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

MASTER

OAK RIDGE NATIONAL LABORATORY

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COAL TECHNOLOGY PROGRAM
PROGRESS REPORT FOR OCTOBER 1976

Date Published: December 1976

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COAL TECHNOLOGY PROGRAM PROGRESS REPORT FOR OCTOBER 1976

ABSTRACT

This report - the twenty-seventh of a series - is a compendium of monthly progress reports for the ORNL research and development programs that are in support of the increased utilization of coal as a source of clean energy. The projects reported this month include those for coal conversion process development, materials engineering, alkali metal vapor topping cycles, a Critical Components Test Facility, engineering and support studies, environmental assessment studies, and coal-fueled MIUS.

1. SUMMARY

J. P. Nichols

Highlights of our progress in October are:

- In our hydrocarbonization/residue carbonization work, run HC-8 was completed with the 20-atm bench-scale hydrocarbonizer in a recirculating fluidized bed mode. Runs RC-15 and RC-16 in the 1-atm residue carbonizer showed very encouraging results - it appears that the system may be sufficiently operable for material balance experiments to begin.

- In our work for experimental engineering support of in situ gasification, a two-dimensional pyrolysis gas production mechanism, assumed to explain data obtained with as-received coal samples, has been verified by utilizing specimens which were dried at 125°C under vacuum prior to pyrolysis.

- In our coal-solvent-hydrogen mixing work, installation was completed of a facility for circulating coal/water/air mixtures through candidate reactor designs to test their applicability for hydrogenating coal/donor solvent slurries. A series of venturis is being considered as a candidate reactor, and a preliminary estimate was made of the number of venturis and the size of each venturi which would be required for full scale coal hydrogenation plants producing 50,000 and 100,000 bbl/day of liquid products.

- In our materials engineering effort, we continued the preparation of our report on the needs for piping and pressure vessel data, continued the testing of methods for the nondestructive examination of thin coatings, completed nine runs to determine the rate of iron carbonyl formation with 1/2% Mo steel exposed to simulated synthesis gas, and continued thermal analysis work in support of design studies of prestressed concrete pressure vessels.

- We continued water tests to evaluate nucleation characteristics of our gas-fired potassium boiler, as well as the design and fabrication of components that will be needed when we begin tests with potassium.

- We began work on a fluidized bed coal-fired alkali metal vapor topping cycle project this month by compiling data on the properties of potassium and cesium and beginning furnace-boiler design studies.

- We completed, for ERDA/FE review, two preliminary reports that summarized the status of our conceptual design studies relating to a Critical Components Test Facility.

- In our project for engineering studies and technical support, we continued work in process modeling and on the Process Research Digest and initiated a project to survey industrial equipment capabilities.

- We are substantially underway in our process and program assistance work, with studies of low-Btu gasification, direct combustion, in situ gasification, beneficiation, gas cleanup, and transportation.

- In our environmental assessment work, we completed a preliminary draft of suggested generic environmental requirements for use in RFP's.

2. COAL CONVERSION PROCESS DEVELOPMENT

J. R. Hightower, Jr.

Coal conversion process development activities are carried out in the Chemical Technology Division. This section discusses hydrocarbonization studies in a 20-atm bench-scale facility, studies of residue carbonization in an atmospheric pressure carbonizer, engineering support studies for in situ gasification, and studies for identifying and characterizing alternate reactor concepts for coal hydroliquefaction.

2.1 Hydrocarbonization Research

H. D. Cochran, P. R. Westmoreland, C. H. Brown,
J. C. Rose, and L. S. Dickerson

Installation of equipment, including a Mikro Pulverizer hammer mill and drum lifting equipment, was completed in the Laboratory's solids handling facility. An inert gas purge system permits grinding and sizing to be performed without exposure to air.

Routine experimentation with the 20-atm bench-scale coal hydrocarbonization system resumed in October:

HC-8 - Recirculating fluidized bed reactor at 800°F and 300-psig H₂ pressure, Wyodak subbituminous coal fed at 13.6 lb/hr for 6.2 hr.

Samples and data from this experiment are being analyzed. Material balances will be reported in the November monthly report.

Table 2.1 compares properties of Wyodak coal which was prepared in March 1976 at Morgantown Energy Research Center (used in runs HC-1 through HC-7) and Wyodak coal which was prepared in our solids handling facility for HC-8 (as described in the September 1976 monthly report). Higher moisture content is one difference in HC-8 coal, since more heat is consumed when moist coal is dried in the reactor. Subsequent modifications to the drying procedure have been shown consistently to permit attaining moisture levels less than 5 wt % without uptake of oxygen. The coal prepared under inert gas blanket for run HC-8 appears also to be lower in oxygen content.

Particle size and flow properties are important in feeding coal by pneumatic transport. Higher mean particle size for run HC-8 suggested that more gas is required to transport this coal. A lower angle of internal friction indicates that HC-8 coal should be more pourable, lessening any bridging problems in the feed hopper.

Table 2.1 Comparison of MERC-prepared and ORNL-prepared Wyodak subbituminous coal

	MERC-prepared coal used in HC-1	ORNL-prepared coal used in HC-8
Moisture	9.95%	15.3%
Ash, moisture-free	7.00%	8.17%
Volatile matter, moisture-and-ash free	46.9%	46.9%
Fixed carbon (by difference), maf	53.1%	53.1%
Ultimate analysis, moisture-and-ash free		
Carbon	71.3%	72.8%
Hydrogen	5.59%	5.62%
Nitrogen	1.02%	1.01%
Sulfur	0.45%	0.64%
Oxygen (by difference)	21.7%	19.9%
Calorific content (Btu/lb maf coal)	11,960	12,450
Particle size: mesh, mean diameter, wt %		
+20	0.00%	0.1%
-20 +28 (707 μ)	0.01%	0.2%
-28 +35 (500 μ)	0.06%	0.4%
-35 +48 (353 μ)	0.19%	13.8%
-48 +60 (273 μ)	10.61%	13.1%
-60 +80 (210 μ)	30.84%	22.6%
-80 +100 (162 μ)	33.69%	9.8%
-100 +200 (105 μ)	10.15%	--
-100 +115 (136 μ)	--	12.5%
-115 +150 (115 μ)	--	6.7%
-150 +170 (96.1 μ)	--	7.3%
-170 +200 (80.7 μ)	--	4.0%
-200 +325 (57.1 μ)	13.95%	7.2%
-325 (42.2 μ)	0.50%	2.5%
Mean particle size, weight basis	168 μ	191 μ
Angle of internal friction	81°*	74°
Angle of repose	35°*	35°

* MERC-prepared coal used in HC-6.

2.2 Residue Carbonization

H. D. Cochran, J. B. Gibson
R. L. Andrews, and J. R. Cox

This project for supporting research and development on residue carbonization is supported by the Division of Coal Conversion and Utilization of ERDA. The work in the year beginning November 1, 1975, consists of three tasks: (1) a review and evaluation of experience with the low-temperature carbonizer in the Cresap, West Virginia pilot plant, (2) modification of an existing reactor to permit continuous feed of solids-laden residues, and (3) operation of the reactor with three feedstocks (vacuum distillation bottoms from the H-Coal process and solvent extraction underflows from SRC and CSF product liquids) at three temperatures up to 1200°F.

A new rotary feeder for the residue carbonization experiment (based on a design used in the 20-atm hydrocarbonizer) was fabricated and installed during October. Following installation of the feeder, two residue carbonization runs, RC-15 and RC-16 were attempted. These runs used a 4/1 ratio of Wyodak char to residue as feed and a reaction temperature of 1130°F. Run RC-15 was interrupted by plugged effluent lines and restarted three times. Total run time was about 1 hr with 5 lb of the char/residue mixture fed. Run RC-16 ran for about 1 hr but there was considerable difficulty in maintaining a steady feed rate. The run was aborted, after feeding 3.5 lb, due to this feeding problem. During cleanup an obstruction was found at the base of the feed hopper.

In all previous runs the material recovered from the scrubber resembled the residue feed. In RC-16 the recovered material had a completely different texture. Instead of being a hard, brittle material at room temperature, it was a soft tar. A more complete analysis will be performed on the material during the coming month.

During the course of this project, significant progress has been made toward the immediate goal of an operable fluidized-bed carbonizer for processing residues. The first experiments were plagued with problems of plugging in the feed line and serious agglomeration in the reactor. Subsequent experiments were terminated by plugging of the effluent line. Gradual improvements in the equipment and the procedures have significantly reduced these operability problems, and we are optimistic that, with an operable system, we can now work toward the objective of attaining volatile yield data as a function of reactor temperature.

2.3 Experimental Engineering Support of an In Situ Gasification Process

R. C. Forrester III, G. D. Owen, S. M. Gibson, and J. D. Cohill

2.3.1 Large-Block Pyrolysis Studies

Recent results of ORNL's two-dimensional pyrolysis studies have demonstrated that gas production accompanying high-temperature pyrolysis of large coal blocks is significantly greater than that obtained from powdered coal subjected to the same external heating rates and maximum temperatures.¹ It was assumed that this increase resulted from steam/char interactions which occurred as water, held in the cooler interior regions of the block, was volatilized and forced to diffuse through the surrounding hot char bank. In order to evaluate the validity of this assumption, experiments have been performed which involved the thorough drying of test specimens at 125°C under a vacuum of 68 cm of mercury. As reported last month, gas production during pyrolysis of the dried blocks was observed to be different from that accompanying tests using as-received blocks in that two distinct regions of activity were not observed as before.² Instead, a single production peak was recorded. Furthermore, overall gas production was significantly reduced, as shown by Fig. 2.2.

Chars produced by both experiments were mildly pyrophoric, self-heating upon exposure to air to maximum temperatures of 70 and 105°C. The char produced by pyrolysis at 0.3°C/min was significantly more resistant to crushing than any produced previously. Analysis of condensable materials recovered during the drying stage of these tests revealed a total carbon content of 50 to 75 ppm. No gas samples were taken during that period.

2.3.2 ERDA's UCG Program

Recent results of ORNL studies supporting ERDA's program for UCG development were presented at the 51st Annual Fall Technical Conference and Exhibition of the Society of Petroleum Engineers of AIME, held in New Orleans, Louisiana, October 3-6, 1976. The paper was entitled "Two-Dimensional Pyrolysis Effects During In Situ Coal Gasification: Preliminary Results."

2.4 Coal-Solvent-Hydrogen Mixing

J. R. Hightower, Jr. and R. C. Lovelace

The objective of the work reported here is to demonstrate that a highly turbulent plug flow reactor of novel design would allow adequate hydrodesulfurization and hydroliquefaction without the use of hydro-treating catalysts. The present work plan calls for two phases: (1) simulated contactor studies which have the objective of identifying suitable reactor configurations (we will investigate a reactor consisting of several venturis in series and one with vortex flow similar to a cyclone device) through tests with water/coal slurries and air, and (2) hydrogenation verification studies in which coal is hydrogenated in a suitable reactor whose design would be based upon results from the first phase studies.

The project work plan was reviewed by ERDA's Division of Fossil Energy Research-Processes (H. Podall, Associate Director) in late September 1976, and redirection of the project was requested. The simulated contactor studies are being slowed down while the specific requests for project changes are being evaluated.

Installation of the facility for circulating water/coal slurries through the various contactors was completed. A device for injecting a KCl tracer pulse was fabricated, installed, and tested; electrodes for use with conductivity meters were fabricated, installed, and calibrated for measuring KCl tracer concentrations at the inlet and outlet of the contactor; and a venturi meter has been fabricated but not calibrated to measure the flow rate of the coal/water slurry through the contactor.

A very preliminary design was made for venturi reactors serving for hydrogenation in plants producing 50,000 bbl/day and 100,000 bbl/day fuel oil using a flowsheet identical to a Synthoil process in all respects except for the reactor used for hydrogenation of coal/solvent paste. This assumes that the product yield and character are the same using the venturi reactor as if the packed Synthoil reactor were used (this is the basic hypothesis of this project which is yet to be demonstrated).

The required residence time of the coal/solvent paste in the venturi was assumed to be the same as that used for a recent economic analysis of the Synthoil process.³ This was calculated to be 5.5 min using the paste space velocity of $280 \text{ lb}_m/\text{hr}\cdot\text{ft}^3$ of catalyst, an estimated⁴ fractional void in the bed of 0.34, and a paste density³ of $10 \text{ lb}_m/\text{gal}$. The oil yield was assumed to be 3 bbl/ton of coal.³ The allowable liquid velocities through the venturi were assumed to be the maximum values reported by Jackson and Collins⁵ for a 4-in. entrance diameter venturi; the velocities were 4 ft/sec at the entrance and 39 ft/sec at the throat. The design pressure was 4500 psi and the design wall temperature was 900°F (480°C). The material of construction was assumed to be 347 stainless steel.

The number of venturis and the size of each venturi are shown in Table 2.1. The venturis are fairly large devices and a large number are required to give the necessary paste residence time. The reactor would probably be fabricated by mandril forging sections of the venturis and welding the sections together to fabricate the reactor. The total quantity of fabricated metal required for the 50,000 bbl/day plant is 1400 tons; the total quantity of metal required for the packed bed reactors in the 50,000 bbl/day plant in Ref. 3 was 1875 tons. Thus, if the cost per ton of fabricated metal were the same for both reactors, the venturi reactor would cost less than the packed bed reactors.

2.5 References for Section 2

1. J. P. Nichols, editor, Coal Technology Program Annual Interim Report for Fiscal Year Ending June 30, 1976, ORNL-5208.
2. R. C. Forrester III, "Two-Dimensional Studies of Coal Pyrolysis: Preliminary Results," presented at the 2nd Annual Underground Coal Gasification Symposium, Lakeview Inn and Country Club, Morgantown, West Virginia, August 10-12, 1976.
3. Economic Analysis of Synthoil Plant Producing 50,000 Barrels per Day of Liquid Fuels from Two Coal Seams: Wyodak and Western Kentucky, ERDA 76-35, November 1975.
4. A. S. Foust et al., Principles of Unit Operations, John Wiley & Sons, New York, 1964, p. 538.
5. M. L. Jackson and W. D. Collins, Ind. Engr. Chem. Proc. Des. Dev. 3, 487 (1964).

Table 2.2 Estimated sizes for single train venturi reactors for
noncatalytic hydroliquefaction of coal to produce
50,000 and 100,000 bbl/day liquid products

Plant capacity (bbl/day)	No. of Venturis required	Venturi dimensions					Weight of metal (tons)
		Entrance diameter (in.)	Throat diameter (in.)	Length (ft)	Entrance wall thickness (in.)	Throat wall thickness (in.)	
50,000	175	28	9	17.5	5.25	1.75	8
100,000	121	40	13	25	7.5	2.5	23

3. MATERIALS ENGINEERING

R. T. King and D. A. Canonico

3.1 Pressure Vessel and Piping Technology Assessment

D. A. Canonico, R. H. Cooper, B. E. Foster,
R. K. Nanstad, G. C. Robinson, and G. M. Slaughter

A review of the piping and pressure vessel needs for coal conversion systems is in progress. The program will identify those areas where additional material property data and data needs are required in order to assure that the pressure boundary components in conversion systems can be designed, fabricated, and operated in a safe and reliable manner.

The Materials Compatibility Considerations and Fabrication chapters were submitted to Fossil Energy Research (FER) for their review. The Materials Selection chapter was returned to ORNL and the suggestions made by FER were considered in preparing the final draft. Final drafts were also completed on the Scope and Design chapters.

3.2 Inspection Techniques for Wear- and Process-Resistant Coatings

R. W. McClung and G. W. Scott

3.2.1 Review and Evaluation (G. W. Scott)

We prepared and presented a 30-min overview summary for an in-house review of the ORNL Metals and Ceramics Division activities that provided all participants a useful look at one another's work and problem areas.

R. W. McClung attended the AGA-EPRI-ERDA Materials Meeting in Germantown, Maryland, September 30-October 1. We are concerned about the rate of information transfer from the coating evaluation and selection program. The last data received from Argonne were contained in their CTP quarterly report ANL-76-60, which covered the period through March 31, 1976. We did receive correspondence from D. Stahl identifying the commercial suppliers of their specimens and materials.

3.2.2 Specimen and Standard Fabrication (J. D. Hudson and D. P. Edmonds)

During this reporting period the Welding and Brazing Lab has concentrated on the preparation of ZrO_2 coatings with known defects. The following samples have been prepared:

- (a) Ultrasonic specimen, Incoloy 800 plate specimen with sandblasted and nonsandblasted areas.

- (b) Ultrasonic specimen, 0.254-mm (0.010-in.) thick ZrO_2 coating with areas of nonbonding and spalling or missing coating. These specimens also have reference sandblasted and nonsandblasted (as-milled) surfaces.

3.2.3 Thermal Testing (W. A. Simpson, Jr.)

We are continuing to use computer modeling to design and optimize thermal testing procedures for the detection of unbonded regions of zirconium oxide coated on an IN-800 substrate. Because of the highly encouraging results obtained from a preliminary computer study, we have begun to examine possible experimental approaches for inspection of the samples. One vendor of plasma torches was contacted, but because of cost we are investigating in-house sources of suitable heat injection devices. An infrared camera capable of operation in the nonscanning (stopped) mode will be available shortly for limited periods of time.

3.2.4 Liquid Penetrant Testing (S. D. Snyder)

In the past we used the I.D.E.A. technique of liquid penetrant testing on an uncracked (ZrO_2) coated specimen.¹ This provided information toward the optimization of penetrant dwell time and excess penetrant removal from this porous coating, but did not show anything about the process when the coating is cracked. Consequently, with the availability of a cracked specimen, the I.D.E.A. technique was applied to the use of ZL-22A high resolution fluorescent penetrant in the following manner:

- (1) Penetrant soak for 1 min.
- (2) Penetrant strip with H_2O at 29°C (85°F) for 5 min.
- (3) Interim dry-blow with air and dry in 49°C (120°F) oven for 1 min.
- (4) Emulsify with ZE-3 emulsifier for 1 min.
- (5) Water wash at 29°C (85°F) for 30 sec.
- (6) Dry-blow with air and dry in 49°C (120°F) oven for 1 min.
- (7) Repeat steps 4, 5, and 6 four more times.

The results were unusual in that there was a reversal of the normal penetrant behavior where the penetrant was removed from the cracks faster than from the porous coating. Also, the emulsifier (colored red) apparently became trapped between the coating and the substrate and continued to bleed from the cracks after the drying steps. Thus, in this one instance the technique does provide an indication of cracks but with poor definition, since the detector is the bleeding red emulsifier in contrast with the fluorescent penetrant that remains trapped in the porous coating.

3.2.5 Ultrasonics (G. W. Scott)

We began a detailed experimental evaluation of conventional ultrasonic techniques which might be used for in-situ inspection from the outside (uncoated side) of a component. We fabricated a flat plate specimen from 1/8-in. IN-800 plate. One surface of the specimen, simulating the "outside," was left in the as-milled condition. The other ("inside") surface has an

as-milled area, a bare sandblasted area, and an area coated with ZrO_2 containing two defects. One defect is an area left in the as-milled² condition and sprayed over with the coating; the second is a sandblasted area left uncoated. The prepared, or "inside," surface was encapsulated to keep it dry while the specimen was immersed in water for evaluation of immersion-coupling inspection techniques.

A pulse-echo interrogation with longitudinal waves at normal incidence has potential for detecting the presence of the coating by resolving the two surfaces of the coating or by some change in the reflections. Higher frequencies appeared to offer the best capability for resolving the coating surfaces.

A 25-MHz transducer was focused on the rear, "inside" surface of the specimen. The coating surfaces were not resolved and only slight signal loss was detectable over the coated areas. By adjusting the focal point of the transducer to obtain the largest signal amplitude for any multiple of the rear-surface echo, it was possible to get a usable signal loss. The largest-amplitude multiple was the third; its amplitude decreased approximately 1 db over the coated areas. The two flaws in the coated area were detectable by the return of the signal to its full amplitude.

While the 25-MHz experiment can be called successful, it should be interpreted with some caution. The sensitivity may not be sufficient to distinguish between coating flaws and other factors such as substrate material variation or part geometry changes.

3.2.6 Electrochemical Testing (G. W. Scott)

We have made differential capacitance measurements as a function of immersed area on electrode specimens of sandblasted IN-800 (bare) and IN-800 coated with 0.010 in. ZrO_2 . Expected experimental values were estimated for IN-800 from published results from other metals; estimates for ZrO_2 were calculated for an impervious dielectric coating of the same thickness. The measured capacitance per unit area for the metal was close to the estimated value; measured values for the ZrO_2 coating were much larger than the estimates, indicating that this coating leaves a significant area of bare metal exposed.

As a check on the initial results, a known impervious coating was improvised by wrapping an IN-800 electrode with mylar tape. The results from this specimen agree closely with estimates for an impervious coating, thereby indicating the validity of the estimating model.

Further experiments will determine available sensitivity, probe sizes, and other design parameters for an inspection system.

3.2.7 High Voltage Testing (G. W. Scott)

We performed tests on the tensile-cracked ZrO_2 specimen, described in earlier reports, which was originally prepared for liquid penetrant testing. After finding evidence of cracks extending through the substrate (see Sect. 6.2.4), we decided to determine whether more arc current could flow through a crack than through the normal coating porosity. The specimen was scanned with our locally-assembled system and an increased arc current was noted in the region of the specimen containing the cracks. We are performing additional experiments to determine the effects of probe tip size, conductor spacing, and voltage on crack sensitivity and resolution.

We received the commercial instrument ordered previously. It has been checked out for operation, but not yet incorporated into any experiments.

3.3 Iron and Nickel Carbonyl Formation and Prevention

J. H. DeVan, H. Inouye, and J. Brynestad

A total of nine runs were completed to determine the generation rates of iron carbonyl from a 1/2% Mo steel in CO and a gas mixture of 17% CO, 51% H_2 , 30% CH_4 , and 2% CO_2 (in vol %). The runs were conducted under a wide range of test conditions to determine whether the present experimental setup was capable of generating enough iron carbonyl to be accurately detected by the atomic absorption spectrometer; and if so, whether the setup was also capable of determining the effect of gas composition, temperature, pressure, and the gas velocity. The tentative results to date are described below:

(1) Effect of Pressure

At 200°C and 1000 psi CO, the generation rate of iron carbonyl from four 24-in.-long steel pipes having a total surface area of 1.932 ft² was too high to be measured by the spectrometer; however, measurements up to 160 psi carbon monoxide were within the range of the spectrometer and can be described by Eq. (2):

$$\dot{r} = 0.0325 P_{CO}^{1.67}$$

where $\dot{r}$ = generation rate of $Fe(CO)_5$ at 200°C (g/ft²/yr)

P_{CO} = pressure of carbon monoxide (psi).

This result compares favorably with a pressure dependency of $P_{CO}^{1.76}$ found for iron at 200°C and 2200 psi carbon monoxide (ref. 2).

(2) Effect of Gas Composition

The generation rate of iron carbonyl in 100 psi of mixed gases, where $P_{CO} = 171$ psi, is $20.7 \text{ g/ft}^2/\text{yr}$ at 200°C . A value of $174.7 \text{ g/ft}^2/\text{yr}$ is the calculated generation rate for pure carbon monoxide at 171 psi using Eq. (1). These results indicate that the other gases in the mixture retard the reaction rate.

(3) Effect of Temperature

The generation rate of iron carbonyl in the mixed gases at 1000°C is $20.7 \text{ g/ft}^2/\text{yr}$ at 200°C , but is in excess of $27.9 \text{ g/ft}^2/\text{yr}$ at 177°C . Thus, the temperature of the maximum generation rate of iron carbonyl from the 1/2-Mo steel appears to be less than the 200°C reported for iron (ref. 2) in carbon monoxide.

(4) Effect of Gas Velocity

Table 3.1 shows the effect of the gas velocity of the iron carbonyl concentration and the generation rate at 200°C in the mixed gases of 1000 psi. A plot of these data shows that the maximum generation rate is obtained at velocities greater than 10-12 cm/min. The data are in agreement with those reported in ref. 2.

3.4 Failure Analysis of Materials and Components

Carbon steel combustor tubes from an oil-coal slurry-fired 100 HP boiler operated at PERC were examined by metallographic and chemical analytical techniques. The nature and amounts of oxides formed were reported to PERC within three weeks and material was supplied for a presentation by PERC personnel. The final report to ERDA-FE and NBS will be available on November 12, 1976.

The tubes did not fail in service.

3.5 Prestressed Concrete Pressure Vessel Studies

The objective is to investigate the potential use of prestressed concrete pressure vessels (PCPV) for coal conversion processes, to identify major problem areas, and to define and outline a test program (or programs) for feature and concept demonstration. Conceptual designs of pressure vessel and liner combinations for commercial size systems are to be developed and studied as vehicles for assessment and guidance.

Emphasis during this month was again on thermal analyses since these are pacing the remaining activities. Also addressed were details of the HYGAS vessel design, and outlines of test programs for feature examination and feasibility demonstration were drafted.

Table 3.1. The Effect of Gas Velocity on the Generation Rate of Iron Carbonyl from a 1/2-Mo Steel at 200°C

Gas velocity ^a (cm/min)	Fe(CO) ₅ concentration (ppb by vol)	Fe(CO) ₅ generation rate (g/ft ² /yr)
2.66	685	7.24
3.70	705	10.35
4.73	657	12.34
7.25	572	16.44
9.88	506	19.83
12.34	467	22.17
15.14	345	20.70

^a17 CO-51 H₂-30 CH₄-2 CO₂ (% by vol) at 1000 psi.

Under task 1, conceptual design layouts for the HYGAS vessel are being prepared. For both the HYGAS and the Synthane vessel, the work essentially has been carried as far as possible without definitive information from the heat transfer and the materials investigations.

Under task 4, heat transfer studies were completed for the cold-liner concept. In these studies, the temperature at the inner surface of the inner hard-face refractory was taken to be 1255 K (1800°F), the nominal temperature for the liner was 339 K (150°F), the maximum concrete temperature was 366 K (200°F), and the ambient temperature for the vessel was 239 K (-30°F). Under these conditions, it was shown that 0.025 m (1 in.) square tubes attached to the liner on 0.18 m (7 in.) spacings provide adequate circuits for cooling. It was further shown that the concrete and liner temperature could be maintained at acceptable off-design conditions when coolant circuit failure results in an effective spacing of 0.36 m (14 in.). For this off-design case, it was further assumed that a 0.0016 m (0.0625 in.) annular air gap due to concrete shrinkage existed around each tube.

Progress is being made in the analysis of a corresponding hot-liner concept, but a number of iterations remain before the results needed are obtained. Refractory studies were temporarily discontinued pending completion of the thermal analyses.

Under tasks 5 and 6, areas where additional work is needed are being identified and test program definitions are being prepared.

3.6 Other Related Work

R. H. Cooper, Jr., and J. H. DeVan

3.6.1 Fluidized Bed Materials Support Activities

No report.

3.7 References for Sect. 3

1. Coal Technology Program Annual Interim Report for Fiscal Year Ending June 30, 1976, ORNL-5208 (October 1976).
2. J. Brynestad, Iron and Nickel Carbonyl Formation in Steel Pipes and Its Preventions - Literature Survey, ORNL/TM-5499 (September 1976).

4. ALKALI METAL VAPOR TOPPING CYCLES

R. S. Holcomb and G. Samuels, Jr.

4.1 Potassium Vapor Cycle

R. S. Holcomb

4.1.1 Design

Nine drawings were released for construction including one of the total assembly. The others covered the gas separator, the vapor line from the boiler to the condenser, and the condenser air outlet assembly and details.

Design work is proceeding on the piping, the sampler box, the vapor separator modifications, and the modifications of the tower. Design of heaters, thermocouples, potassium level elements, and other electrical and instrumentation is getting started.

4.1.2 Fabrication

Work on the fabrication of the inlet duct and enclosure of the condenser was completed and insulation of these has started. Fabrication of the condenser outlet duct, the gas separator, and the potassium piping between the gas separator and the condenser is in progress.

4.1.3 System Operation

Water tests to determine the argon nucleator flow rates in the various boiler tubes continued using the equipment described in the last monthly report. The collection rate increases during heatup and reaches a maximum just before full boiling occurs. The nucleator flow rate at this point appears satisfactory for all the tubes checked. However, it was expected that the increased circulation rate during boiling would cause a more uniform distribution of the argon.

To investigate this further, an existing single tube electrical heated boiler was reactivated. Using the same sampling equipment and with a known argon nucleator flow rate through the single tube, the collection rate curve was found to be very similar to the curves from the large boiler. It was noted that the flow rate from the sampler pump decreased considerably when boiling started due to a vapor lock. This coupled with the increased water circulation rate accounts for at least part of the decreased gas collection rate. Additional analysis of the data is being done and other tests may be needed.

Near the end of the period the steam sampler was installed and tests are in progress to determine the relative flow rates through each tube at various power levels.

A short run was made at a power level of about 4×10^6 Btu/hr.

4.2 Coal-Fired Alkali Metal

G. Samuels, Jr.

Work on the coal-fired alkali metal program was started this month. The initial effort has been concentrated in reviewing available data on the physical properties of potassium and cesium, and in preparing several concepts for the headering and manifold arrangement of a vertical tube boiler.

The thermodynamic properties which will be used are those published by Ewing et al., in NRL Reports 6233 and 6246. However, the data reported do not cover the lower temperatures needed for our current work, and it was necessary to prepare a computer program to extend the range to lower temperatures of interest. Other physical properties data and heat transfer coefficients are also being compiled to be used as a project data book. This work should be completed during the first half of November.

Initial work on the furnace-boiler design is being directed toward a furnace with a diameter of about 12 ft. The boiler concept uses 18 modules located on a hexagonal pattern. Several sketches were prepared showing header and manifold arrangements.

5. CRITICAL COMPONENTS TEST FACILITY

R. E. MacPherson

During the month of October, the reports on the feasibility studies of the CCTF and VTM were completed. Those reports are X-OE-25 entitled "Feasibility Study for Critical Components Test Facility," and X-OE-26 entitled "Feasibility Study for Coal Process Valve Testing." Both reports are presently marked "Preliminary" and will be released following internal and ERDA-ORO reviews.

ORNL is currently continuing to investigate the detailed component testing module requirements in the CCTF. A 4- to 5-month preliminary conceptual design study is being scoped to develop module requirements on a "nonsite-specific" basis. Information is also being generated to aid ERDA-FE in a site selection process for the CCTF.

During the month a meeting was held in Tonawanda, New York, with P. R. Blackburn, who is the Linde associate project manager for Coal Conversion Technology, to discuss the Coalcon valve test requirements and explore other equipment test requirements. Mr. Blackburn is responsible for their lockhopper valve testing program. Linde has designed a valve test facility consisting of three identical test loops which will permit testing three valves concurrently. The Linde test conditions are different from those for the ORNL VTM, the major difference being that the Linde test facility will allow only about 550 lb of coal flow per valve operating cycle vs 14,000 lb per cycle in the ORNL VTM. The Linde test loops dump the coal charge at atmospheric pressure, whereas the ORNL VTM can dump at any pressure up to 600 psig.

They plan to have their test facility in operation by mid-1977; however, Mr. Blackburn thinks the schedule may slip by 6 months. This facility will be used for testing only Coalcon valves, and they have no interest in testing any other valves. They plan to complete all valve testing by the middle of 1979.

6. ENGINEERING STUDIES AND TECHNICAL SUPPORT

J. R. McWherter

6.1 Process Modeling Support

R. Salmon, D. S. Joy, and J. K. Huffstetler

Assistance in obtaining information on coal conversion subjects is being provided as necessary.

6.2 Process Research Digest

F. J. Endelman

Dr. Suman P. N. Singh has been assigned to write the Digest article on flash hydrolysis. This report will cover the ERDA contracts with the Brookhaven National Laboratory, the City College of the City University of New York, and the Institute of Gas Technology for work in this area. The substitution of the IGT work for that of the Rocketdyne Division of Rockwell International was directed by ERDA on October 18.

Carbon copies of letters of introduction to ORNL from the ERDA project officers to these contractors have been received. We now are proceeding to establish direct communication with them and the contractors of the other projects to be reported upon in the first issue of the Digest.

6.3 Survey of Industrial Equipment Capabilities

V. A. DeCarlo

This is a new study which is to be accomplished during FY 1977. The work is being conducted for the ERDA Fossil Demonstration Plants Division under ORNL 189 No. 00077 (FE Control No. 7181). A work statement has been prepared and is being reviewed by ERDA/FDP which divides the study into four tasks as follows:

- (1) Rotating components
- (2) Valves and other pressure let-down devices
- (3) Hot gas clean-up
- (4) Heat recovery equipment.

The main objective of the study is to determine the ability of industry to meet the requirements of future demonstration and commercial scale coal conversion plants in these four areas.

Work has begun on two of the tasks, rotating components and hot gas clean-up. Operating processes are being reviewed for several proposed plants such as Synthoil, COED, Coalcon, and Clean Boiler Fuels from Coal to identify problem areas. Next month work will begin on the other two tasks, valves and other pressure let-down devices and heat recovery equipment.

7. PROCESS AND PROGRAM ASSISTANCE

J. R. McWherter

Process and program analysis studies are being conducted for the ERDA Fossil Energy Office of Program Planning and Analysis. This effort includes research studies on most of the coal conversion and utilization processes. The program objective is to provide, on a consistent basis, technical and economic evaluations of competing processes and systems for coal conversion and utilization.

7.1 Low-Btu Coal Gasification

J. P. Belk, H. F. Hartman, and D. E. Reagan

An initial survey of low- and intermediate-Btu coal gasification processes was completed during the month. Brief summaries of the processes were prepared and contained descriptive and background information. The summaries will be used to select processes for more detailed data collection and evaluation. An outline was prepared of the work plan and the probable schedule. Material containing background and status information was prepared for an October 29 meeting with ERDA-Fossil Energy-Program Planning and Analysis personnel where the subprogram activities and plans were discussed. A proposed common economic basis was reviewed and revised for possible use in the low-Btu study. Background material was reviewed and transmitted to personnel involved in: (1) coal basis selection, (2) coal transportation studies, and (3) environmental aspects review.

7.2 Direct Combustion

H. I. Bowers, R. L. Sinard, and I. Spiewak

During October the Work Statement was revised again after discussions with Headquarters personnel.

7.3 Liquefaction

A subcontract for this work is being negotiated.

7.4 High-Btu Gas

A subcontract for this project is planned.

7.5 In Situ Coal Gasification

W. C. Ulrich and R. C. Forrester III

The survey of literature pertaining to the various concepts and processes proposed for in situ coal gasification continued. A visit was made to the Morgantown Energy Research Center on October 15, 1976, to discuss the status of the Longwall Generator concept with members of the staff.

A statement of work for the evaluation project was submitted to the Office of Program Planning and Analysis (OPPA) of ERDA/FE for review.

A meeting with representatives of OPPA was held at ORNL on October 29. The in situ evaluation project plan was presented and discussed, and the statement of work was further reviewed.

7.6 Beneficiation

G. R. Peterson and Suman Singh

A detailed beneficiation flowsheet, material balance, and equipment list were prepared for the Battelle hydrothermal process based on a feed rate of 13,000 TPD of Lower Kittanning bed Pennsylvania coal. It may become necessary to redevelop these data for feed rates up to 35,000 TPD.

Beneficiation studies of the Pennsylvania coal via mechanical beneficiation are underway. The washability data have been reviewed and washability curves developed. To explore the effects of the degree of beneficiation on costs of beneficiation, two washing procedures are planned. One method will provide a wet cleaning of the coarse coal (5 in. x 1/4 in.), sizing of the clean coal, and subsequent recombination with the fine coal (1/4 x 0). This system should have the lowest cost and remove a significant amount of ash and sulfur from the Pennsylvania coal selected for the evaluation. The second cleaning process planned will provide a "deep cleaning" of the coal. The cleaning system will include heavy medium separation and froth flotation.

Progress is being made in establishing a data base for the various coal beneficiation methods that have been proposed or currently being developed.

Several processes have been identified for the chemical desulfurization of coal. These processes are the following:

- (1) Meyers or TRW process
- (2) Ledgemont Pyrite Removal process
- (3) PERC processes for chemically cleaning coal
- (4) SURC Chemical Comminution process.

In addition, the following coal beneficiation processes are also being evaluated as a part of the above study:

- (1) High Intensity Magnetic Separation process
- (2) Bacterial Leaching of Pyritic Sulfur process
- (3) Ilok process for desulfurizing coal.

State-of-the-art description of the above processes has been completed. A technical evaluation of the applicability of the processes for the beneficiation of coal is presently being performed.

7.7 Gas Cleanup

M. S. Edwards

Two trips were made to investigate high-temperature gas cleanup concepts. The first, to Morgantown Energy Research Center, involved a meeting with E. C. Oldaker, the Manager of MERC's high-temperature gas cleanup studies. Aspects of design and commercialization of MERC's iron oxide/flyash process were discussed. The second trip was to the National Coal Association's meeting in Louisville, Kentucky, to attend a paper presented by Carl Jones of Stone & Webster on the comparison of high-temperature and low-temperature gas cleanup systems in combined cycle applications. Stone & Webster has just finished the comparison under contract from Electric Power Research Institute, but the report itself will not be available for several months.

The program manager of the gas cleanup study at ERDA/FE, B. Baratz, visited ORNL at the end of October. The work statement submitted previously for the gas cleanup study was approved with no revisions.

Additional discussions were undertaken with the investigators of various coal conversion routes, principally in situ gasification, low-Btu gasification, and advanced power systems investigators. The interface between the gas cleanup study and in situ gasification was thoroughly defined. More discussion is still required to adequately define the interface with the other programs.

7.8 Coal Prices and Volumes

O. L. Culberson and H. F. Soard

Data for 168 contract purchases of eastern U. S. coals reported in Coal Week¹ for the month of June 1976 have been recorded. Each such transaction is described by mine of coal origin, consumer destination, quantity, higher heating value (HHV), sulfur and ash contents, and delivered price. Mr. H. F. Soard has just joined this project, and is currently determining distances between the coal origins and destinations in order that the coal price may be reduced to a minemouth basis. When this has been accomplished, a multiple regression analysis will be performed to express minemouth coal price as a function of HHV, contract quantity, and sulfur and ash contents. Following completion of this task, a similar analysis will be performed on western U. S. coals.

¹Anon., Coal Week, 2 (35), 6 (1976); 2 (36), 8 (1976); 2 (38), 8 (1976)

7.9 Energy Transportation Costs

O. L. Culberson

Published data from Coal Week² on the cost of shipping coal by single rail cars were correlated by least-squares fit to obtain the expression:

$$y = \frac{23.07x}{4 + x}$$

where:

y = transportation cost, \$/ton

x = distance, 100's of miles; $0.85 \leq x \leq 12$

Cost of shipment by rail unit train had previously been determined to be:

$$y = \frac{27.64x}{11 + x}$$

Distances were estimated using highway mileages from our seven coal fields to our four non-minemouth points of consumption. These distances with the above expressions led to coal shipping costs as follows:

²Anon., Coal Week, 2 (37), 3 (1976)

From	\$ / ton to							
	Wilmington, DE		Chicago, IL		Houston, TX		San Fran., CA	
	Small Plant	Large Plant	Small Plant	Large Plant	Small Plant	Large Plant	Small Plant	Large Plant
Western WV	12.6	8.5	12.8	8.7	17.2	14.2	20.0	19.4
Northern CO	19.0	17.3	17.2	14.2	17.2	14.2	16.8	13.6
Western PA	9.7	5.9	12.2	8.0	17.8	15.2	20.0	19.4
Western KY	15.2	11.4	10.2	6.3	15.7	12.1	19.6	18.7
Central IL	15.5	11.8	9.2	5.4	15.5	11.8	19.5	18.3
Wyoming	19.0	17.2	17.0	13.8	17.5	14.7	16.7	13.4
N. Dakota	18.6	16.6	16.1	12.7	18.1	15.8	18.2	16.0

The costs of transporting low-Btu and high-Btu gas will be developed next.

8. ENVIRONMENTAL ASSESSMENT OF COAL CONVERSION TECHNOLOGY

C. R. Boston

Preliminary drafts of environmental requirements for RFP's were delivered to ERDA/FE-Division of Fossil Demonstration Plants. Further revisions are underway to accommodate FDP's changing needs. Comments on a Preliminary Draft Environmental Impact Statement for the Coal RD&D Program were also delivered to ERDA. The longer-term activities in this project are in the startup phases. Staffing is now approximately 80% complete.

9. COAL-FUELED MIUS

R. S. Holcomb 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. IAA-H-40-72 and by the Energy Research and Development Administration, Office of Fossil Energy (formerly Office of Coal Research, Department of the Interior), under ERDA Contract No. E(49-18)-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.

9.1 Program Planning

The program is currently undergoing extensive review with respect to additional market potential in the commercial/industrial sector. A redirection towards a broader range of applications and possible scale-up of unit size to the range of 5 to 20 MWe is under consideration. The test unit under design, however, is expected to retain the same basic features as originally planned.

9.2 Furnace Procurement

The two bids that were received for the furnace have been allowed to expire and the order will not be placed. It is now planned to prepare performance specifications and request furnace manufacturing firms to submit designs to the specifications. Procedures will be established for evaluation of the designs and selection of the vendor to supply the

furnace. The specifications will be prepared and potential vendors will be contacted as soon as possible after additional funds are received for the redirected program.

9.3 Instrumentation and Controls

Computer simulator studies of system control are being conducted. The instrument application diagram of the system was issued for review.

9.4 Coal Metering and Feed System

Installation of the endurance test system was completed except for the solids level instruments on the feed tank. The test should be ready for startup in November 1976.

9.5 Other Systems

Design work on other systems of the experimental unit has been halted temporarily until the new furnace design has been selected from those to be submitted by vendors. The design of the other systems can then be completed taking into account the effects of the new furnace design.

9.6 Supplemental Studies

The corrosion test was begun on October 25 and will require a minimum of 21 days for completion.

Writing of the report on the heat transfer test is about halfway completed.

10. ANALYTICAL CHEMISTRY

W. R. Laing

No report this month.

11. CHEMICAL RESEARCH

L. M. Ferris

This work is reported quarterly.

12. LIFE SCIENCES

C. W. Gehrs

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