About one-third of the nonmagnetic char was recovered as a product containing about 9% ash. The remaining two-thirds contained about 15% ash. Tests using the middling and magnetic fractions resulted in less distinct ash separations. Sulfur forms analyses are pending. Further testing with the 14- x 100-mesh char showed that about one-third of the nonmagnetic fraction could be upgraded to about 0.5% sulfur and about 9% ash. The remaining nonmagnetic char contained about 1.4% sulfur and 16% ash. These, and earlier laboratory test results, indicate that only select portions of the total material processed will be cleaned to target specifications (less than 1% sulfur and less than 10% ash). The remaining material may be suitable as a blending feedstock for metallurgical coke.

Combined fluidized-bed and magnetic separation tests are planned using the P011 Indiana No. 3 char produced at UNDEERC. The material is to be separated in the fluidized bed into different size and specific gravity ranges prior to conducting magnetic separations. Multiple stages of separation will be performed if warranted by initial test results. A similar test series is planned using the Indiana No. 3 feed coal.

Tests are continuing to evaluate upgrading flowsheets for the Indiana No. 3 (Chinook) feed coal and product chars. The current strategy is to utilize a feed coal particle top size of about 1/8 inch. This will allow for

improved liberation of impurities from the feed coal. Gravity separations performed on this material should remove a greater amount of ash minerals and pyrite, resulting in lower conversion of sulfide sulfur forms to organic sulfur during carbonization or calcining. This will also help to reduce the level of cleaning required following the carbonizing step. Since gravity separations will have been performed on the feed coal, only magnetic separations should be required after carbonizing. Upgrading of the calciner char is anticipated to include only gravity separations on the finer fractions of the char. Gravity separations performed on the feed coal should reduce or eliminate the need for any further gravity separations, except for the fines that may be produced during the fluidized-bed operations. Upgrading of the finest fraction should result in the cleanest char. The coarser fractions from the calciner should represent a somewhat lower quality.

4.2 Liquid Characterization

Samples of two tars and a sieve tower liquid were characterized during the month. The samples were produced during Runs P010 (Wyoming coal) and P011 (Indiana No. 3 coal). Results of the tar sample characterization are shown in Table 2. These samples contain a high level of pyridine insolubles. Pyridine is similar in solvent strength to quinoline, but is a little more convenient to work with. It seems likely that these pyridine insolubles are coal or char dust entrained from the mild gasification reactor. This is confirmed by analytical data on the pyridine insolubles, also reported in Table 2, which are typical of analytical results for char. Another interesting feature of the tar analyses is the relatively high-sulfur content. This may be caused by residual petroleum-derived oil which was used in the condensation system. It should be noted that the sample of tar from Run P011 was taken later in the run. Some results from similar material sampled earlier in the run and analyzed at UNDEERC indicated a lower fraction of particulates.

Future testing of these materials may involve removal of the char dust by filtration, followed by characterization of the filtrate for anode binder specifications. Some upgrading tests will also be attempted.

A light liquid was also obtained from the sieve tower in Run P010. Analytical data for this sample are shown in Table 3. The sample is hydrogen-rich, and heteroatom concentrations, except for oxygen, are low. The sample was extracted with aqueous caustic, and 4 weight percent was precipitated as cresylic acids. The cresylic acids analyzed to 17 weight percent oxygen, which is nearly identical to the oxygen content of phenol. Future testing of this material will involve examination of the extraction raffinate as diesel fuel.

5.0 XYTEL-BECHTEL INC. ACTIVITY

Under the terms of Subcontract UND 4509-0926, Xytel-Bechtel, Inc. (XBI) is to execute Subtask 4.7, which, under the terms of the primary DOE METC contract, is to provide a preliminary engineering design for a one-ton/hour mild gasification process development pilot plant (PDPP) (see Appendix A--Scope of Work).

TABLE 2

RESULTS OF CHARACTERIZATION OF UNDEERC PRU TARS (wt%)

	P010	P011
Starting Coal	Wyodak	Indiana #3
Carbon	83.3	80.2
Hydrogen	5.3	4.5
Sulfur	1.3	1.5
Nitrogen	1.1	3.4
Oxygen	6.3	4.9
Ash	6.4	8.2
Pyridine Insoluble	36	38
Carbon	74.4	68.5
Hydrogen	2.4	2.0
Sulfur	0.1	4.1
Nitrogen	2.1	2.2
Ash	19.8	21.5
Toluene Insoluble	42	52
Coking Value	53	62

TABLE 3

ANALYTICAL RESULTS FOR P010 SIEVE TOWER LIQUID (wt%)

Carbon	87.5
Hydrogen	10.3
Sulfur	0.9
Nitrogen	0.3
Oxygen	2.6
Cresylic Acids	4.0

XBI will perform the detailed work based on information provided by EERC and AMAX. EERC will have the lead responsibility for providing information on mild gasification.

5.1 Commercial Terms

The commercial terms for execution of the engineering design work were agreed to by UNDEERC and XBI.

5.2 Project Kickoff

In early March 1991, the UND progress reports for 1989 through the third quarter of 1990 were submitted to XBI for review. Messrs. Ron Gravois and Tal Angelosante represented XBI at the kickoff meeting held at EERC on March 7 and 8. It was attended by EERC representatives and conducted by Bob Ness. Also in attendance were a representative from the METC and two from AMAX.

A summary of the pilot plant activities and results obtained from the 100-lb/hr process research unit (PRU) was presented. Most of the work to date has involved the low-sulfur Wyodak (Wyoming) coal and Illinois Basin coal. More studies must be run on the high-sulfur bituminous Indiana coal for evaluation of the products and yields derived.

EERC proposed to extend the period of performance for the base contract to continue evaluating the following:

- Pelletizing clean char.
- Char-cleaning studies.
- Char-briquetting tests.
- Activated-char tests.
- Upgrading/evaluation of condensable co-products.
- Outside A&E technical/economic assessment. Bob Ness stated that this would be awarded to XBI at a later date.
- Market update. Support the market assessment by S.E. Sinor.

All participants were given a tour of the EERC facility, including the 4-lb/hr continuous fluid-bed reactor and the 100-lb/hr PRU.

5.3 Design Basis

5.3.1 Feed Coals - Indiana No. 3 and Wyodak

Indiana No. 3 will come from the Chinook Mine in Perm, Indiana. It will be washed. The Wyodak coal will come from the Belle Ayr/Eagle Butte Mine, Wyoming. The coals will be received via coal car and sized to 2" x 0. The design coal feed rate is 1 tph (for cleaned and screened coal).

5.3.2 Product Gas and Fines

All product gas and fines are to be burned in a fluidized-bed combustor (AFBC) to provide process heat and sulfur reduction.

5.3.3 Char Product

Char product is to be cooled from 1400° to 100°F in an inert atmosphere.

5.3.4 Liquid Products

Condensable material will be collected and sent to an upgrading company for evaluation. Any remaining material is to be burned in the AFBC.

The pilot plant is to consist of five primary areas:

- Area 100 - Coal Preparation
  This area is to include receiving, storage, handling, crushing, and screening equipment.

- Area 300 - Utilities
  Natural gas, cooling water, and electricity should all be assumed to be present at the boundary of the building. The proposed building is 3200 square feet in size and is to include receiving (20'L x 40"W x 30'H), change facilities, lunch room, and shop (60'L x 16'W x 18'H),

offices (60'L x 24'W x 60'H). Provisions are to be made for treating wastewater.

- Area 300 - Carbonization
  This area is to include the AFBC, carbonizing reactor, and cyclones.

- Area 700 - Char Upgrading
  This area will include cooling and storage of char. Details to be specified.

- Area 500 - Gas Quench and Liquid Separation
  This area is to include the venturi scrubbers, separator vessels, circulation pumps, and holding tanks.

5.3.5 Location

Two sites are to be used for the cost estimate: Grand Forks and Bismarck, North Dakota.

5.4 Process Design

5.4.1 Literature Search

Process engineers are reviewing all reports submitted by UND as well as other Bechtel information on coal handling, grinding, reaction, and beneficiation. One of the areas of concern is whether the fluidized-bed combustor can be operated under pressure to provide the heat for process with flue gas. Detailed information on the heat and mass balance will be required by UND to influence this decision and to proceed with the design basis.

5.4.2 Heat and Mass Balance

XBI is adapting a basic program to perform a heat balance around the carbonizer. This will help to define the process configuration.

5.4.3 Conceptual Process Flow Diagrams

Preliminary process flow diagrams are being developed for the areas of carbonization, calcining, and gas quench based on UND literature. These will be updated as the process design evolves.

5.4.4 Process Design Basis

After a thorough review of the UND literature, XBI has begun to prepare a process design basis document to establish material flows and conditions for all feed and product streams for both feed coals.

5.5 Personnel

The XBI personnel assigned to the project are the following:

Ron Gravois	-	Project (part-time)
Scott McFeely	-	Process Lead (full-time)
Brian Davis	-	Process (part-time)
Tal Angelosante	-	Process (part-time)

APPENDIX A

SCOPE OF WORK

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

Subtask 4.7 - Preliminary Engineering Design

Based on the results obtained in Task 2, 3, and 4, Xytel-Bechtel, Inc. (XBI) shall prepare a preliminary engineering design for a one-ton/hour mild gasification process development pilot plant (PDPP), consisting of process flow diagrams with detailed heat and material balances, process and instrumentation diagrams (P&IDs), plot plans and equipment arrangement drawings, conceptual drawings, utility requirements, equipment specification sheets, and electrical one-line drawings, sufficient to define the cost and construction schedule for an integrated process development pilot plant, complete with mineral char and liquid upgrading equipment.

AMAX R&D will assist EERC in all aspects of this work. XBI will perform the detailed work based on information provided by EERC and AMAX. EERC will have the lead responsibility for providing information on mild gasification based on Task 2 and 4 results. AMAX will have the lead responsibility for providing information on char upgrading based on Task 3 results and its general understanding of char-upgrading processes, as well as processes to produce a metallurgical coke substitute, such as the Pellet Technology, FMC, and Bergbau Forschung processes.

AMAX will participate in the selection of the engineering company and assist in reviewing the work as it progresses. They will review the draft final report and provide input on coal cleaning, char upgrading, char uses, and liquid uses, based on their technical and business background.

Deliverables

XBI will provide the following deliverables to EERC:

1. Monthly status reports
2. Process flow diagram with heat and material balance (conceptual design)
3. PDPP piping and instrument diagram
4. Utility P&ID
5. Plot plan and equipment arrangement drawing
6. 3D CADD conceptual drawing
7. Utility requirements
8. Equipment specification sheets
9. A schedule for the engineering, procurement, and construction of the PDPP

10. A capital cost estimate

11. A final report, including process flow diagrams with mass and energy balances, P&IDs, and cost/schedule estimates

EERC shall provide the reports specified in the reporting requirements checklist to Morgantown Energy Technology Center. Special reports shall include a topical report describing the results of Subtask 4.7.

END

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