ORNL/TM-5674

Coal Technology Program
Progress Report for September 1976

OAK RIDGE NATIONAL LABORATORY
OPERATED BY UNION CARBIDE CORPORATION FOR THE ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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PROGRESS REPORT FOR SEPTEMBER 1976

Date Published: November 1976

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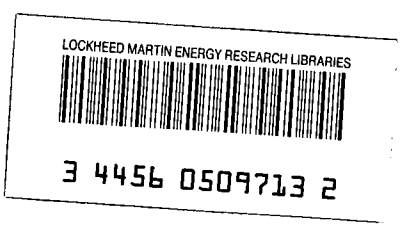

PREVIOUS REPORTS IN THIS SERIES

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ORNL/TM-5045, Progress Report for September 1974
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COAL TECHNOLOGY PROGRAM PROGRESS REPORT FOR SEPTEMBER 1976

ABSTRACT

This report - the twenty-sixth 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 hydocarbonization research, separations technology, in situ gasification, coal-solvent-hydrogen mixing, materials engineering, alkali metal topping cycles, critical components testing facility, engineering studies and technical support, process and program assistance, environmental impact studies, analytical chemistry, and coal-fueled MIUS.

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1. SUMMARY

J. P. Nichols

Highlights of our progress in September are as follows:

- In our hydrocarbonization research project the solids handling facility was sufficiently completed to permit shakedown and preparation of approximately 100 lb of coal for run HC-8. Equipment failures hindered two attempts to start experiment HC-8. Trace element concentrations in products from run HC-2 were measured.

- In our separations technology work, bench-scale filtrations using coal and char precoats were compared to standard filtrations using diatomaceous earth precoats. In both cases the filtration rates and filtrate clarities were comparable to standard values; thus, considerable economic savings are possible due to the lower cost of coal (factor of 10 lower), the process savings due to substitution of a material readily available to the process, and subsequent recovery of the energy value of the coal or char precoat by gasification. The demonstrated use of the char removes doubt about temperature limitations of carbonaceous precoats.

- In our coal block pyrolysis studies, a two-dimensional pyrolysis gas production mechanism, assumed to explain data obtained with as-received coal samples, was tested by utilizing a specimen which was dried at 125°C under vacuum prior to pyrolysis.

- A new project, entitled Coal-Solvent-Hydrogen Mixing, is being undertaken to determine if a highly turbulent plug flow reactor of novel design will allow adequate hydrodesulfurization and hydroliquefaction of coal/donor solvent slurries without use of hydrotreating catalysts.

A series of venturis is being considered as a candidate contactor and a literature search for operating characteristics of venturis with multiple flowing phases was conducted. Limited information dealing with gas-liquid and solid-liquid flows was obtained. Installation was started of a facility to study flow through model contactors using coal-water-air mixtures to simulate coal-solvent-hydrogen mixtures.

o In our Materials Engineering work, we continued to make suggested revisions of our document that assesses piping and pressure vessels for coal conversion systems, began experimental determinations of the rate of carbonyl formation in 1/2% Mo--steel pipe, shipped surveillance specimens for use in a test of fireside corrosion in a fluidized-bed combustor, and continued design studies of prestressed concrete pressure vessels for coal gasifiers.

o In our project for development of an alkali metal vapor topping cycle, we continued tests of the gas fired boiler with water, and continued, approximately on schedule, with the design and fabrication of components that will be required for tests with potassium.

o At the request of the Division of Fossil Demonstration Plants, we completed initial feasibility studies of modules for testing of full-sized lock hopper valves and a Critical Components Test Facility (CCTF). The CCTF suggested by ORNL would provide all of the facilities required to support the operation of modules for testing components such as valves and valve operators, coal feeders, slurry pumps, other rotating machinery, and coal beneficiation equipment.

o Technical support for ERDA/FE included continued assistance on process modeling of coal conversion processes and on the Process Research Digest to be published for ERDA/DFER. A work schedule and production budget were prepared for the Digest and will be transmitted to ERDA/DFER.

o The process and program analysis of coal conversion processes for the Fossil Office of Program Planning and Analysis is continuing. The low-Btu coal gasification processes are being surveyed in preparation for selecting certain ones for detailed evaluations. Revised work statements for program analysis were sent to ERDA/FE/OPPA for Low-Btu Gas, Direct Combustion, and other subprograms. A survey of the literature on in situ coal gasification was started. Preliminary material balances have been prepared for beneficiation of Pennsylvania coal by mechanical separation, by the Battelle process (BHCP), and by the TRW process (ferrous sulfate). An extensive data base for the design and economic considerations of gas cleanup processes was compiled. A study of the transportation of coal between specified origins and destinations was initiated. The effect of transportation and sulfur content are being considered in the study of the price of coal.

- We began a new project to assist the Division of Fossil Demonstration Plants with environmental assessment of coal conversion technology by reviewing two documents at the request of FDP, initiating studies related to the landfill disposal of solid wastes, and beginning the preparation of an Environmental Monitoring Handbook and Programmatic Impact Statements.

- Our work in Analytical Chemistry included continued fractionation of fossil-derived oils for skin painting tests and continued chemical characterization studies of fossil-derived materials.

2. HYDROCARBONIZATION RESEARCH

H. D. Cochran, Jr.

2.1 Bench Scale Hydrocarbonization

H. D. Cochran, Jr., C. H. Brown, Jr., P. R. Westmoreland, J. C. Rose, and R. L. Andrews

2.1.1 Work Accomplished

The solids handling facility was sufficiently completed to permit shakedown and coal preparation. All machinery shown in the floor plan of the facility (August monthly report, ORNL/TM-5654) was installed except for the Mikro Pulverizer hammer mill, which was not received. Modification of the bench-scale system heating system and placement of new control thermocouples were completed, permitting operation at high temperature with better control.

Approximately 100 lb of coal, dried to 10% moisture and sized at -50+140 mesh, was produced for HC-8 from 505 lb of coal containing approximately 27% moisture. Chunks of coal from a sealed drum were vacuum-dried in batches at 50°C. Particle size reduction was accomplished in two steps, coarse grinding in the large-capacity hammer mill and final reduction in a small roller mill. Similarly, size classification was accomplished in a two-step semi-batch operation. After oversize coal was skimmed off in the Sweco sieve shaker for reprocessing in the roller mill, fines were removed in two continuous Sonic-Sifters. Yields and operation will be improved by installation of the Mikro Pulverizer hammer mill, which will perform the second size reduction step more efficiently.

Equipment failures hindered two attempts at the beginning of experiment HC-8, but they were corrected for resumption of experiments in early October.

Trace element analyses of HC-2 products were provided by the Analytical Chemistry Division. Since no data existed on distribution of trace elements among products of any hydrocarbonization processes, a paper detailing the results is in preparation so that this information may be rapidly disseminated. Tables 2.1 and 2.2 show both the analytical data and the calculated concentrations in oil, assuming that ash measured in the oil exists in char. Judging by Al, Si, and Mg content in the water, char is also present in the water in small amounts; however, no correction for char content was made.

2.1.2 Work Forecast

The Mikro-Pulverizer hammer mill should be received and installed during October.

Routine experimentation should resume in October. A continuing problem of seal leakage in the scrubber water recirculation pump will be eliminated when parts arrive.

Table 2.1 Quantitative trace element analyses of experiment HC-2 products (precision = ± 20%)

Element	Trace element concentration, ppm or μg/g				
	Coal	Char	Scrubber water	Oil with char carryover	Char-free oil[a]
Ba	510.	1570.	0.4	10.	5.
Cd	0.2	0.9	<1.	0.1	0.1
Cr	7.	18.	0.1	3.	2.9
Cn	9.	17.	0.06	2.5	2.4
Fe	1700.	3800.	1.	95.	83.
In	0.2	0.7	<1.	0.1	0.1
Mo	0.2	1.1	0.01	0.1	0.1
Ni	5.	10.	1.	7.	7.
Pb	3.	7.	0.003	0.2	0.18
Sr	320.	490.	0.1	4.	1.6
Tl	0.3	0.4	0.02	0.1	0.1
Zn	6.	14.	0.06	1.5	1.45

[a]Computed from ash balance, assuming all of 0.039% ash in oil exists in char.

Table 2.2 Semiquantitative trace element analyses of HC-2 products (precision = ± 50%)

Element	Trace element concentration, ppm or μg/g				
	Coal	Char	Scrubber water	Oil with char carryover	Char-free oil[a]
Al	20000.	60000.	1.	160.	<30.
As	3.	8.	0.004	0.2	0.2
Au	<1.	<1.	<0.01	<0.1	<0.1
B	880.	2000.	0.5	2.	<<2.
Br	6.	16.	0.02	0.1	0.05
Ce	8.	16.	0.001	0.1	0.05
Co	1.	3.	0.03	0.2	0.2
Cs	<1.	<1.	<0.01	<0.1	<0.1
Ga	9.	25.	0.008	0.1	0.02
Ge	1.	3.	<0.01	<0.1	<0.1
Hf	<1.	<1.	<0.01	<0.1	<0.1
Hg	<1.	<1.	<0.01	<0.1	<0.1
Ir	1.	3.	<1.	<1.	<1.
K	40.	70.	0.1	0.3	0.07
La	16.	31.	<0.001	0.04	<<0.04
Mg	5000.	15000.	7.	200.	150.
Mn	10.	30.	0.01	1.	0.9
Na	120.	360.	0.2	6.	5.
Nb	3.	6.	0.01	1.	1.
Os	<1.	<1.	<0.01	<0.1	<0.1
P	200.	500.	0.2	6.	4.
Pd	<1.	<1.	<0.01	<0.1	<0.1
Pt	<1.	<1.	<0.01	<0.1	<0.1
Rb	<1.	<1.	<0.01	<0.1	<0.1
Re	<1.	<1.	<0.01	<0.1	<0.1
Rh	<1.	<1.	<0.01	<0.1	<0.1
Ru	<1.	<1.	<0.01	<0.1	<0.1
Sb	5.	14.	0.001	0.02	<<0.02
S	1000.	800.	6.	100.	100.[b]
Si	5000.	≥10000.	0.5	400.	--

Table 2.2 (Continued)

Element	Trace element concentration, ppm or μg/g				
	Coal	Char	Scrubber water	Oil with char carryover	Char-free oil[a]
Sn	3.	5.	5.	0.4	0.4
Te	1.	3.	<0.01	0.2	0.2
Th	<1.	<1.	<0.01	<0.1	<0.1
Ti	260.	630.	0.1	30.	30.
U	<1.	<1.	<0.01	<0.1	<0.1
V	20.	36.	0.02	0.07	<<0.07
Y	3.	10.	<0.001	0.1	0.07
Zr	10.	21.	0.3	7.	7.

[a]Computed from ash balance, assuming all of 0.039% ash in oil exists as char.

[b]Probably zero, since silicon, like aluminum, generally occurs as a refractory oxide which would remain in the char.

2.2 Bench Scale Residue Carbonization

2.2.1 Work Accomplished

There were several design modifications made to the residue carbonizer during September. These modifications are an attempt to reduce the extensive solids carryover and plugging problems which have caused the termination of many runs. The changes include the addition of a 3-ft section with an 8-in.-diam expanded head to the top of the reactor and the addition of a high efficiency internal cyclone which eliminated the 1/2-in. pipe connecting the reactor and the previous cyclone. Another modification made was to replace the flat bottom of the gas scrubber with a conical bottom to facilitate the recovery of liquids. A specially designed plug valve is being fabricated to replace the ball valve feeder in order to improve solids feeding.

Run RC-14 was attempted near the end of the month, but it did not achieve steady operation due to feeding problems and a plug which developed in the internal cyclone. The Wyodak char to residue ratio in the feed was 2/1. This is the lowest char/residue ratio attempted in the carbonizer and may be the cause of the plug which developed in the cyclone.

2.2.2 Work Forecast

The plug valve feeder should be completed and installed during October. Residue carbonization runs with a char/residue feed ratio of 4/1 will be made during the month.

3. SUPPORTING RESEARCH AND DEVELOPMENT IN SEPARATIONS TECHNOLOGY

B. R. Rodgers

3.1 Bench-Scale Filtration Tests

B. R. Rodgers and D. A. McWhirter

Operating procedures and construction details of the 3 in. ID bench-scale filters has been described elsewhere (ORNL-5159). The filtrations described below were carried out at 300°F and employed precoats of 170/200 mesh and 140/170 mesh Illinois #6 coal and char (from the ORNL Hydrocarbonizer), and Johns-Manville's "Fibra-Flo", a mixture of diatomaceous earth and asbestos fibers. Solvent Refined Coal Unfiltered Oil (SRC-UFO) from the Wilsonville SRC plant was used in all cases. The quantity of precoat material was varied from 50 to 100 grams with little effect on filtration rates. Initial filtration rates varied from 10 to 29 gal/hr·ft^2 (93-270 lb/hr·ft^2). These values should be compared to typical SRC Pilot plant values of 50-150 lb/hr·ft^2. These results verify the lab-scale results first reported in October 1975 (ORNL-4968). Additional details will be reported in the Coal Program Quarterly Report for the period ending September 30, 1976.

3.2 Duplication of Tretolite Additive Tests

P. R. Westmoreland and B. R. Rodgers

Laboratory-scale sedimentation tests with and without Tretolite 771-119 additive were carried out in order to confirm or to reject earlier lab-scale tests.[1] Recent bench-scale sedimentation tests have not substantiated these extremely low ash concentrations which earlier tests had indicated would be achievable by settling a Tretolite-UFO mixture. In alternating experiments, SRC-UFO or SRC-UFO with 4700 ppm (vol) of Tretolite 771-119 were settled. Different temperature conditions were used for consecutive paired experiments.

Data from the experiments are presented in Fig. 3.1. At each temperature, addition of Tretolite reduced ash content in the "clarified" upper fractions - 67% reduction in fraction 4 at 230-250°C (48 min), greater than 93% at 300-330°C (48 min), and 69% at 350-390°C (30 min). However, actual ash content in the upper fractions was significantly different for the series of experiments TD1-2 and TD4-7. Probable cause is that two separate samples of UFO were drawn for feed material in the two sets of experiments. UFO may have been insufficiently agitated before feed material was withdrawn. If this was the case, experiments TD4-7 are more accurate, and very low ash concentrations measured in TD1, TD2, and earlier experiments are in error.

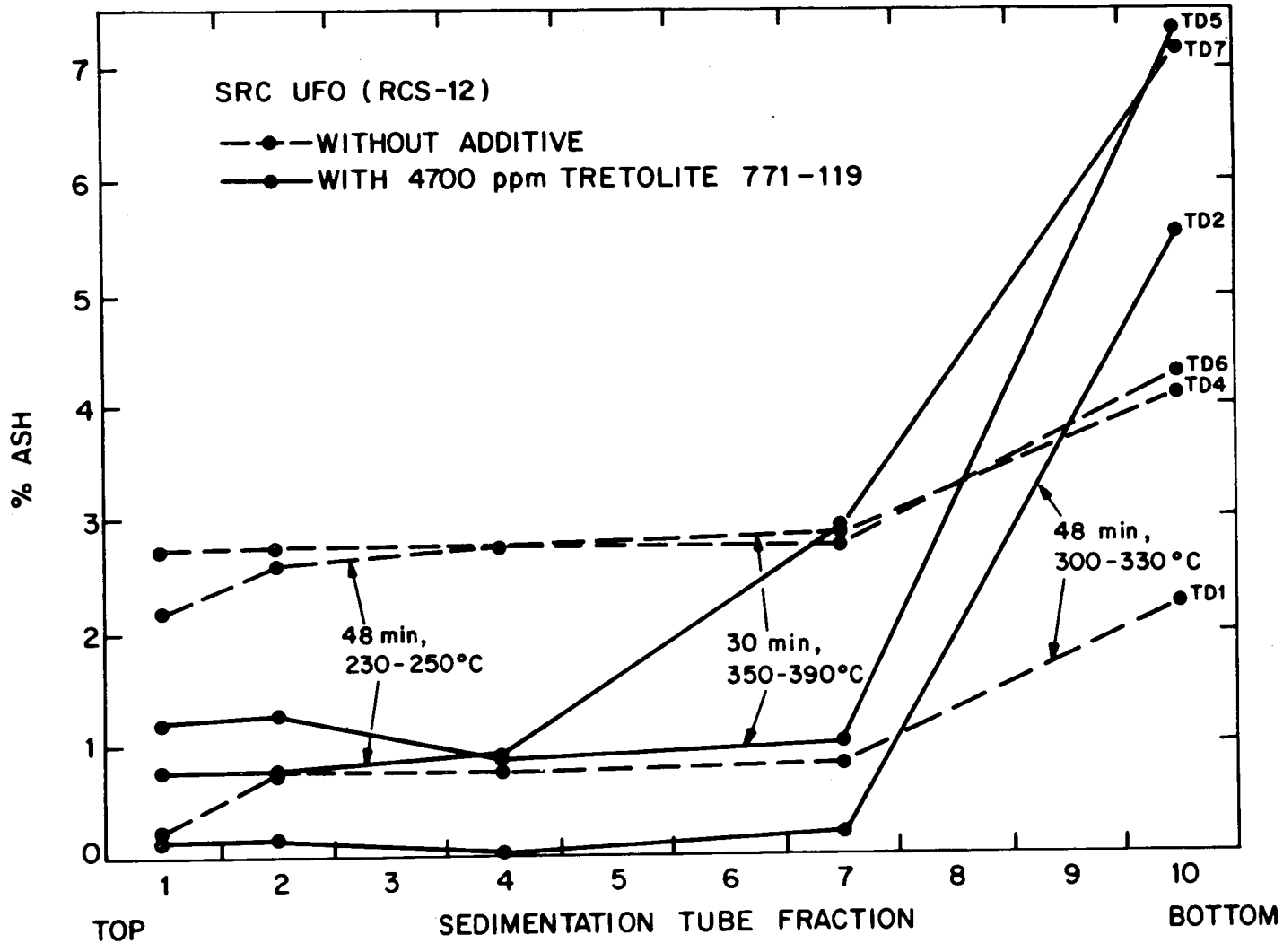


Fig. 3.1. Effect of Tretolite additive on settling of SRC unfiltered oil at different temperatures.

References for Section 3

1. J. P. Nichols (editor), Coal Technology Program Quarterly Progress Report for the Period Ending March 31, 1976, ORNL-5159 (July 1976).

4. EXPERIMENTAL ENGINEERING SUPPORT OF AN *IN SITU* GASIFICATION PROCESS

R. C. Forrester III

4.1 Large-Block Pyrolysis Studies

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

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 this month which involved the thorough drying of a test specimen at 125°C under a vacuum of 68 cm of mercury. Gas production during subsequent pyrolysis 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. Gas samples are being analyzed and a comparison of these results with those obtained from earlier tests will be presented in the next report of this series.

4.2 ERDA's UCG Program

R. C. Forrester III

Technical problems associated with UCG processes, as well as details of the ERDA program for development of *in situ* conversion technology, were described recently at the 11th Intersociety Energy Conversion Engineering Conference which was held at the Sahara Tahoe Hotel, State Line, Nevada, September 12-16, 1976. The paper, entitled "Recovery of Inaccessible Coal Reserves by *In Situ* Gasification," generated a number of questions and seemed to be well-received. Staff members from the Laramie ERC were present and helped to answer inquiries concerning environmental effects of recent field tests.

References for Section 4

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.

5. COAL-SOLVENT-HYDROGEN MIXING

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

Improvements in hydrodesulfurization and hydroliquefaction of coal in hydrogen donor solvent systems can possibly be achieved by improving the design of the reactor in which the hydrogenation is carried out. Results obtained in the development of the Synthoil Process[1,2] at Pittsburgh Energy Research Center (PERC) suggest that under conditions of high turbulence and plug flow in a reactor, adequate desulfurization and liquefaction may be achieved utilizing only the catalytic activity of the ash minerals. 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 hydrotreating 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.

Progress on the first phase (simulated contactor studies) since the start of the project has consisted of a review of the literature for flow characteristics of venturis operating with gas-liquid or liquid-solid two-phase flow and the design and start of installation of a facility to circulate water/coal slurries through a three-phase contactor and to measure pressure drop across and residence time distributions in the contactor.

An extensive literature review was made for two-phase (gas-liquid and solid-liquid) flow characteristics in venturis in which data on pressure drop and gas-liquid mass transfer coefficients were collected. The review indicated that although there is some information in the literature on two-phase flow through venturis which can be used to guide design of experiments, there is no information on longitudinal mixing or dispersion in venturis and there is no information on pressure drop in three-phase flow through venturis. This information is necessary in order to rationally choose a reactor configuration from among several candidates.

Simulated contactor studies will be carried out in a facility for circulating water/coal slurries at rates up to 20 gpm through contactors which are to be tested. A flow diagram of this facility is shown in Fig. 5.1. The facility consists of a stainless steel 55-gal agitated surge tank where coal/water slurries are made up, a slurry pump capable of delivering 20 gpm coal/water or coal/glycerol solution slurries, a means for injecting pulses of KCl tracer, means for measuring KCl concentration at the contactor inlet and outlet, a deionizer to prevent buildup

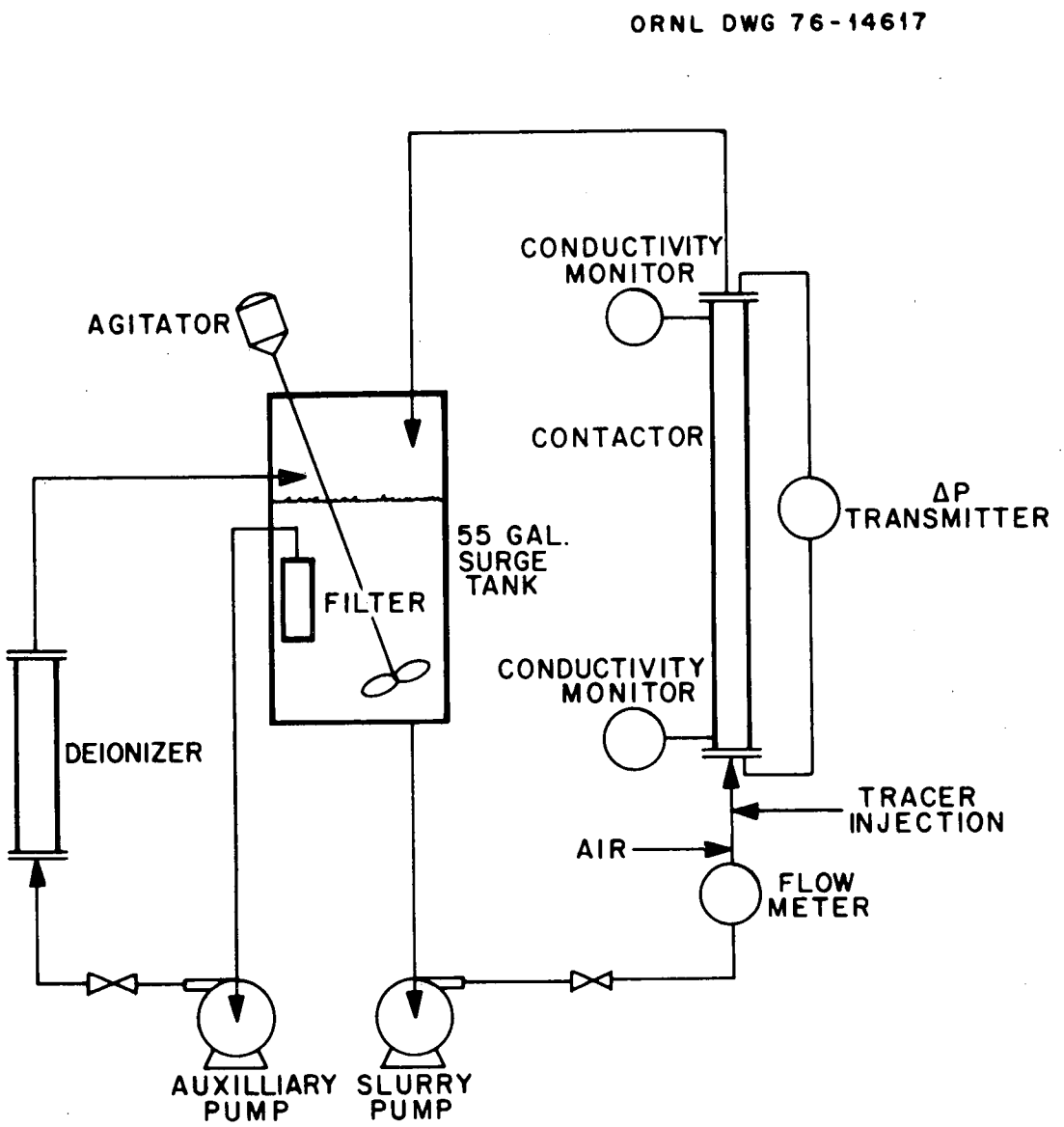


Fig. 5.1. Schematic flow diagram of coal slurry recirculating loop for simulated contactor studies.

of the KCl tracer in the system, and instrumentation for measuring pressure drop across the contactor.

All components of the facility except the deionizer and the pressure drop instrumentation have been installed. The flow meter for measuring slurry flow rate will be a venturi meter. This venturi has been designed and is being fabricated.

References for Section 5

1. I. Wender, Internal Quarterly Technical Progress Report, Oct. - Dec. 1975, PERC/QTR-75/4, January 1976, p. 22.

2. I. Wender, Internal Quarterly Technical Progress Report, Jan. - Mar. 1976, PERC/QTR-76/1, April 1976, p. 48.

6. ALKALI METAL TOPPING CYCLES

A. P. Fraas

6.1 Design

Three drawings were completed and released - two of the potassium drain tank enclosure and one of the condenser air inlet duct and damper. Two drawings are of the argon separator, and one of the potassium drain lines are nearing completion. The condenser outlet air plenum is in the design phase. Drawings of the potassium vapor line, insulation, and shielding are underway, and assembly drawings of the entire system are underway.

A piping flexibility analysis is being made to determine the stress and deflections in the potassium piping system.

The potassium vapor process flow diagram was approved by the project management and will be released next month. Work on the instrument application diagram and tabulation is underway.

Modifications are being made to the structural drawings to provide the necessary changes to accommodate the system revisions.

6.2 System Fabrication

Work was initiated on the condenser enclosure and air ducts and on the potassium fill-and-drain tank. Work was also begun on the potassium vapor piping connection to the potassium condenser.

6.3 System Operation

A method was devised for determining the argon nucleator flow rate to the individual tubes of the boiler. As indicated on the attached sketch water and argon are removed from a low level in a boiler tube and passed through a flask where the argon is separated from the water and the collection rate is measured. The water is returned to the boiler tube so that there is essentially no net disturbance in flow at the entrance or exit of the boiler tube.

Using this setup, tests are being made to assure that there is adequate nucleation in each tube during approach to boiling and during boiling. At the present time, results are very encouraging.

The boiler has been operated at various power levels up to about 2×10^6 Btu/hr.

6.4 Schedule

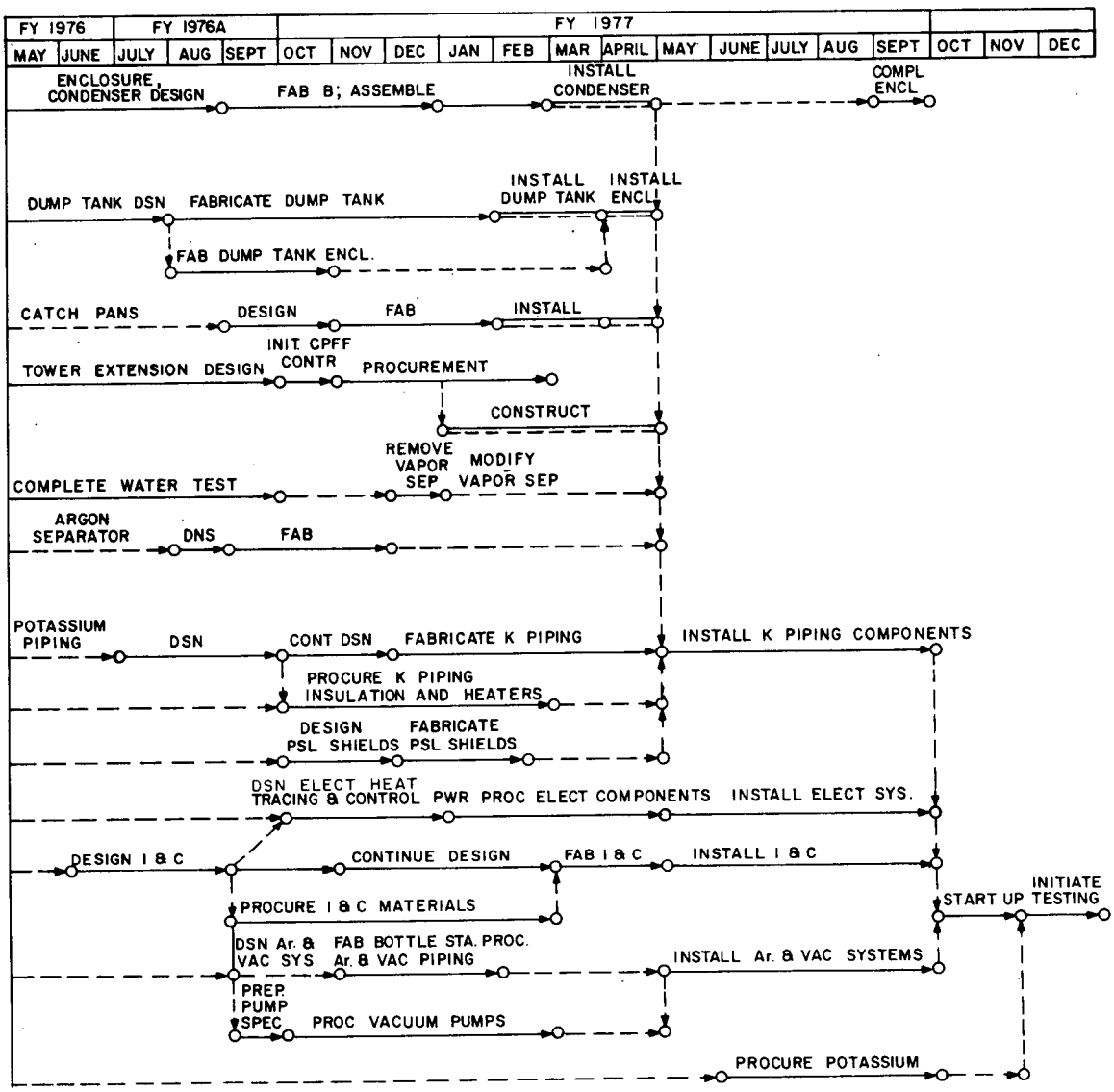
The schedule for design and installation of the potassium test is attached as Table 6.1.

7. LIFE SCIENCES

C. W. Gehrs

This work is reported quarterly.

Table 6.1. Potassium vapor topping cycle schedule for potassium test



8. MATERIALS

R. T. King and D. A. Canonico

The materials engineering and associated technology reported here are in support of activities directed by the Materials and Power Generation Branch of the Division of Fossil Energy Research. Other related work not funded directly by this division of ERDA/FE is included also.

Summary

The review of piping and pressure vessels activity has produced a revised chapter on Material Selection for the Pressure Vessel and Piping Assessment and an article, "Pressure Vessel for Coal Conversion Systems" for the ERDA newsletter.

ORNL was represented at a FER meeting to define responsibilities in Failure Analysis tasks.

Our prepared poster set was exhibited (as planned) at the ORNL Metals and Ceramics Division Information Meeting, September 15–16. We have started fabrication of single-layer specimens including Ni-Cr-Al (75-24-1) alone and mixed with ZrO_2. We have completed a computer study which indicates the feasibility of thermal inspection for unbonds in ZrO_2 coatings. A crack specimen for liquid penetrant testing has been prepared and it has been used for preliminary testing with a filtered-particle product which shows promise. New experiments with electrochemical techniques are in progress; the objective is to identify feasible active-probe techniques. Some new microwave techniques for measuring properties of thin dielectric layers have been found in recent literature; preliminary feasibility tests are being assembled.

Experimental determinations of the rate of carbonyl formation in a 1/2% Mo pipe at 200°C, 1000 psig, containing 30% CH_4, 51% H_2, 17% CO, and 2% CO_2 are in progress.

Conceptual design and thermal analysis on prestressed concrete pressure vessels (PCPV) for Synthane and Hygas vessels continued. Penetration and interval structural elements were considered.

Sixteen surveillance-test tubes have been shipped for use in a Fluidyne Engineering Corporation fluidized bed test facility, including Alloy 600, Alloy 800, aluminized Alloy 800, and types 310, aluminized 310, 304, and 316 stainless steels.

8.1 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 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 Selection Chapter has been submitted to Fossil Energy Research for their review. An article, "Pressure Vessel for Coal Conversion Systems," based on the assessment has been prepared for use in the *Materials and Components in Fossil Energy Applications*, ERDA Newsletter.

8.2 Inspection Techniques for Wear- and Process-Resistant Coatings

R. W. McClung and G. W. Scott

8.2.1 Review and Evaluation (G. W. Scott)

Reviews of recent electronic literature turned up some new microwave techniques which have been applied to the nondestructive measurement of dielectric properties and flaw detection in ceramic circuit substrate materials. These techniques are being carefully examined for potential application or adaption to inspection of ceramic coatings.

The poster set prepared for the ORNL Metals and Ceramics Information Meeting was displayed on September 15 (one-half day) and 16 (all day). The posters and mountings have been retained for future use. They sketch the objectives of the program, list materials and coating structures, list some uses of NDT and some NDT methods, and show preliminary data on x-ray fluorescence and liquid penetrant tests.

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

The Welding and Brazing Laboratory is preparing plasma sprayed coatings to be used for development of nondestructive examination (NDE) techniques. We have sprayed ZrO_2 and Ni-Cr-Al (75-24-1) coatings on Incoloy 800 substrates. During this reporting period the following samples have been prepared.

(a) Immersible electrolytic specimen, with 0.254-mm (0.010-in.) thick coatings on all sides and edges of Incoloy 800 strips.

(b) Free standing films of Ni-Cr-Al, thicknesses varying from 0.076-mm (0.0003-in.) to 0.508-mm (0.020-in.), for eddy current evaluation.

(c) Free standing films of 50% ZrO_2/50% Ni-Cr-Al, thicknesses of 0.254-mm (0.010-in.) and 0.508-mm (0.020-in.), for eddy current evaluation.

(d) Plate specimen, with 0.076-mm (0.0003-in.) and 0.152-mm (0.006-in.) thick coatings of Ni-Cr-Al on 3.175-mm (0.125-in.) thick Incoloy 800.

(e) Plate specimen, with 0.254-mm (0.010-in.) and 0.508-mm (0.020-in.) thick coatings of 50% ZrO_2/50% Ni-Cr-Al on 3.175-mm (0.125-in.) Incoloy 800.

Free standing films of the Ni-Cr-Al materials were obtained by spraying directly onto a polished Al_2O_3 surface from which the coating easily spalls away.

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

We have completed an initial computer study of the propagation of thermal waves in zirconium oxide coatings. The results are highly encouraging and indicate that practical bond integrity evaluation can probably be performed using thermal techniques. For example, an unbonded region having a diameter of 0.100 in. (2.54 mm) will provide a temperature differential with respect to a bonded region in excess of 11°C, assuming a heat injection rate of 1.0 BTU/in.^{2}·sec (39.1 cal/cm^2·sec). Moreover, with continuous heating this differential will be reached at about 0.05 sec, and will decrease very slowly, falling to about 8°C at 0.5 sec. While such an extended heating period is not particularly desirable, the indication is that for heating times of the order of 0.1 sec, a low-mass fast-response thermistor could be used as a detector. We have such a thermistor and are investigating the possibility of incorporating it into a thermal probe.

8.2.4 Liquid Penetration Testing (S. N. Snyder)

In our last monthly report we stated that the first artificial-crack specimen coated with ZrO_2 would be stressed to 3500 lb in an attempt to produce cracks in the coating for liquid penetrant experiments. This was done, but no cracks were produced. The specimen was stressed further to a load of 4000 lb with cracks appearing visually at a load of 3850 lb. The cracks that could be seen visually were widest at the edges of the specimen and narrowed as they extended into the specimen. A microscopic examination of the specimen at 100× magnification showed that cracks had been formed across the entire width of the specimen, but none was continuous from edge to edge. We have photographed the cracked area at 40× magnification.

We have deviated slightly from our pursuit of optimizing the liquid penetrant test procedure to make a "quick and dirty" test with fluorescent filtered particles. This technique makes use of a liquid dispersion of properly sized and shaped particles which, when applied to a porous material, will be absorbed in proportion to its surface area. More fluid is absorbed at a defect than anywhere else due to the extra absorption area of the sidewalls within the defect, and the absorption is so pronounced that the suspended particles are drawn to the site of the defect. Being wider than the dimensions of the top edges of the crack, the particles filter out to form an indication.

This first attempt with the Magnaflux Corporation filtered particle product, PARTEK, shows more promise for crack detection without extreme background interference in the porous ZrO_2 coating than does the fluorescent post-emulsified liquid penetrant. Also, the filtered particle test procedure is much simpler and quicker than the liquid penetration test procedure.

8.2.5 Electrochemical Probe Testing (G. W. Scott)

Since our earliest experiments with a galvanic probe, which yielded some anomalous results in ZrO_2 coatings, we have done electron microprobe studies which showed that free Zr grains are present in the coatings. These grains can interfere with testing for holes in the coating by galvanic methods.

We have initiated experiments to test the feasibility of some active-probe electrochemical techniques. The first experimental objective will be to determine if an active probe technique can be made independent of any galvanic potentials set up by coating materials.

8.2.6 Microwave Testing (G. W. Scott)

We have fabricated two small stripline resonators which will be tested for their sensitivity to coating thicknesses or uniformity variation. Similar resonators have been used to test aluminum oxide circuit substrate materials in the 0.010–0.025-in. thickness range. We are modifying the methods slightly, from a microstrip (single gound plane) to a stripline (double ground plane) configuration.

8.3 Iron and Nickel Carbonyl Formation and Prevention

[Note: Mr. Henry Inouye (Ext. 3-1707) is now the lead engineer on the above program in place of J. Brynestad]

H. Inouye and J. H. DeVan

The rate of iron carbonyl formation is now being determined for the 1/2% Mo steel piping supplied by Pittsburgh Energy Research Center. Initial tests have been conducted at 200°C at 1000 psig using the gas mixture 30% CH_4, 51% H_2, 17% CO, and 2% CO_2. The levels of iron carbonyl in the effluent gas have been two to three orders of magnitude above the 1 ppb limit of detection of our analytical system. The dependence of formation rate on gas velocity is still being analyzed and will be reported next month. Additional tests will be conducted to measure the effects of temperature and pressure on formation rates using the same steel specimens.

Prior to the above experiments, we evaluated the extent to which our type 304 stainless steel reaction vessel reacted with the above mentioned gas mixture. With the vessel maintained at 200°C, gas was flowed through the vessel at varying flow rates at a constant pressure of 1000 psig. The level of iron carbonyl was below our limit of detection at all of the flow rates investigated; however, we detected a trace of nickel in the effluent gas throughout the duration of our test run, approximately 8 hr. The level of nickel was below the range for which we have calibrated standards for our spectrometer, and we are now attempting to prepare suitable standards. Although this nickel concentration is not of significance to the present experiments, we shall continue to monitor the behavior of the pressure vessel as an indication of the reactivity of stainless steel with our test gas.

8.4 Failure Analysis of Materials and Components

D. A. Canonico and D. P. Edmonds

We attended a meeting convened by FER at which representatives of the organizations responsible for the Failure Prevention tasks discussed their individual responsibilities and roles. The report on the post-service examination of the trim-set from the Synthoil PDU has been completed.

8.5 Prestressed Concrete Pressure Vessel Studies

W. L. Greenstreet

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.

There was continued emphasis during this month on establishing design details and preparing layouts of PCPV conceptual designs and on thermal analyses. Results from the latter are required for completion of vessel conceptual designs.

Under task 1, preparation of drawings showing design details for the Synthane vessel utilizing a hot liner concept was continued. Manway configurations, dictates of refractory requirements, and cover plate locations were addressed during this period. Also receiving attention were closures for other vessel opening, tendon layouts, and reinforcing bar requirements. Circumferential prestressing arrangements that are compatible with locations of radial penetrations were developed as were details of tendon anchor zone reforcement for vertical tendons. A support base system was integrated into the overall vessel design.

Drawings similar to those for the Synthane vessel are now in preparation for the HYGAS vessel. In the case of both the Synthane and the HYGAS gasifier vessel, final dimensioning will depend upon the results from the heat transfer calculations now under way.

Under task 4, some of the preliminary heat transfer analyses have been completed for hot liner concepts, and examination of a cold liner design was initiated. In the analyses being done, heat transfer in the liner-vessel interface region is considered in conjunction with heat flow through the thickness of the structural concrete vessel and insulation on the outer surface. Also involved are coolant tube configurations and locations; coolant inlet temperature, outlet temperature, and flow rates; heat transfer characteristics in the neighborhoods of the coolant tubes; and consequences of off-design conditions, such as, coolant circuit failure.

8.6 Other Related Work

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

8.6.1 Fluidized Bed Materials Support Activities

During this reporting period sixteen surveillance test tubes were fabricated under the supervision of J. F. King of the ORNL welding and brazing group and subsequently shipped for insertion into a fluidized bed coal combustion test facility designed and built by the Fluidyne Engineering Corporation. This fluidized bed test facility will have a 0.457 × 0.457 × 0.61 m (18 × 18 × 24 in.) combustion zone which will contain fifty-five 12.7 mm (1/2 in.) OD heat exchanger tubes which are used to maintain a continuous bed temperature of 1650°F. Sixteen of these heat exchanger tubes will also serve as surveillance test tubes. In order to maximize the number of materials that could be evaluated in the facility, the 0.457 m length of each tube exposed to the fluidized bed environment was divided into three 0.152 m (6 in.) segments. This arrangement gave a total of 48 test locations. The number of test locations occupied by each of the seven candidates will be as follows: Incoloy 800-13, Incoloy 800 Aluminized-4, 310 stainless-10, 310 stainless aluminized-3, 304 stainless-4, 316 stainless-9, and Inconel 600-5.

9. CRITICAL COMPONENTS TEST FACILITY

R. E. MacPherson, Jr.

To assure the availability of components considered critical to the operation of coal conversion demonstration plants, it is essential that a test facility be made available wherein existing commercially available equipment can be tested under simulated or actual plant conditions. Towards this end, the ERDA Division of Fossil Demonstration Plants has requested ORNL to prepare a proposal for the design, construction, and operation of a Critical Components Test Facility (CCTF).

The CCTF proposed by ORNL would provide all the facilities required to support the operation of individual test modules for testing such critical components as valves and valve operators, coal feeders, slurry pumps, compressors, waste cleanup systems, gas cleanup systems, turbines, and coal beneficiation equipment. The proposed site of the CCTF is the decommissioned power plant, adjacent to the Oak Ridge Gaseous Diffusion Plant (ORGDP). Scoping studies indicate that the existing installation consisting of a high-bay area of approximately 25,000 ft^2 and the adjacent turbine bay area of about 50,000 ft^2 would adequately house the test modules.

ERDA-FE also requested a proposal for the design, construction, and operation of a Valve Test Module (VTM) because reliability data on commercially available lock-hopper valves are urgently needed.

During the month of September, the on-going work on the feasibility studies of the CCTF and VTM was completed, and drafts of reports summarizing the results of each were prepared. The two drafts are X-OE-25 titled "Feasibility Study for Critical Components Test Facility" and X-OE-26 titled "Feasibility Study for Valve Testing."

The feasibility study report draft on the CCTF includes a layout of the building showing locations and areas required for each test module and a brief description of each module, as presently conceived, with its utility service requirements. Schedules, facility specifications, and methods of accomplishment are discussed.

The feasibility study report draft on the VTM includes the descriptions of four systems that were considered. To facilitate the testing of valves, we are proposing to supplement the VTM with a Valve Screening Module (VSM) to do preliminary valve evaluations and screening tests in relatively simple test stands. A description of the test stands and their uses was also included.

The VTM is expected to be operational in 12 to 18 months and the VSM is expected to be operational in about 6 months following ERDA-FE approval and funding.

10. ENGINEERING STUDIES AND TECHNICAL SUPPORT

J. R. McWherter

10.1 Process Modeling Support

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

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

A draft of a proposal to purchase copies of a commercial computer code was sent to the Fossil Demonstration Plants Division for consideration.

10.2 Process Research Digest

F. J. Endelman

A work schedule and production budget were prepared during this reporting period. The information will be transmitted in detail to the ERDA/DFER project officer.

The cost of converting a typed final draft manuscript for a single issue of the Digest into 100 copies of a printed product document was estimated. One month will be required, on the average, to write the report for each topic included in the Digest. It is estimated that publication of each issue of the Digest, assuming four topics/issue, will require a total of seven months. Thus, publication dates for three issues of the Digest in 1977 tentatively have been set for mid-April, -August, and -December.

ORNL requested that ERDA/DFER issue letters of introduction to contractors of the research projects to be reported upon in the first issue of the Digest before ORNL contacts the contractors. Such contacts will become increasingly important as time proceeds to the first scheduled publication date, as long as the unavailability of the contractors' progress reports from NTIS and other public sources continues to limit the information base for this issue.

11. PROCESS AND PROGRAM ANALYSIS

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.

11.1 Low Btu Gas

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

Coal gasfication processes are being surveyed in preparation for selecting certain ones for detailed technical and economic evaluations. Processes of more interest are those which have a greater potential for early commercialization. A revised work statement was prepared and proposed that the following be provided: (1) a brief summary of significant processes, (2) a source book containing technical and economic data on the more promising processes, (3) an assessment of the most favorable processes for coal resource regions and (4) an assessment of the work needed for future development of favorable processes.

11.2 Direct Combustion

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

During September the work statement for direct combustion was revised and submitted to ERDA for review and comment.

H. I. Bowers and R. L. Simard met with the Federal Energy Administration (FEA) on September 29 to discuss the analysis of reliability data collected by FEA. Subsequently we were requested by ERDA/FE/OPPA to include this analysis in the task on direct combustion. Arrangements are being made to obtain the data tapes from FEA and work on this subtask will be commenced immediately since significant results are desired by December 1.

11.3 Liquefaction

A subcontract for this work is being negotiated.

11.4 High Btu Gas

A subcontract for this project is planned.

11.5 In Situ Coal Gasification

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

A survey of the literature pertaining to the various concepts and processes proposed for in situ coal gasification was started. The purpose of the survey is to develop the information and data necessary to provide for technical and economic evaluations of the candidate projects: Linked Vertical Well (LVW), Packed Bed Process (PBP), Longwall Generator (LG), and Steeply Dipping Bed (SDB).

Contacts with individuals in various organizations directly involved with these projects were also initiated to supplement and update the literature survey.

11.6 Beneficiation

G. R. Peterson

A selection has been made of coals which will be used in evaluating the beneficiation costs for current and potential beneficiation processes. Bituminous coals were chosen from the Northern Appalachian Region (Pennsylvania Coal) and the Eastern Midwest Region (Illinois and Western Kentucky). A subbituminous coal and a lignite coal were selected from the Western Region (Wyoming and South Dakota, respectively). These coals were chosen on the basis of their particular sulfur content. Table 11.1 summarizes the characteristics of the different coals.

Washability test data for the coals have been obtained. An analysis of the data indicated that mechanical beneficiation will meet the desired sulfur level in one case with a reasonable coal yield. Table 11.2 reports the theoretical coal yields and the sulfur content for mechanical beneficiation of the various coals. Mechanical beneficiation of the Illinois coal will provide a desired sulfur level (2.9% S) at a theoretical yield of 87.4%.

Preliminary material balances have been prepared for the beneficiation of Pennsylvania coal by mechanical separation, by the Battelle process (BHCP), and by the TRW process (ferrous sulfate).

Table 11.1. Characteristics of Coal Selected for Evaluation

	Ash (%)	Sulfur (%)[a]			HV[a] (Btu/lb)
		Total	Pyrite	Organic	
Northern Appalachian Region					
State: Pennsylvania County: Somerset Coal bed: Lower Kittaning	27.3	2.90	2.46	0.44	11,049
Eastern Midwest Region					
State: Western Kentucky County: Hopkins Coal bed: 6	15.2	2.55	1.69	0.86	12,071
State: Illinois County: Williamson Coal bed: 6	18.5	4.21	2.58	1.63	11,382
Western Region					
State: Wyoming County: Sweetwater Coal bed: Rock Springs No. 11	6.2	0.96	0.23	0.73	12,577
State: South Dakota (exact bed has not been picked out)					

[a]Moisture-free basis.

Table 11.2. Yields and Sulfur Content of Mechanical Beneficiation

Raw coal - wt % sulfur		Desired product sulfur level (wt % S)	Mechanical beneficiation (wt % S)	Theoretical yield (wt %)
Pennsylvania	2.9	0.7	1.28	72.7
			1.17	69.2
			0.99	59.6
			0.74	20.8
Western Kentucky	2.55	0.7	1.70	85.2
			1.60	82.8
			1.51	80.9
			1.23	40.2
Illinois	4.21	0.7 and 2.9	2.63	86.8
			2.52	82.2
			2.44	74.1
			2.33	48.8
Wyoming	0.96	0.6	0.82	94.0
			0.81	91.1
			0.80	69.5

11.7 Gas Cleanup

M. S. Edwards

In preparation for the Gas Cleanup Systems study to be performed for the OPPA evaluations project, an extensive data base for the evaluation of design and economics of gas cleanup processes was compiled. Specifically, data for low temperature and high temperature acid gas removal processes were located. Some additional information in reference to particulate and alkali removal is still required for the complete design of recommended gas cleanup systems. Design criteria for the gas cleanup systems required for various methods of coal conversion and utilization must now be selected. A continuing dialogue with the investigators of methods of coal conversion and utilization is required to ensure consistent and compatible design and economics. A tentative listing of criteria and process information necessary for a realistic design was determined. These values require careful definition to provide an adequate (and technically defensible) basis for design. Coordination of effort with the investigators of low Btu gasification and in-situ gasification was initiated.

11.8 Coal Price

O. L. Culberson

It was determined that this effort will be in the form of a multiple regression analysis of published information on contracted delivered prices for coal between specified origins and destinations. In addition to price and quantity, each transaction shows coal characteristics in terms of rank of coal, type of mine, and the heating value, sulfur content and ash content. A multiple regression analysis of 100 to 200 of these transactions should give a satisfactory relationship between coal price and coal quantity and properties. Sulfur content will be of particular interest. The relationship will be for data reported for June 1976, and no effort is planned to forecast future coal prices. Since the data contain transportation costs, this regression analysis must await the imminent completion of information on coal transportation costs in order to back out shipping costs to arrive at estimates of mine-mouth prices.

11.9 Coal Transportation

O. L. Culberson

Data is regularly published on the cost of transporting coal from a variety of coal fields to a variety of destinations in the U.S., for shipments by rail in single cars, multiple cars and unit trains, and by barge. Sixteen such data points for shipments by unit train have been correlated by a least-squares fit to obtain:

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

where: y = transportation cost, \$/ton
x = distance, 100's of miles

The distances contained in the data range from 80 miles to 1155 miles.

The same kind of expression will be developed for single car shipments, for which 17 data points are available. An attempt also will be made to develop this kind of expression for rail multiple cars and barge, but the data for these modes are limited.

12. ENVIRONMENTAL ASSESSMENT OF COAL CONVERSION TECHNOLOGY

C. R. Boston

Work is now getting underway on this newly funded project. Staffing is approximately 75% complete. Accomplishments to date include reviews of two documents for ERDA/FE/FDP: (1) Draft EPA Regulations on Coal Conversion; and (2) The Coalcon Environmental Assessment, prepared by R. Weston Corporation. Work is underway on the preparation of environmental requirements for RFP's and an information overview of problems related to landfill disposal of solid wastes from coal conversion. The latter will lead to actual programmatic studies to be undertaken for the Division of Fossil Demonstration Plants. Work is also underway on the preparation of an Environmental Monitoring Handbook and Programmatic Impact Statements.

13. CHEMICAL RESEARCH

L. M. Ferris

This work is reported quarterly.

14. ANALYTICAL CHEMISTRY

W. R. Laing

14.1 Bioactivity Testing of Fossil-Derived Materials

B. R. Clark, I. B. Rubin, and H. Kubota

Skin painting tests with mice have been started to assess the relative carcinogenic potentials of several available fossil-derived crude oils and some fractions thereof. J. M. Holland, Biology Division, is conducting the tests on samples prepared in the Analytical Chemistry Division--Bio/Organic Analysis Section. The samples chosen for this study are: Synthoil, COED syncrude, shale oil, a petroleum blend (six different crudes), and polycyclic aromatic hydrocarbon (PAH) isolates from each of the materials. The Synthoil and shale oil samples, along with their PAH isolates, will be applied at four dose levels to determine response curves. Since a substantial amount of data have been obtained on these samples with respect to mutagenic activity, some comparisons may be possible to evaluate the mutagenic screening as a possible predictor of carcinogenic activity.

14.2 Chemical Characterization of Fossil-Derived Materials

C. -h. Ho, A. R. Jones, and B. R. Clark

Paraffin and polycyclic aromatic hydrocarbon isolates have been prepared from Synthoil, COED syncrude, shale oil, a petroleum blend, a Louisiana-Mississippi sweet petroleum and by-product waters from the COED process, the Synthane process, and oil shale retorting. A complete tabulation of the analytical data (part of which have appeared in previous reports) will be published in the Coal Technology Program Quarterly Progress Report for Period Ending September 30, 1976 (ORNL-5224).

The recent acquisition by the Analytical Chemistry Division of an interfaced gas chromatograph--mass spectrometer--data system will facilitate identification of compounds and compound classes in the various materials and fractions. This capability makes separations development work much easier and efficient. Substantial progress is expected in this area during FY 1977.

14.3 General Analysis Laboratory

L. J. Brady

A total of 157 samples were submitted for analysis during the month. Eighty-four of these samples were derived from solids-liquid separation tests.

Tests on 14 samples derived from hydrocarbonization tests were completed. Proximate and ultimate analyses were done on the coal and char samples, and ash, carbon, and sulfur tests were done on the liquid samples.

There were 56 gas samples from in situ gasification (pyrolysis) tests that were tested for CO, N, and CH_4.

An order has been submitted for a new Varian integrator to be used with the gas chromatographs which are used for testing gas and liquid samples from the coal programs.

15. COAL-FUELED MIUS

A. P. Fraas and W. R. Mixon

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.

15.1 Furnace Procurement

The two bids received for fabrication of the furnace and tube bundles were evaluated, and the low bid was found acceptable. It appears likely, however, that ERDA will require procurement of a commercially designed furnace. In this event, bids received for fabrication of the ORNL design will be permitted to expire (on October 18) and bids will later be requested from furnace manufacturers for both design and fabrication.

15.2 Turbine-Generator Unit

Analysis of the electronic control package and design of turbine instrumentation and controls continued.

15.3 Site Preparation

Drawings for enlargement of the control room were completed. The completion of drawings for relocation of existing equipment has been postponed until a special development test being conducted in this building can be completed. This delay is not expected to affect the installation schedule of the coal-fired gas turbine system.

15.4 Cold Flow Tests of a Fluidized Bed

Analysis of fluidizing and heat transfer tests using smaller sized limestone in the small cold flow model was completed. Heat transfer coefficients of about 70 Btu/hr-ft^2-°F were measured over the velocity

range of 1 to 2.5 ft/sec for room temperature air fluidizing the bed. This corresponds to a heat transfer coefficient of about 90 Btu/hr-ft^2-°F at a bed temperature of 1650°F.

15.5 Coal Metering and Feed System

The flow splitter feed system was not operated this month. The components were removed from the test stand and are being installed in a new test system that will operate continuously in order to complete a 1000 hr endurance test.

15.6 Ash Handling System

Work continued on the design of the ash handling system and preparation of specifications for the components of the system.

15.7 Supplemental Studies

All corrosion test specimens had been shipped to FluiDyne by the end of September. The corrosion test is expected to begin early next month.

The analysis of heat transfer test data is nearly complete and the report will be prepared next month.

Internal review of the draft of the report on system performance with alternate cycles has been completed. A copy of the draft was submitted to ERDA and HUD for review.

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