**Coal Technology Program
Progress Report for January 1976**

MASTER

OAK RIDGE NATIONAL LABORATORY

OPERATED BY UNION CARBIDE CORPORATION FOR THE ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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COAL TECHNOLOGY PROGRAM
PROGRESS REPORT FOR JANUARY 1976

MARCH 1976

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37830
operated by
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PREVIOUS REPORTS IN THIS SERIES

ORNL-TM-5044, Progress Report for August 1974
 ORNL-TM-5045, Progress Report for September 1974
 ORNL-TM-5046, Progress Report for October 1974
 ORNL-TM-4787, Progress Report for November 1974
 ORNL-TM-4796, Progress Report for December 1974
 ORNL-TM-4850, Progress Report for January 1975
 ORNL-TM-4873, Progress Report for February 1975
 ORNL-TM-4892, Progress Report for March 1975
 ORNL-TM-4946, Progress Report for April 1975
 ORNL-TM-4966, Progress Report for May 1975
 ORNL-TM-5010, Progress Report for June 1975
 ORNL-TM-5037, Progress Report for July 1975
 ORNL-TM-5092, Progress Report for August 1975
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 ORNL-TM-5186, Progress Report for October 1975
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COAL TECHNOLOGY PROGRAM PROGRESS REPORT FOR JANUARY 1976

ABSTRACT

This report - the eighteenth of a series - is a compendium of monthly progress reports for the ORNL research and development projects that are in support of the increased utilization of coal as a source of clean energy. The projects reported this month include those for hydrocarbonization research, solid-liquid separations, engineering evaluations of nuclear process heat for coal conversion, analytical chemistry, engineering evaluations of the Synthoil and Hydrocarbonization processes, coal-fueled MIUS, and experimental engineering support of an in situ gasification process.

1. SUMMARY

J. P. Nichols

Highlights of our progress in January are summarized below:

° Most of the effort during this month in hydrocarbonization research has dealt with several needed modifications to equipment. A run with the atmospheric pressure hydrocarbonizer containing an axial draft tube to induce solids recirculation was completed successfully. The liquid yields from this run appeared to be substantially greater than in previous runs without the draft tube. Coal feeding problems hampered three attempts to operate the bench-scale system and corrective actions are underway.

° Additive agglomeration tests in supporting research and development in separations technology have continued to show that a commercial additive from Tretolite will significantly improve settling rates of solids in Solvent Refined Coal Unfiltered Oil (SRC-UFO). Some success has been obtained with inorganic additives, and it has been decided to continue these studies as a small scale effort. It is believed that investigation of these cationic/anionic inorganic materials will contribute to the understanding of agglomeration in heavy organic mixtures such as coal-derived liquids.

° In engineering evaluations of nuclear process heat for coal conversion, two draft reports from United Engineers were reviewed - "Evaluation of a Coal Liquefaction Process Using Either a Nuclear or Fossil Heat Source" and "An Evaluation of Pollution and Water Consumption Related to Selected Coal Conversion Processes" - and after comments by us will constitute a portion of our report to Fossil Energy on the application of nuclear process heat to coal conversion.

° In analytical chemistry, aqueous by-products from the Synthane gasification process and simulated in-situ oil shale retorting are being analyzed for organic content. Also, preparations are being made to commence a plant microcosm exposure study with simulated stack gas emissions.

° In the engineering evaluations work for Synthoil, costs were obtained for most equipment items in the coal preparation section. The flowsheet and overall material balance for a combined centrifugation-filtration plant were completed. Compilation of utilities requirements for the overall facility indicate that a low-Btu gas production facility using additional fresh coal will be required. For Hydrocarbonization, the design and cost estimate for the coal beneficiation system and cost estimate for the hydrogen plant were received. A preliminary flowsheet and a layout plan for coal and limestone preparation were prepared.

° The detailed design effort of Phase III and the preparation of a bid package for procurement of the fluidized bed furnace in the coal-fueled MIUS program continues. An initial mixing test of coal injected into a fluidized bed was completed in the cold flow model and development of the vibrator-eductor coal feed system continued.

° In experimental engineering support of an in situ gasification process, scouting studies defining the effectiveness of aerosol disengagement techniques were continued. Electrostatic precipitation and packed tower scrubbing were examined, and only packed tower scrubbing appeared to be completely effective.

2. HYDROCARBONIZATION RESEARCH

H. D. Cochran, Jr.

Summary

During January, the Lucite model of the recirculating fluidized bed reactor was used to help solve problems encountered in the atmospheric pressure hydrocarbonizer. A run with the atmospheric pressure hydrocarbonizer containing an axial draft tube to induce solids recirculation was completed successfully. The liquid yields from this run appeared to be substantially greater than in previous runs without the draft tube. Reduced cracking of volatilized liquids in the reactor is the postulated reason for increased liquid recovery. Coal feeding problems hampered three attempts to operate the bench-scale system during the month. It is believed that the problems are understood, and corrective actions have been taken.

2.1 Experimental Development

P. R. Westmoreland, J. B. Gibson,
R. L. Andrews, and J. C. Rose

Ambient mock-ups

Ambient studies with the 4-in.-ID recirculating fluidized bed have made significant contributions to the experimental program. Run AHC-10 (discussed below) was marked by excessive solids carryover, by difficulty in setting gas flows to cause solids recirculation, and by difficulty in determining whether recirculation was occurring.

Although part of the solids carryover in Run AHC-10 resulted from an unusually high fines content, the reactor geometry was also important. Mock-up tests using char from Run AHC-9 (3.4 wt % less than 200 mesh) indicated that the coal plume above the draft tube exceeded 12 in. throughout the optimal range of operation, resulting in substantial solids carryover. Reducing the draft tube length from 33 in. to 24 1/4 in. (1 1/2 in. above the char withdrawal pipe) and addition of a solids deflector sharply decreased carryover. These modifications were duplicated on the atmospheric hydrocarbonizer.

Coal prepared for Run AHC-11 was tested in the mock-up to establish gas flow settings for stable operations. Stable recirculation occurs only within an envelope of flow settings. Using the minimum fluidization flows calculated by the correlation of Wen and Yu¹ with minimum entrained flow for the draft tube approximated according to Kunii and Levenspiel,² feed rates were predicted which fit within the envelope of stable recirculation. Ambient mock-up tests of the coal prepared for Run AHC-11 confirmed stable recirculation at the calculated gas feed rates, so the correlations were extended to the high-temperature hydrogen operation for Run AHC-11.

Characterization of solid recirculation performance in Lucite models will be continued as necessary in support of recirculating fluidized-bed operation.

High-temperature studies

Two recirculating fluidized bed experiments were attempted in the atmospheric pressure reactor. Run AHC-10 was terminated because of a plugged line. In the next attempt, Run AHC-11, smooth batch operation of recirculating fluidized-bed hydrocarbonization was achieved. A final carbon balance on Run AHC-8 showed 100.2% carbon recovery, and a preliminary balance on Run AHC-9 was determined.

Upon installation of the draft tube reactor, Run AHC-10 was attempted using argon for carbonization at 1060°F. Over 25 g of coal fines were blown out of the reactor during start up. (Excessive fines, about 13 wt %, resulted from inadequate sieving of the feed.) Bed pressure drop data could not be interpreted as to whether recirculation was taking place. Run AHC-10 was aborted about 25 min into the run because of a 25-g plug in the cyclone-to-condenser line. As described above, later experiments using ambient mock-ups proved that this carryover resulted both from fines content and from a long draft tube.

Results from mock-up testing contributed to successful operation in a second atmospheric hydrocarbonization experiment with the recirculating fluidized bed, Run AHC-11. Maximum temperature was 1020°F, and hydrogen was used as the fluidizing gas. The yield of liquids and tar from Run AHC-11 appeared to be substantially greater than in earlier runs without the draft tube, c.f. Runs AHC-8 and AHC-9. It is postulated that, as a result of the substantially reduced vapor residence time in the reactor with the draft tube installed, thermal cracking of the liquid products is reduced and greater recoveries may be achieved.

Final analysis results from Run AHC-8 (batch fluidized-bed hydrocarbonization to 1060°F) indicated 100.0% recovery of carbon, with 13.2% gas yield of the carbon and with 12.2% liquid yield. A preliminary carbon balance on Run AHC-9 (batch fluidized-bed hydrocarbonization to 1000°F) gives only 89.3% recovery. Solids and tar were not recovered from the cyclone-to-condenser line. Gas yield was 12.5% and liquid yield was 11.1%, suggesting that char recovery may have been incomplete.

No further atmospheric pressure hydrocarbonization experiments are planned although the equipment, as modified for residua carbonization, is suitable for further experiments should they be necessary. Carbon balances and yield data from Runs AHC-9 and AHC-11 will be reported when completed, and a final, summary report of the atmospheric pressure hydrocarbonization experiments will be included in a later progress report.

2.2 Bench-Scale System

H. D. Cochran, Jr., and G. L. Yoder

Design and review

Preliminary design of modifications to the bench-scale system necessary to raise its pressure capability from 20 to 80 atm is underway. Three major modifications will be required:

- (1) A booster compressor for hydrogen having the following specifications is required: suction pressure 300 to 1250 psig, discharge pressure 1250 psig, capacity 70 scfm. At present it appears that an oil- and water-free, air-driven, nonlubricated compressor is the optimum choice. Detailed specifications are being prepared for bids.
- (2) At 80 atm the present preheater has a maximum wall temperature of 1050°F. Consequently, for operation at 80 atm and 1250°F, additional preheat will be required.
- (3) The reactor, presently rated for 20 atm at 1250°F, will have to be replaced.

For safety reasons, the 80-atm system will be limited to a hydrogen inventory of $\leq 260 \text{ ft}^3$ STP and a hydrogen supply rate of $\leq 70 \text{ scfm}$. It is also necessary to have a coal feed rate of 10 lb/hr and a solids residence time on the order of 30 min. For fluidization of the solid with $\leq 70 \text{ scfm}$ of hydrogen in a bed having a solids residence time of about 30 min at about 10 lb/hr feed rate, some compromise on reactor inside diameter and height is necessary. A 2.5-in.-ID reactor appears to be the best compromise. Larger reactor diameters with reasonable particle size require more gas for fluidization. Smaller reactor diameters require excessive reactor height to attain the desired solids residence time.

Initial design efforts have focused on an integral reactor-preheater with the following characteristics: (1) a cold wall outer shell for pressure containment, (2) insulated internally from (3) internal heaters, and (4) a 2.5-in.-ID reactor core. Design difficulties, as yet unresolved, are associated with the flanged end closures and the inlet and outlet nozzles which are complicated by thermal expansion.

Bids will be solicited for the hydrogen booster compressor, and a detailed design of the integral reactor-preheater will be prepared for preliminary review.

Fabrication and installation

Several subsystems for the bench-scale hydrocarbonizer were completed as follows: the hydrogen supply station was completed and gas supply valving was connected. Combustible gas monitors and hydrogen detectors in the process area were tied into the control panel. The oxygen analyzer in the process gas line (to verify purging before hydrogen introduction) was tied into the control panel. Heated lines between the preheater and reactor and between the reactor and scrubber were insulated. The heat exchangers on the scrubber were cleaned and reinstalled.

No effort is planned in this area except maintenance as required.

Operation

Continued difficulties in feeding coal to the reactor have frustrated plans for operation during January. It is believed that the problems are understood, and corrective actions are underway.

In attempts to prepare and feed coal meeting the feeder design specifications, -50 by +150 mesh, excessive fines (~ 20 wt % $< 10 \mu$) caused the coal to lose its free-flowing characteristics, and bridging was experienced in the feed hopper above the coal feeder. The fines are difficult to remove by screening; holdup times of 0.5 to 1.0 hr on the screen are required. Facilities for air classification, to elutriate fines before screening, are not presently available to this project.

As an interim solution to this problem, coal screened to -20 by +50 mesh (~ 2 wt % $< 10 \mu$) was fed to the reactor in three attempts to run under reactor conditions of 20 atm and 1200°F with nitrogen. Although it was possible to feed for short periods of time, saltation of larger particles in the pneumatic transport line repeatedly plugged the line.

Three actions have been taken to overcome these problems:

- (1) Approximately 1 ton of Wyodak coal was shipped to Morgantown Energy Research Center (MERC) for grinding and classification to -50 by +150 mesh specifications. This material should be available within a few weeks to sustain our operation for about 5 runs.
- (2) Gas supply controls for the pneumatic transport of coal have been modified to permit sustained pneumatic transport of -20 mesh coal.
- (3) A 5-lb batch of carefully screened -50 by +150 mesh coal (~ 2 wt % $< 10 \mu$) has been prepared to demonstrate that coal which meets feeder design specifications can be fed successfully.

Operations are presently delayed for the following safety-related shakedown tests which are now in progress: (1) leak test of the hydrogen supply station, (2) witnessed pressurization test of the bench-scale system at design temperature to pressure 1.15 times design pressure, (3) DOP test of in-line filter to assure 99% removal efficiency for 0.3μ particulates, (4) smoke generator verification of airflow in process room, and (5) proof testing of the emergency shutdown system.

Four runs are planned for February as follows: (1) demonstrate feeding of 5 lb of -50 by +150 mesh coal to reactor at 20 atm and 1200°F with nitrogen, (2) feed approximately 50 lb of -20 by +50 mesh coal to reactor at 20 atm and 1200°F for material balance with nitrogen, (3) feed approximately 100 lb of -50 by +150 mesh coal (if available from MERC) to reactor at 20 atm and 1200°F with nitrogen, and (4) feed approximately 100 lb of -50 by +150 mesh coal to reactor at 5 atm and 1050°F with hydrogen if (3) is successful.

2.3 Residua Carbonization

H. D. Cochran, Jr., and J. B. Gibson

Two runs with coal were attempted in the residua carbonizer during January. In the first run severe problems were experienced with carryover of coal particles from the reactor to the condenser which resulted in plugging of a line and termination of the experiment. Ambient studies were conducted with a Lucite model of the reactor and draft tube to provide a better understanding of the carryover problem. As a result of these studies, the draft tube was reduced in length from 33 in. to 24 in. and a cup-shaped deflector was added 9 in. above the top of the draft tube. As a result of these changes, carryover was greatly reduced in the second run which was completed successfully. More information on these runs is given separately in the report on the Hydrocarbonization project.

The feed system and char receiver were fabricated this month, and installation of these vessels will begin shortly. In preparation for feeding studies, we have begun grinding residua. The heat generated by the grinding operation tended to make the particles of residua agglomerate. Mixing dry ice with the residua in the grinder alleviated the sticking problem.

The first draft of the review and evaluation of the CSF carbonizer has been completed. The draft has undergone internal review and is now being revised.

The revised draft of the review and evaluation will be completed this month and will be distributed for comments. Installation of the feed vessel and char receiver will be completed and shakedown of the feeding system will begin. If the feeding system performs adequately,

the first experiment with residuum will be attempted; otherwise, one additional experiment with coal will be made while feeding problems are resolved.

2.4 References for Sect. 2

1. C. Y. Wen and Y. H. Yu, A.I.Ch.E. Journal 12, 610 (May 1966).
2. D. Kunii and O. Levenspiel, Fluidization Engineering, New York, John Wiley & Sons, 1969, p. 78.

3. SUPPORTING RESEARCH AND DEVELOPMENT IN SEPARATIONS TECHNOLOGY

B. R. Rodgers

Summary

Additive agglomeration tests have continued to show that a commercial additive from Tretolite will significantly improve settling rates of solids in Solvent Refined Coal Unfiltered Oil (SRC-UFO). Discrepancies were noted between the bench-scale and laboratory-scale results; namely, longer settling times were required on the bench-scale apparatus to produce a given clarity of upper phase. Some success has been obtained this month with inorganic additives, and it has been decided to continue these studies as a small scale effort. It is believed that investigation of these cationic/anionic inorganic materials will contribute to the understanding of agglomeration in heavy organic mixtures such as coal-derived liquids.

3.1 Additive Agglomeration Studies: Laboratory-Scale

S. Katz and B. R. Rodgers

Agglomeration-settling tests have continued using the laboratory-scale test system and procedures described elsewhere.¹ Previously, series of settling tests for 70 min at temperatures of 210, 245, 280, and 315°C with 0, 250, 500, and 1000 ppm of additive (petroleum type additive from Tretolite) indicated that better settling occurred at higher temperatures and additive concentrations.² However, systematic variations in the ash values resulted; notably, the ash values were either consistently high or consistently low for all tests performed on a particular day. This indicated that the ash content of the starting material might have varied from day to day; possibly due to settling in the storage container. To eliminate the possibility that inadequate mixing of the starting UFO contributed to the observed variations, more vigorous premixing was used

for the test series described below. To reduce other systematic errors, a sedimentation run with UFO only was made just before a run with additive; this allowed direct comparisons at the same conditions. The tests indicate that significant settling can be achieved in less than 30 min (the ash content of the upper 30% fraction was less than 0.15%). The efficacy of the Tretolite additive for improving agglomeration is verified by these results.

The effectiveness of various additive materials such as the Tretolite additive, improvements in testing techniques, and consideration of possible agglomeration or recrystallization mechanisms has stimulated reevaluation of previous studies of inorganic additives. An additional screening test using 80-200 mesh silica gel at a concentration of 4000 ppm showed no improvement in settling rates. Other inorganic materials are under evaluation. A literature search will be made of other materials which are compatible with coal liquefaction liquids.

3.2 Additive Agglomeration Studies: Bench-Scale

B. R. Rodgers, S. Katz, and D. A. McWhirter

Initial results indicate that longer settling times may be required to produce a given clarity of overflow for the bench-scale apparatus compared to the laboratory-scale apparatus. Nine runs this month at temperatures from 390 to 600°F, and Tretolite additive concentrations from 1500 to 4000 ppm, have shown that 100 min is required to obtain a 30% upper phase which meets EPA standards for SRC product. While this time is not excessive for a settling operation, it is considerably longer than the 30 min required to obtain this clarity using the laboratory-apparatus. Reasons for the discrepancies are under study.

3.3 References for Sect. 3

1. J. P. Nichols (Program Director), Coal Technology Program Quarterly Progress Report for the Period Ending September 30, 1975, ORNL-5093 (December 1975).
2. J. P. Nichols (Program Director), Coal Technology Program Quarterly Progress Report for the Period Ending December 31, 1975, ORNL-5120 (in publication).

4. ENGINEERING EVALUATIONS OF NUCLEAR PROCESS HEAT FOR COAL CONVERSION

W. R. Gambill

Second drafts of two reports were received from United Engineers. These reports, titled "Evaluation of a Coal Liquefaction Process Using Either a Nuclear or Fossil Heat Source" and "An Evaluation of Pollution and Water Consumption Related to Selected Coal Conversion Processes" will, after final comment by us, constitute a portion of our February report to Fossil Energy on the application of nuclear process heat to coal conversion. Other sections of the report will address a VHTR-driven direct steam/coal gasification plant (for which a revised block diagram has been prepared and cost estimates are being made); external hydrogen supply from a thermochemical water splitting unit to processes converting coal to methanol, syncrude, SNG, and gasoline; and a review of materials problems related to He-heated process heat exchangers for reforming, coal gasification, and thermochemical hydrogen production.

E. Clark, Chief of gasification programs (DCCU), in a meeting with I. Spiewak and W. R. Gambill in Washington on January 13, indicated that support for this evaluation study would be continued at a level of \$125,000 for the period January through September 1976 - i.e., through the transition quarter. A revised work statement reflecting the priorities established at the meeting has been transmitted to Mayo Carrington.

5. ANALYTICAL CHEMISTRY

W. D. Shults

Summary

Aqueous by-products from the Synthane gasification process and simulated in-situ oil shale retorting are being analyzed for organic content. Organic concentrates are being prepared by different methods for comparison of overall and class extraction efficiencies. A method for producing concentrates of the nitrogen and sulfur containing classes of compounds is being investigated. Preparations are being made to commence a plant microcosm exposure study with simulated stack gas emissions.

5.1 Analyses of Aqueous By-Products from
Fossil Fuel Conversion Process

B. R. Clark, M. R. Guerin, C. H. Ho, and I. B. Rubin

Synthane Coal Gasification

Three portions of the aqueous condensate from the U.S. Bureau of Mines Synthane coal gasification process were fractionated by the Stedman procedure. Ether was the extractant in the first two runs and methylene chloride was used in the third. The first test was carried out to get a feel for handling aqueous samples and backwashing of the extracts was not done; consequently, results of the first two experiments were not expected to check, and did not. Moreover, results from the methylene chloride experiment did not check well with the results from the second ether experiment. Carbon-14 labeled phenol was added in the latter two tests. In both cases, only about 70% of the label was recovered. In the ether extraction all of this phenol was found in the ether-soluble weak acid fraction, while in the methylene chloride extraction, about 85% of the recovered phenol was found in that same fraction and the remainder in the ether-soluble strong acid fraction.

Simulated In-Situ Retorted Shale Oil

Separation of the oil/water product was completed by batch centrifugation. Final yields were approximately: 27% water, 55% oil, and 18% oil/water/solid gelatinous phase. Some preliminary analyses of the neat aqueous phase, using GLC with a Tenax column, indicate monocarboxylic acids as major components. Phenol appears as a trace as well as many nitrogen containing compounds not yet identified. More thorough studies of neat aqueous samples are in progress and organic extracts are being prepared by several methods to obtain concentrates for trace component analyses.

Whole samples of the shale oil and Synthane aqueous by-products have been submitted to collaborators in the Biology and Environmental Sciences Divisions for some mutagenicity and acute toxicity studies.

5.2 Separation of Sulfur and Nitrogen Classes of Compounds from Complex Mixtures

C. H. Ho and A. R. Jones

The production of fractions concentrated in sulfur and/or nitrogen components of a complex mixture is important in the analyses of these materials.

It has been reported that tobacco smoke condensate (TSC) may be separated into three distinct fractions on the basis of the metal-binding activity of its components: (1) a noncomplexing fraction containing TSC constituents which do not bind to metal ions, (2) protonated ligands containing those constituents of TSC able to form zero-charge chelates with metal ions, and (3) nonprotonated ligands forming complexes with metal ions. An attempt is being made to apply the rather poorly defined procedures to TSC and nonpetroleum-derived fuel oils to determine the extent to which sulfur- and nitrogen-containing substances may be concentrated.

A solution of 200 mg of TSC in methyl isobutyl ketone (MIBK) was filtered through a 1- μ Fluoropore membrane and placed on a 2.5-cm-diam by 18-cm-long column of carboxymethyl cellulose (Cu^{2+} form) which had been equilibrated with MIBK. Elution with that solvent washed a brown band through the column to give a yellow eluate. This solution was reduced in volume at 44° and 40 mm pressure to 370 mg of an amber oil which smelled of MIBK. A second brown band was eluted with 95% ethanol. This was concentrated to 95 mg of amber oil of mild odor. All of the remaining dark material at the head of the column was then eluted as a yellow band. Some solvent evaporation yielded 3.117 g of slightly yellow wax with no odor.

The first material eluted is reported to be free of copper. The other fractions, which should be copper ligands, will be freed of the metal, and all three will be examined by gas chromatography with S- and N-specific detectors.

5.3 Plant Microcosm Exposure Studies with Simulated Stack Gas Emissions

A. D. Horton and B. R. Clark

Current studies involve the development of methods to introduce benzene and phenol into an air stream at a controlled rate.

Benzene delivery is accomplished using a glass bulb with an inlet extending to the bottom of the bulb and a vapor outlet at the top. The bulb is charged with benzene; nitrogen is bubbled through the benzene at various flow rates and collected in a gas trap cooled with dry ice. At N_2 flows ranging from 7.1 to 96.8 ml/min, all of the benzene vapor is collected in the first trap of a double trap arrangement. The production of benzene vapor vs flow rate is nonlinear, ranging from 0.8 ml/min vaporized at 7.1 ml N_2 /min to 10.1 ml/min vaporized at 96.8 ml N_2 /min.

The projected air flow through the microcosm is 15 to 20 ft³/min. Benzene vapor injected into the air stream at the above rates would produce a concentration range in the air of 1.9 to 23.7 Vppm at the 15 ft³/min rate, and 0.6 to 7.1 Vppm at the 50 ft³/min rate. The desired range of benzene concentration is 0.10 ppm. This same technique can be used for injecting other volatile liquids.

5.4 Coal and Fly Ash Analysis by a Modified Multielement Isotope Dilution SSMS Technique

D. L. Donohue, J. C. Franklin, and J. A. Carter

A technique has been developed for the analysis of up to 14 elements in coal and fly ash samples by spark source mass spectrometry. This involves the deposition of separated stable isotopes for the elements Pb, Tl, Te, Ba, Cd, In, Mo, Sr, Zn, Cu, Ni, Fe, Cr, and Ca from a solution onto high purity silver powder. This "spiked" silver can then be standardized to determine the true concentrations of the separated isotopes, as well as the "blank" level of normal isotopes for the same elements.

In the analysis of coal, the sample is ashed at 500°C in a muffle furnace, then homogenized on a Wig-L-Bug mixer. Fly ash samples are also homogenized in this way. A weighed amount of sample ash is then intimately mixed with a known amount of spiked silver powder, and formed into electrodes for sparking in the mass spectrometer.

Ion detection is by photographic plate, which is then read on a microdensitometer PDP-8/E system. Calculations of sample concentrations differ from standard isotope dilution work in that a sensitivity factor is necessary because the sample and spike isotopes do not reach chemical (only physical) equilibrium. Accuracy is established by using NBS SRM's 1632 (coal) and 1633 (fly ash). The precision is about 10%. Advantages

of this method include speed and simplicity of sample work-up with a minimum of chance for contamination. An additional advantage is that small samples can be analyzed (from 1 to 10 mg of fly ash).

5.5 Analytical Services

W. R. Laing and L. J. Brady

A total of 177 samples were submitted for analysis during the month. Most of these samples were derived from solids-liquid separation tests. Samples that ranged in weight from 1 to 5 g were burned and the ash was found to be in the range from less than 0.1 to 10%. The use of the new drying apparatus, which consists of eight infrared heat lamps with the heat output controlled by use of Variacs, resulted in a reduction of processing time and made it possible to report the results within two days from the time the samples were submitted for analyses.

A sample consisting of a mixture of methyl naphthalenes plus impurities was tested on the gas chromatograph. A simulated boiling range distribution test indicated that the boiling range was 224 to 262°C. The mixture was found to be two parts 2-methyl naphthalene to one part 1-methyl naphthalene. Four additional elution peaks were observed which together represented about 1% of the sample. These components were not identified chemically.

6. ENGINEERING EVALUATIONS OF THE SYNTHOIL AND HYDROCARBONIZATION PROCESSES

J. M. Holmes, R. Salmon, and E. G. St. Clair

Summary

Synthoil. Costs were obtained for most equipment items in the coal preparation section and a list of equipment power requirements was prepared. The flowsheet and overall material balance for a combined centrifugation-filtration plant were finalized. Process flow diagrams, heat and material balances, and equipment lists were finalized for Units 13 and 15. Minor changes were made to computer codes for calculating material balances for gasification and low-temperature carbonization, and work continued on listing and sizing equipment for these two sections. Compilation of utilities requirements for the overall facility indicate that a low-Btu gas production facility using additional fresh coal will be required.

Hydrocarbonization. The design and cost estimate for the coal beneficiation system was completed by McNally-Pittsburg Corporation and the final report was received. A cost estimate for the hydrogen preparation plant was received from C&I/Girdler Incorporated. Vessels for the hydrocarbonization system were designed and costs are now being estimated. A computer code was developed for the design and costing of the DEA scrubbing systems for H_2S and CO_2 removal. Equipment lists were prepared for the DEA acid gas removal plant (Unit 13) and the filtration plant (Unit 19). A preliminary flowsheet and a layout plan for coal and limestone preparation were prepared.

6.1 Synthoil Process

R. Salmon, E. G. St. Clair, M. S. Edwards,
W. C. Ulrich, and D. A. Dyslin

Process flow diagrams, heat and material balances, and equipment lists were finalized for Unit 13, recycle gas and fuel gas treating (DEA), and Unit 15, low-temperature CO shift feed-gas treating.

Preliminary utilities requirements for the overall Synthoil facility were compiled. It appears that fuel gas production within the facility is not adequate to supply heat, steam, and power requirements, and that a low-Btu gas facility using additional fresh coal will therefore be required.

Work on various sections of the flowsheet progressed as follows:

(1) Coal Handling and Preparation: Costs were obtained for most equipment items in the coal preparation section, and a list of power requirements was prepared.

(2) Gasification: The gasifier material balance was revised to account for a change in the moisture content of the coal feed. Work continued on listing and sizing equipment for the unit.

(3) Low-Temperature Carbonization: Paul M. Yavorsky of the Pittsburgh Energy Research Center (PERC) visited ORNL and talked with members of the evaluation project about the Synthoil process. One result of this discussion was a revision to the computer code for calculating the carbonizer material balance. The change gives a liquid recovery rate from carbonization of the solid-liquid separation residue more nearly representative of that predicted by PERC for this operation. Work continued on listing and sizing equipment, and a preliminary plot plan of the unit was prepared.

(4) Solids-Liquid Separation: The flowsheet and overall material balance for a combined centrifugation-filtration plant were finalized. Equipment sizing is underway.

6.2 Hydrocarbonization Process

J. M. Holmes, D. S. Joy, G. R. Peterson, M. S. Edwards,
C. B. Smith, and D. A. Dyslin

The designing and cost estimating of major equipment items in the hydrocarbonization plant is continuing. The various sections of the flowsheet progressed as follows:

(1) Coal Handling and Preparation: A preliminary flowsheet and a layout plan for coal and limestone preparation were prepared. Reviewers questioned the use of a reclaim coal pile and gravity-feed active piles vs the use of stacker-reclaimers as proposed by the McNally-Pittsburg Company. Further study is being done on this question.

(2) Coal Beneficiation: The design and cost estimate for the coal beneficiation system prepared by McNally-Pittsburg Corporation was completed and the final report was received. A revision to the flowsheet is under preparation to include raw coal needed for the fluid bed combustion system which will not have to be beneficiated. This addition was made because of an improvement in the clean/low-grade ratio produced by beneficiation which reduced the amount of low-grade coal available for fluid bed combustion while maintaining a constant feed of clean coal to the hydrocarbonizers.

(3) Hydrocarbonization: Vessels for the hydrocarbonization system were designed and costs are now being estimated. The process design and analysis (PDA) computer code is being used for sizing of the columns, compressors, heat exchangers, and pumps. A decision was made to include a chiller on the hydrocarbonizer recycle stream to reduce the losses of naphtha in the stream as it is reheated by the fluid bed combustor.

(4) Acid Gas Removal: Information was received from the Benfield Corporation concerning the design and cost estimation of two CO₂ removal plants. A PDA computer code was developed for the design and costing of the DEA scrubbing systems for H₂S and CO₂ removal. The DEA acid gas removal plant (Unit 13) equipment was sized, and utility requirements were estimated. Equipment lists were compiled for costing purposes.

(5) Hydrogen Preparation Plant: A cost estimate for the hydrogen preparation plant was received from C&I/Girdler Incorporated. The estimate also included process and utility consumption information.

(6) Oil-Solids Separation: The filtration plant (Unit 19) equipment was sized, and utility requirements were estimated. Equipment lists were also prepared. Capital cost for Unit 19 is estimated at \$9 million.

(7) Ammonia Recovery: A letter was written to Chevron Research providing specifications for ammonia and H₂S removal from the sour water. Chevron will furnish a conceptual design and cost estimate for this plant.

7. COAL-FUELED MIUS

A. P. Fraas and W. R. Nixon

This project for analysis, design, and demonstration of a concept utilizing a fluidized-bed coal combustion system as a heat source for a gas turbine generator suitable for applications in Modular Integrated Utility Systems (MIUS) is carried out under the ORNL-HUD-MIUS Program within the Energy Division. Work is supported by the U.S. Department of Housing and Urban Development under HUD interagency Agreement No. 1AA-H-40-72 and by the Energy Research and Development Administration, Office of Fossil Energy, under OCR contract No. 14-32-0001-1742. The project consists of four phases: I - Conceptual Preliminary Evaluation; II - Conceptual Design; III - Detailed Design and Construction; and IV - Shakedown, Performance, and Endurance Tests.

Summary

The detailed design effort of Phase III and the preparation of a bid package for procurement of the fluidized bed furnace continued. An initial mixing test of coal injected into a fluidized bed was completed in the cold flow model and development of the vibrator-eductor coal feed system continued.

Furnace Procurement

The Combustion Engineering plant at Chattanooga was visited to discuss fabrication of the fluidized bed furnace. Results were most encouraging in that their facilities are more than adequate for the job, they have many of the required materials in stock, they can (and in fact prefer to) prepare the required shop drawings, and they are very interested in doing the job.

A package of drawings and specifications will be prepared as soon as possible and forwarded to vendors with a request for bids.

Turbine-Generator Unit

The progress of the turbine evaluation study was reviewed in a meeting with AiResearch on January 14. The aerodynamic performance and control studies have been completed. Analysis of the bearings and seals is continuing. There appears to be no aerodynamic or surge problems with closed cycle operation. A reference design speed of 75% of the full turbine speed was selected for use for the remainder of the study. A 6-module recuperator appears to be a better choice for the system than a 4-module unit, and the 6-module design will be emphasized in the rest of the study.

Cold Flow Tests of a Fluidized Bed

An initial coal mixing test was completed by injecting coal at the lower center of the bed, running fluidizing air for short time intervals, and sampling the bed at four points at three levels. The fluidizing velocity for the test was slightly lower than the minimum operating velocity that had been selected from flow visualization tests. The results at 20 seconds showed good vertical mixing, but no horizontal mixing. After 40 seconds the coal distribution was uniform in the vertical direction but still very little horizontal mixing was found. The results after 80 seconds showed only a small amount of horizontal mixing. The results indicate that good vertical mixing, but essentially no horizontal mixing, will occur at very low fluidizing velocity. Additional tests will be run at higher velocities to establish the mixing characteristics over the operating range of the fluidized bed.

Coal Metering and Feed System

Tests of the vibrator-eductor coal feeder system included measurements of the reproducibility of total feed rate and the feed rate of each of the four eductors. The total feed rate was about 500 lb/hr at the maximum setting on the vibrator and was reproducible to within about 5%. The variation of flow among the four lines was about 15%. Fine adjustment of the eductor nozzles was found to reduce this variation. Work will be continued to find the optimum setting for each of the eductors to obtain consistent flow rates with the minimum variation among the four lines.

8. EXPERIMENTAL ENGINEERING SUPPORT OF AN IN SITU GASIFICATION PROCESS

R. C. Forrester, III

Summary

Experimental work for this program was suspended in July to permit reassembly of the apparatus in an area which provides better containment of liquid and gaseous effluents and facilities more appropriate for associated clean-up operations. In the interim, analysis of data from early experimental work has been performed with the objective of revealing any preliminary implications of the first pyrolysis tests.

Scouting studies defining the effectiveness of aerosol disengagement techniques were continued under the immediate supervision of C. J. Golden, a summer employee presently completing requirements for the Master's degree in Chemical Engineering. Electrostatic precipitation and packed tower scrubbing (using water as the working fluid) were examined in detail, though only packed tower scrubbing appeared to be completely effective.

8.1 Large-Block Pyrolysis Studies

R. C. Forrester, III, P. R. Westmoreland,
F. H. Wilson, and G. D. Owen

Work accomplished during the first third of FY 1976 has included the development of a computer code for rapid data analysis and an evaluation of the first thirteen pyrolysis experiments. These results were presented at the Second Underground Coal Gasification Working Group for Laboratory Research meeting at the Morgantown Energy Research Center, October 16, 1975. Some of the calculational results have been transmitted directly to Jack Campbell of LLL for use in their kinetic model code.

As a result of funding delays, installation of the experiment in building 4505 was only completed in December with preliminary tests underway during the first week of January. These tests were successful and the generation of data has resumed.

8.2 Coal-derived Aerosols: Disengagement Techniques

C. J. Golden, P. R. Westmoreland, and R. C. Forrester, III

As mentioned previously in earlier reports, rapid cooling of condensable organic vapors produced by pyrolysis of coal leads to the formation of aerosols which are extremely difficult to separate from the product gas stream. Such difficulties led us to conduct some quick scouting tests of traditional disengagement techniques with the objectives of improving the pyrolysis experiment and providing recommendations for others in the ORNL Coal Technology Program who might have aerosol disengagement problems. Qualitative Evaluations of systems tested to date are shown below.

Table 8.1. Qualitative evaluation of some traditional disengagement techniques for trapping coal-derived aerosols

Technique	Evaluation and Comment
Spray towers	ineffective ^a
Bubble columns	ineffective ^a
Packed towers	very effective ^a
Cartridge-type string filters	very effective
Electrostatic precipitation	partially effective
Glass wool demisters	partially effective
Internal cooling coils and glass wool	effective

^aThese were one-stage devices using water as the working fluid. Repeat tests using a low vapor pressure organic solvent (such as methylnaphthalene) are planned.

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