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

OAK RIDGE NATIONAL LABORATORY

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

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COAL TECHNOLOGY PROGRAM
PROGRESS REPORT FOR JUNE 1976

AUGUST 1976

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Oak Ridge, Tennessee 37830
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COAL TECHNOLOGY PROGRAM PROGRESS REPORT FOR JUNE 1976

ABSTRACT

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

1. SUMMARY

J. P. Nichols

Highlights of our progress in June are summarized below:

° In our Hydrocarbonization Research Program, two successful experiments were completed in the bench-scale hydrocarbonizer, HC-5 and HC-6. The 80 atmosphere bench-scale hydrocarbonization system has been cancelled.

° In our Solids-Liquid Separations project the bench-scale settler modifications were completed and the unit is back in operation. Six additive settling runs this month gave maximum settling rates comparable to those obtained with the lab-scale apparatus.

Toluene settling rates were also comparable to lab-scale data. One decane settling run gave the highest settling rates, but increased the solids content by a significant amount.

A program review was conducted by Dr. John Shen, ERDA/FE - Division of Fossil Energy Research, on Wednesday, June 16, 1976 at ORNL.

° High-Temperature Pyrolysis studies equipment problems described during the last report period have been resolved and large block pyrolysis experiments have been resumed. Pyrolysis tests employing Wyodak Subbituminous blocks at 800°C were repeated, using heating rates of 3°C/minute and 0.3°C/minute. Analysis of oils produced by these experiments have shown that significantly more oil is produced by pyrolysis at the lower heating rates and that these oils have a rather narrow boiling range. The boiling range becomes progressively narrower, occurring at lower temperatures as the maximum pyrolysis temperature increases.

° Experimental engineering support of an In Situ Gasification Process had a third working group meeting for laboratory studies in support of ERDA in situ coal gasification programs at Morgantown ERC on June 3.

Experimental work in progress at LLL, MERC, LERC, ORNL, ANL, and Sandia were described informally and discussed critically by all participants. Also participating as observers were representatives from the Universities of West Virginia, Alabama, and Pennsylvania (State). Following descriptions of current research, Al Sikri (USERDA-Washington) led a general discussion of the laboratory research programs' relationship to the remainder of the ERDA UCG development effort and entertained suggestions as to the direction which those programs might take in the near future to best serve overall objectives.

° The Analytical Chemistry progress was on the development of a biological screening activity for testing fossil fuel related materials. Chemical characterization of aqueous by-products and product liquids has included development of separation and fractionation methods and direct methods in analysis of classes of compounds, e.g., polynuclear aromatic hydrocarbons and paraffins. Work is nearly complete in the development of sample introduction and gas analysis systems for operating a plant exposure system in collaboration with the Environmental Sciences Division.

Data has been obtained on the relative mutagenicity of the various chemical class fractions of fossil derived liquids. Fractions produced for mutagenesis testing were: shale oil by-product water, shale oil, Synthoil product oil and a petroleum crude.

° Engineering evaluations of the Synthoil and Hydrocarbonization Processes progress was as follows:

Synthoil. Major effort was placed on acquiring equipment cost data and developing computerized estimating procedures. A computer program that calculates the price per barrel of liquid product as a function of various parameters such as coal price, debt-to-equity ratio, estimated capital and operating costs, etc., was made operational. First drafts of process descriptions for Volume I of the Final Report were completed for all units. Additional documentation required for Volume II was also assembled and used to draft descriptions of existing data gaps and process development and equipment problem areas.

Hydrocarbonization. A meeting was held with U.S. Steel personnel to discuss the ORNL hydrocarbonization design which is based in part on the U.S. Steel "Clean Coke" process. In general, agreement was reached on the flowsheets and the assumptions used for the design basis. U.S. Steel had reservations concerning the recycle of off gas from the second stage carbonizer to the first stage because this configuration has not been tested in their Process Development Unit.

The design and cost evaluation continued with emphasis on capital and operating cost estimation. Foster Wheeler completed the design study of a fluid bed combustor for the hydrocarbonization facility. Comments on the draft Foster Wheeler report have been prepared and preparation of the final report is in progress.

° The Materials effort for the Pressure Vessel and Piping Technology Program is continuing. Various sections of the report have been written and are being reviewed in preparation for a final draft submission to Fossil Energy Research in July.

Coating systems for NDT studies have been selected, and we are attempting to make specimens with known cracks in the coating to evaluate detectability with liquid penetrants. We are computing heat-transfer effects of laminar and unbond defects in coating-substrate systems with a computer code in an effort to determine feasibility and provide hardware design data for thermal inspection techniques. Prototype electrolytic and corona probe systems have been fabricated; preliminary experiments were inconclusive regarding feasibility.

The atomic absorption spectrophotometer has been optimized for the direct monitoring of iron and nickel carbonyl in carbon monoxide and H_2 -CO- CH_4 mixtures. We have found the detection limit for iron carbonyl in these gases to be about 1 ppb. We were asked by PERC to evaluate the efficiency and useful lifetime of the charcoal trap which will be used to purify the gas mixture fed into our reaction chamber.

The examination of the pressure let-down trim set from the Synthoil PDU at PERC is almost completed. We have made a plexiglass model of the valve and are studying flow patterns of liquids past the trim set in an attempt to determine the cause of premature failures. Investigations into the failure of the type 316 stainless steel heater tube from Project Lignite are underway.

In our Prestressed Concrete Pressure Vessel Program, refractory and liner design studies were increased and emphasis was given to obtaining improved data on temperatures and temperature distributions in the vessel-liner-refractory composite structures. Work was continued on the design of the closure in the top head for the Synthane gasifier vessel. Examinations were initiated on circumferential prestressing methods and configurations as well as preloads and preloading techniques. Studies of refractory lining materials and properties were initiated.

° Coal-Fueled MIUS Program distributed bid packages for the fluidized-bed furnace with tube bundles to 17 vendors on June 10, 1976.

Specifications for the turbine recuperator modules and the regenerator were prepared, but procurement of these units and placement of the turbine modification contract are awaiting receipt of additional

funds. Work continued on building modification design and analysis of the turbine control system was initiated. Endurance tests of the coal feed system and prototype coal feed nozzles and work on the supplemental studies continued.

2. HYDROCARBONIZATION RESEARCH

H. D. Cochran, Jr.

2.1 Bench-Scale Hydrocarbonization System

H. D. Cochran, Jr., G. L. Yoder,
P. R. Westmoreland, and R. L. Andrews

2.1.1 Work Accomplished

Design and Review. According to instructions from the Division of Coal Conversion and Utilization, all effort on the 80 atm bench-scale hydrocarbonization system has been curtailed.

Operation. Routine experimentation continued in June with two hydrocarbonization experiments as follows:

HC-5 - Uniformly fluidized bed reactor at 965°F and about 300 psig H₂ pressure, Wyodak subbituminous coal fed at 15.5 lb/hr for 7.7 hr.

HC-6 - Uniformly fluidized bed reactor at 1030°F and about 150 psig H₂ pressure, Wyodak subbituminous coal fed at 16.3 lb/hr for 5.1 hr.

Samples and data from these experiments are being analyzed. Material balances will be reported in the July monthly report.

Preliminary material balances and gas compositions determined for experiments HC-1, HC-2, HC-3, and HC-4 are summarized in Table 2.1. Material balances were closed satisfactorily in HC-3 and HC-4, and valid yield data were obtained from all experiments. Critical data such as coal analyses are being verified.

2.1.2 Work Forecast

Fabrication and Installation. a 3/4 in. draft tube with deflector will be installed in the 20 atm reactor.

Operation. Routine operation of the bench-scale reactor will continue with a 20-atm test of the recirculating fluidized-bed reactor. About 1 ton of Wyodak coal has been ordered and should arrive in July. This coal will be ground, dried, and sieved for future experiments.

Table 2.1 Summary of preliminary material balances
for experiments HC-1 through HC-4

Average bed temperature, °F	910	1000	1030	1075
Reactor pressure, psig	300	300	300	300
Experiment	HC-3	HC-4	HC-2	HC-1
Carbon balance (% of carbon fed)				
Char	70.3	66.5	51.3	51.5
Liquids	14.7	13.0	14.3	9.88
Gases	14.0	23.4	23.5	38.6 ^a
Overall	99.0	102.9	89.0	100.0
Carbon balance (% of carbon recovered)				
Char	70.5	64.6	57.5	-
Liquids	14.8	12.7	16.0	-
Gases	14.1	22.7	26.4	-
Overall ash balance (% of ash fed)	92.4	97.3	71.9	90.1
Sulfur balance				
Char	41	38	20	52
Liquids	7.4	4.9	5.7	7.1
Gases	52 ^a	53 ^a	74 ^a	41 ^a
Oil yield (weight % of maf coal)	11.2	10.3	11.2	8.7

^aCalculated by difference

2.2 Residue Carbonization

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

2.2.1 Work Accomplished

Three residue carbonization runs, RC-7, RC-8, and RC-9, and seven agglomeration tests were performed in June. The first residue carbonization run was at 1200°F and the latter two at 975°F while the agglomeration tests ranged from 390°F to 1110°F.

Since the previous residue carbonization runs had all resulted in a plugged draft tube, in RC-7 it was attempted to operate the reactor as a spouted bed (i.e., no draft tube). The run lasted 3 1/2 hours at a feed rate of 1/4 lb/hr. It was terminated due to system overpressure after plug developed in the line between the reactor and

scrubber. During clean-up a 3 in. deep, very dense plug was found which covered the distributor plate. This indicated that there had been a substantial layer of molten residue in the bottom of the reactor. In Run RC-8 the reactor was operated with a draft tube and with the feed nozzle inserted 5 in. up into the draft tube. The run lasted 2 hr at a feed rate of 1/4 lb/hr and was terminated when the feed line plugged. During clean-up it was discovered that the draft tube was plugged and that a very large agglomerate, composed of many small agglomerates was stuck near the base of the draft tube. This agglomerate did not have the appearance of having been molten. The conditions for Run RC-9 were the same as in RC-8 with the exception that the feed was a mixture of Wyodak char and residue with a char to residue ratio of 4:1. Early in the run the feed nozzle plugged and was replaced by a nozzle that was flush with the distributor plate. The run lasted 3 1/2 hr at a feed rate of 2 lb/hr and was terminated due to system overpressure when a plug developed in the line between the reactor and scrubber. Besides the plug which caused shutdown, the draft tube was found to be plugged and a 6 in. stalagmite was hanging from the deflector cup over the draft tube. The plug in the line between the reactor and scrubber was a hard, dense plug, but the plug in the draft tube and the stalagmite were soft and porous. No large agglomerates were found in the downcomer region of the fluidized bed.

All of the agglomeration tests were performed with mixtures of Wyodak char and residue in the ratios of 2:1, 4:1, and 6:1. Agglomeration in all these tests was slight and the agglomerates were softer and more porous than those formed in tests with residue alone. In all cases the total weight of agglomerates recovered was less than the weight of the residue in the feed mixture.

2.2.2 Work Forecast

More runs are planned in order to determine conditions for which agglomeration problems are a minimum in both the residue carbonizer and the agglomeration test apparatus. These plans include operations with char/residue feed mixtures and a larger diameter draft tube.

3. SUPPORTING RESEARCH AND DEVELOPMENT IN SEPARATIONS TECHNOLOGY

B. R. Rodgers

3.1 Bench-Scale Tests

B. R. Rodgers and D. A. McWhirter

The bench-scale settler modifications, discussed last month, were completed and the unit was reinstalled on June 7. Three sets of duplicate runs were made at 530°F using 4000 ppm petroleum additive, an inorganic additive, and a base run with no additive. The settling rates were within the range of values for comparable runs on the lab-scale apparatus.¹ An additional run at 530°F using 20% toluene gave a settling rate of 8.8 inches/hr compared to 7.0 inches/hr for the lab-scale run,² at 390°F. A 20% decane run comparable to the toluene run gave the highest settling rate, 18.3 inches/hr. However, filtration tests show that the solids content is increased by the addition of solvent:

No additive (or < 4000 ppm additive)	~ 7 wt %
20% toluene	15.3 wt %
20% decane	32.4 wt %

The faster settling and filtration rates obtained with toluene and decane must be balanced with the increased solids processing load.

A sealed filtrate weighing receiver has been constructed and installed on the bench-scale filter. Tests are planned to compare lab-scale coal precoat results using bituminous coal with bench-scale results using both bituminous coal and char.

Additional data will be given in the Quarterly Report for the period ending June 30, 1976, in preparation.

3.2 References for Section 3

1. J. P. Nichols (Program Director), Coal Technology Program Quarterly Progress Report for the Period Ending March 31, 1976, ORNL-5159, page 36 (July 1976).
2. J. P. Nichols (Program Director), Coal Technology Program Quarterly Progress Report for the Period Ending December 31, 1975, ORNL-5120, page 24 (March 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, T. M. Somers, and G. D. Owen

A malfunctioning reactor temperature controller caused a short delay in large block pyrolysis studies near the close of the last report period. This problem was resolved quickly and experiments at 800°C were repeated at both heating rates of interest. As in the earlier tests which were marred by the reactor malfunction, the residual char was found to be pyrophoric and, upon exposure to air, self-heated rapidly to a temperature of 350°C.

Tars from these and earlier experiments were examined in some detail with the results shown below:

Table 1. Analysis of tars produced by large block pyrolysis experiments using a heating rate of 0.3°C/minute

Maximum pyrolysis temperature, °C	Heating Value, BTU/lb	Boiling range, °C
500	18,000	245 - 465
600	16,800	230 - 415
700	17,000	210 - 370

Boiling rate seems to become narrower as the pyrolysis temperature maximum increases, and the boiling points themselves, are in general, lowered with succeeding increases in pyrolysis temperature. Comparison of these data with the work done at LECR tends to contradict their evaluation of the conditions at which their tars were produced; however, the many unknown variables affecting their oils prior to sampling and examination make direct comparisons ill-advised.

4.2 ERDA's UCG Program

R. C. Forrester, III

The third working group meeting for laboratory studies in support of ERDA in situ coal gasification programs was held at the Morgantown ERC on June 3. Experimental work in progress at LLL, MERC, LERC, ORNL, ANL, and Sandia were described informally and discussed critically by all participants. Also participating as observers were representatives

from the Universities of West Virginia, Alabama, and Pennsylvania (State). Following descriptions of current research, Al Sikri (USERDA-Washington) led a general discussion of the laboratory research programs' relationship to the remainder of the ERDA UCG development effort and entertained suggestions as to the direction which those programs might take in the near future to best serve overall objectives.

5. ANALYTICAL CHEMISTRY

W. R. Laing

5.1 Chemical Fractionation of Fossil Derived Liquids for Bioactivity Testing

I. B. Rubin

In this reporting period, fractions produced for mutagenesis testing were: shale oil by-product water, shale oil, Synthoil product oil and a petroleum crude. Separations into chemical classes were performed using the Stedman¹ procedure described in previous reports.

A six-liter batch of by-product water from the retorting of oil shale² was extracted in a continuous extractor to produce sufficient sample for several tests.

A set of samples was delivered to J. L. Epler of the Biology Division for mutagenesis testing, and another set was delivered to S. Herbes of the Environmental Sciences Division for toxicity testing. In addition, a 1-liter batch of shale oil product water was fractionated, using batch extractions rather than the continuous extraction procedure. Despite the differences in handling, proportions of comparable fractions in the two samples agreed well. Soluble acid fractions accounted for about 87% of the total organics, soluble base fractions about 5%, and neutrals about 2.5%.

Two 20- to 25-g portions of shale oil were fractionated. Proportions of comparable fractions agreed well. Soluble acid fractions accounted for only about 2% of the total sample, soluble bases, 7%, and neutrals, 87%. Half of the neutral portion of the second sample was chromatographed on Florisil in the manner used previously for COED Syncrude and Synthoil oil.

5.2 Analysis of PAH Components in Fossil Fuel Related Liquids

B. R. Clark and C. H. Ho

A reliable procedure, which is applied to routine analysis of PAH materials in cigarette smoke, has been used to determine the concentration of PAH's in crude petroleum, shale oil, Synthoil, and Synthane by-product water. Five μ l of the PAH fraction were injected into a gas chromatograph equipped with a packed 3% Dexsil 400 column. The tentative identifications were made by co-chromatography and quantification was achieved using standard solutions. Structural confirmations are underway using GLC/MS. These data are summarized in the following tables.

Crude petroleum contains PAH's in the 2-3 ring range with alkyl substituted naphthalenes predominating. Shale oil is similar in this regard but has about seven times higher overall concentrations of the PAH's determined. Synthoil has significantly higher distribution of PAH's in the 3-6 ring category, e.g., 4300 ppm pyrene, 270 ppm 1,2-benzanthracene, 380 ppm picene and 1500 ppm benzo(g,h,i)perylene. The overall PAH concentrations are somewhat less than in shale oil ($\sim 2/3$) but still about five times greater than in petroleum crude.

Shale oil retort water shows a broad spectrum of PAH materials, but the larger proportion fall into the 2-4 ring category. Synthane by-product water contains principally 2-methylnaphthalene with no detectable PAH's of more than three rings.

5.3 Specific Compound Class Analyses

C. H. Ho and B. R. Clark

In conjunction with fractionation procedures it is desirable to have available methodologies for specific class analyses. Although the sample may be rendered useless, after treatment, for further analyses, a direct method for a specific class of compounds is economical and essential for routine monitoring.

Polynuclear aromatic hydrocarbons (PAH) constitute one important class of compounds which can be isolated by a reliable procedure used with tobacco smoke condensates. This method was applied to the determination of PAH's in by-product waters from oil shale retorting and Synthane coal gasification.

One liter of water was extracted four times with 200 ml of cyclohexane. The cyclohexane fractions were combined and reduced to 20 ml and extracted successively with four portions of DMSO (20 ml each). The combined DMSO extracts were diluted with water to 40% (volume) DMSO and inversely extracted with four 20-ml portions of cyclohexane. The combined cyclohexane extracts were reduced to 5 ml for chromatography on a column (50-ml burette) filled with 10 g of Florisil, using hexane/benzene (6/1) as eluting solvent. About 150 ml of eluate was pooled and reduced to 5 ml for another column separation [50-ml burette, 20 g of activity II neutral alumina, hexane/benzene (2/1) as eluting solvent]. Four hundred milliliters of eluate was collected and reduced to 1 ml for GLC analysis.

Five microliters of the PAH fraction were injected into a gas chromatograph equipped with a packed 3% Dexsil 400 column. The tentative identifications were made by co-chromatography and quantification was achieved using standard solutions. Structural confirmations are proceeding by GLC/MA analysis in W. T. Rainey's group. These data are summarized in Table 1.

Similarly the PAH isolate can be produced from crude products. Data in Table 2 compare a crude petroleum (Louisiana-Mississippi sweet crude) with shale oil and Synthoil.

The paraffin class is rather simply isolated from an aqueous sample or a crude oil. A sample of shale oil (1 g) and a sample of shale oil by-product water (1 liter) were dissolved in or extracted with hexane, respectively. The combined hexane extracts from the water were washed with NaOH solution (pH 11) and HCl solution (pH 4). The paraffin fractions were isolated by alumina column chromatography (neutral activity I), using n-hexane as eluting solvent. The column eluate was pooled and reduced to 1 ml for GLC analysis. A variety of liquid phases and supports were tried in GLC-columns of various lengths. The most satisfactory separation of the paraffin fraction from shale oil and shale oil by-product water was obtained on a 1/8 in. by 10 ft. glass column packed with 3% Dexsil 400.

Table 3 lists the quantitative results obtained for the n-alkanes.

5.4 Separations Using Sephadex LH-20

A. R. Jones

Crude shale oil was dehydrated by azeotropic distillation with benzene. The benzene solution was then filtered through a 1 cm pad of powdered cellulose and 5 and 2 micron nucleopore membranes. There was little insoluble material. The benzene was removed on a rotary evaporator at 52°/50 mm.

A 17.5 g aliquot of the dehydrated and filtered oil was dissolved in benzene and the 50 ml of solution was added with a syringe to a 5 X 100 cm column containing 500 g of Sephadex-LH-20 which had been swollen with 85% methanol and equilibrated with hexane. The column was washed with 3700 ml of hexane (more than 4X the quantity required to elute benz(a)pyrene). In the absence of a suitable detector the eluate was collected in tared flasks to constant weight.

The lipophilic fraction was 92.5% of the original sample. The hydrophilic fraction was eluted with methanol and with acetone. Total recovery was 100%. A second aliquot of 17.9 g was separated with a recovery of 101%. The lipophilic fraction amounted to 94.7% in the run. Over 90% of the lipophilic fraction was eluted in the 500 ml of eluate immediately following the interparticle volume of solvent. The first step of the procedure was thus shown to be loss-free and reasonable reproducible. The fractions are being further characterized.

Table 1.

ORNL DWG. 76-7876

**Estimation of Polynuclear Aromatic Hydrocarbons
in By-Product Water from Fossil Fuel Conversion Processes**

Peak	Compound	Concentration ($\mu\text{g/l}$)	
		Oil Shale Retorting	Synthane Coal Gasification
1	Naphthalene	86	160
2	2-Methylnaphthalene	330	1300
3	1-Methylnaphthalene	110	32
4	Biphenyl	33	2
5	2,6-Dimethylnaphthalene	46	48
6	1,3 + 1,6-Dimethylnaphthalene	250	66
7	1,5 + 2,3-Dimethylnaphthalene	100	24
8	1,2-Dimethylnaphthalene	38	10
9	Acenaphthene	45	IR
10	Fluorene	180	21
11	9,10-Dihydroanthracene	27	2
12	9-Methylfluorene	IR	ND
13	1-Methylfluorene	160	ND
14	Phenanthrene + Anthracene	260	ND
15	2-Methylanthracene	210	ND
16	1-Methylphenanthrene	130	ND
17	9-Methylanthracene	35	ND
18	Fluoranthene	18	ND
19	Pyrene	120	ND
20	1,2-Benzofluorene	10	ND
21	2,3-Benzofluorene	5	ND
22	1-Methylpyrene	61	ND
23	1,2-Benzanthracene	37	ND
24	Chrysene	5	ND
25	1,3,5-Triphenylbenzene + 1,2-Benzopyrene	5	ND
26	Perylene	ND	ND
27	3-Methylcholanthrene	6	ND
28	Picene	ND	ND
29	Benzo (g, h, i) perylene	ND	ND
30	Anthanthrene	ND	ND

ND = not detected

IR = incomplete resolution

Table 2.

ORNL DWG. 76-7875

**Estimation of Polynuclear Aromatic Hydrocarbons
in Crude Petroleum, Shale Oil, and Synthoil**

Peak	Compound	Concentration ($\mu\text{g/g}$)		
		Crude Petroleum	Shale Oil	Synthoil
1	Naphthalene	200	790	440
2	2-Methylnaphthalene	650	4600	1400
3	1-Methylnaphthalene	290	2900	430
4	Biphenyl	23	190	84
5	2,6-Dimethylnaphthalene	250	3400	250
6	1,3 + 1,6-Dimethylnaphthalene	670	5100	170
7	1,5 + 2,3-Dimethylnaphthalene	220	2000	180
8	1,2-Dimethylnaphthalene	37	1100	IR
9	Acenaphthene	IR	280	IR
10	Fluorene	220	940	345
11	9,10-Dihydroanthracene	ND	26	1700
12	9-Methylfluorene	ND	410	90
13	1-Methylfluorene	140	980	1900
14	Phenanthrene + Anthracene	290	620	1400
15	2-Methylantracene	ND	IR	ND
16	1-Methylphenanthrene	330	IR	ND
17	9-Methylantracene	3	ND	IR
18	Fluoranthene	68	400	380
19	Pyrene	IR	170	4300
20	1,2-Benzofluorene	22	53	IR
21,	2,3-Benzofluorene	13	140	IR
22	1-Methylpyrene	36	70	620
23	1,2-Benzanthracene	IR	IR	270
24	Chrysene	IR	IR	130
25	1,3,5-Triphenylbenzene + 1,2-Benzopyrene	20	180	IR
26	Perylene	31	22	ND
27	3-Methylcholanthrene	ND	20	200
28	Picene	ND	ND	380
29	Benzo (g, h, i) perylene	ND	ND	1500
30	Anthanthrene	ND	ND	80

ND = not detected

IR = incomplete resolution

Table 3.

n-Alkanes in Shale Oil and Shale Oil By-Product Water

Compound	Oil (mg/g)	Water (μ g/liter)	Compound	Oil (mg/g)	Water (μ g/liter)
C ₁₁ H ₂₄	7.55	67	C ₂₄ H ₅₀	3.60	48
C ₁₂ H ₂₆	6.65	66	C ₂₅ H ₅₂	3.90	53
C ₁₃ H ₂₈	6.20	65	C ₂₆ H ₅₄	3.45	50
C ₁₄ H ₃₀	4.80	62	C ₂₇ H ₅₆	3.35	51
C ₁₅ H ₃₂	3.30	45	C ₂₈ H ₅₈	2.90	48
C ₁₆ H ₃₄	4.50	53	C ₂₉ H ₆₀	3.20	62
C ₁₇ H ₃₆	8.60	105	C ₃₀ H ₆₂	1.00	30
C ₁₈ H ₃₈	8.80	106	C ₃₁ H ₆₄	0.20	27
C ₁₉ H ₄₀	5.60	70	C ₃₂ H ₆₆	0.10	9
C ₂₀ H ₄₂	4.80	62	C ₃₃ H ₆₈	0.65	12
C ₂₁ H ₄₄	5.70	53	C ₃₄ H ₇₀	0.40	5
C ₂₂ H ₄₆	4.50	59	C ₃₅ H ₇₄	0.15	2
C ₂₃ H ₄₈	5.00	67			

5.5 Development of Sampling Methodology for a Terrestrial Plant-Controlled Atmosphere-Exposure System

A. D. Horton

Some studies have been completed to determine the stability of gas mixtures, especially when H_2S is a component. These are summarized as follows:

(1) In a mixture of all components (except H_2S), methane, ethylene, benzene and xylene responses were constant one hour after introduction into the chamber.

(2) Phenol did not give a measurable response before two hours. The response was erratic, and gradually increased for about six hours until it became constant within $\pm 15\%$. After the phenol bubbler was turned off, the decay of the phenol response was about twice the rate of the build-up, and it dropped by a factor of two in one hour.

(3) There was no significant change in the levels of the organic components upon introduction of H_2S into the chamber. There were, however, three component peaks that appeared in the high boiling region of the chromatogram (max $T = 180^\circ C$). The fluctuation of these peaks was large from run to run, and they varied in magnitude from <1 to $\sim 9\%$ based on the peak height of benzene. These components were present in lesser amounts in the absence of H_2S . The response of H_2S only in the presence of each of the organic components individually and collectively was constant except in the presence of methylamine where the magnitude of the H_2S response was reduced about 25%. No sulfur component peaks, except H_2S , were detected in the presence of the organic components either individually or collectively.

5.6 Analytical Services

W. R. Laing and L. J. Brady

During the month a total of 210 samples were submitted for analysis.

A total of 155 SRC samples were analyzed for ash content. Equipment for the preparation of these samples was down during the first two weeks of this period, but during the last two weeks the rate of submission of samples was about equal to that of previous months.

Three series of liquids, gases, and coal char samples were submitted from hydrogenation tests being conducted by personnel from the Chemical Technology Division. The gases were tested for CO , N , and CH_4 content. The liquids were analyzed for H_2O , C , H , and ash; and proximate and ultimate analyses were made on the solid coal and char samples.

Fifteen charcoal samples were analyzed for ash and sulfur content. Because of the low ash content, 4- to 5-gram samples were burned in order to collect enough residue to weigh. Sulfur was determined gravimetrically after the samples was burned in an oxygen atmosphere in a Parr bomb.

The new Perkin Elmer Model 3920-B gas chromatograph has been put into operation by the factory engineer and is being used to run samples.

5.7 References for Section 5

1. Swain, A. P., Cooper, J. E., and Stedman, R. L., Cancer Res., 29, 579 (1969).
2. Sampler obtained from Laramie Energy Research Center, Laramie, Wyoming.

6. ENGINEERING EVALUATIONS OF THE SYNTHOIL AND HYDROCARBONIZATION PROCESSES

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

6.1 Synthoil Process

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

A major effort this month was placed on acquiring equipment cost data and developing computerized estimating procedures to arrive at an overall capital cost estimate for the Synthoil facility. Vessel costs quoted by the Chicago Bridge and Iron Company were used in the development of costs for all vessels in the Synthoil process. Vendor quotes were supplemented, especially for the smaller vessels, by a combination of open literature and in-house estimating procedures. The resulting small vessel cost figures will be compared with rough estimates supplied by the Nooter Corporation. The tabulation of operating costs of the Synthoil facility was completed.

Last month a computer program was described that calculates product price, based on the discounted cash flow procedure, using inputted capital and operating costs. This program has not been expanded to make parametric studies in which both the coal price and the rate of return on equity are varied over a given range. Several test runs were made to check out this capability. Also, the income tax section of the program was enlarged. Several options are now available. In one of these, a parent corporation is assumed which can take advantage of tax losses suffered during the early years of the project. In another, only the 5-year tax loss carry-forward provision is used.

First drafts of process flow descriptions for Volume I of the Final Report were completed for all units. Additional documentation required for Volume II was also assembled and used to draft descriptions of existing data gaps and problem areas in process and equipment development. Some of these, for example, are solid-liquid separations; Synthoil reactor design, materials of construction, and environmental pollution considerations. Single-page mass and energy flow diagrams for each process unit were prepared for inclusion in Volume III. These simplified diagrams depict each unit as a single "black box" with its appropriate input and output streams.

Material balance calculations for all units were reviewed and refined for compatibility from unit to unit, and minor changes and revisions were made to some process flow diagrams.

6.2 Hydrocarbonization Process

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

A meeting with U.S. Steel personnel was held on June 3-4 in Monroeville, Pennsylvania, to discuss the hydrocarbonization flowsheet developed at ORNL. Attending the meeting were George Peterson, David Joy and John Holmes. Since the core unit of the hydrocarbonization flowsheet is based on part of the U.S. Steel Clean Coke process, it was desirable to present the proposed flowsheet to the U.S. Steel engineers.

In general, U.S. Steel agreed with the flowsheet and the assumptions used to arrive at the design basis. Since a definitive material balance is not available, the estimated material balance used was considered to be reasonable. Also, samples of the oil and tar from the first stage U.S. Steel carbonizer have not been isolated. Oils from the second stage have been recovered and contain 1-1 1/4% sulfur. The composite oil (both stages) was assumed to contain 2.4% sulfur. U.S. Steel suggested that this number may be high but conservative for design purposes.

U.S. Steel engineers also expressed a concern about using the off-gas from the second stage as part of the fluidizing gas for the first stage. The first stage operates at 800-840°F (840 + in the ORNL design). Some of the oils produced in the second stage start to condense at approximately 780-800°F. If significant condensation occurs in the first stage it could cause agglomeration. However, to date no experimental tests have been made to investigate this point.

Materials of construction were also discussed. U.S. Steel favors Incolloy 800 for the second stage carbonizer. They do not favor Monel due to its high nickel content. There is some possibility the low temperature unit (Stage I) could use a stainless steel lining instead of Incolloy 800 but they did not recommend carbon steel.

The design and cost evaluation of the hydrocarbonization facility continued with major emphasis on capital and operating cost estimation. Foster Wheeler completed the design study of a fluid bed combustor (FBC) design for providing the process heat and steam for the facility. The FBC system consists of six modules, each having seven vertically stacked fluidized beds and a bed plan area of 15 by 36 feet. The total cost of the modules with associated equipment including installation but not including structural steel and concrete is 121,433,000. Comments on the Foster Wheeler draft report have been prepared and the final report is in preparation.

7. MATERIALS

W. R. Martin and D. A. Canonico

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

7.1 Pressure Vessel and Piping Technology Assessment

D. A. Canonico, R. H. Cooper, R. D. Nanstad,
G. C. Robinson, and G. M. Slaughter

A review of 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.

Various sections of the report have been written and are being reviewed. A final draft will be submitted to Fossil Energy Research for their review in July.

7.2 Inspection Techniques for Wear- and Process-Resistant Coatings

R. W. McClung and G. W. Scott

7.2.1 Review and Evaluation

We recently received ANL 76-60, the Coal Technology quarterly report for March 1976, giving us a complete set of published results from ANL coating work. We have written to Dr. R. Swaroop at ANL for some additional details, including identification of commercial spray materials used in preparation of ANL specimens.

Systems appropriate for NDT studies at ORNL now appear to include:

- (1) ZrO_2 on any stainless steel substrate in one-, two-, or three-layered systems;
- (2) Cr_3C_2 (Ni Al) on 304 stainless steel and IN-800 substrates, alone and with Ni Cr Al bond coats; and
- (3) Al_2O_3 with bond coats on stainless steel substrates. Systems of types (1) and (2) appear more attractive because they represent two significantly different classes of materials.

7.2.2 Fabrication of Specimens and Standards

All coating work was delayed for approximately one week by a malfunction of the spraying system. Blanks for coating thickness standards have been completed by the machine shop and are now in dimensional inspection.

We are attempting to make an artificial crack-type defect in a ZrO_2 coating for liquid penetrant experiments. Three methods have been considered: (1) applying a tensile stress, preferably short of the elastic limit, to the substrate; (2) fatigue by bending, short of the elastic limit; and (3) bending to permanent deformation. Methods (1) and (2) appear more attractive, since either could produce a very tight crack in the coating with no permanent deformation of the substrate. The initial tensile trial was made with a coated $2 \times 4 \times 1/8$ (in.) blank. The non-standard specimen shape caused an uneven stress distribution which produced spalling instead of cracking. The test will be repeated using a coated specimen machined to a standard shape for tensile testing.

7.2.3 Thermal Testing

We are using a heat transfer computer program, "Heating 3", to simulate the effects of unbond or delamination defects in a ceramic layer - metal substrate system. This program will yield transient temperature profiles which can be used to determine the geometry and response-time requirements for thermal inspection techniques. Initial calculations will be made for IN-800 coated with ZrO_2 . The thermal data available for ZrO_2 were measured for the solid polycrystalline phase; therefore, the computed results will be somewhat conservative when applied to a porous plasma-sprayed ZrO_2 layer.

7.2.4 Electrolytic Probe

A prototype electrolytic probe has been fabricated. We have tried various electrolytes with an aluminum electrode and an IN-800 substrate to determine what voltage is developed. We have tried various organic lacquers, photo-resist (PC-board etching mask material), and a ZrO_2 -coated specimen to determine the effect of a dielectric (insulating) coating layer. The voltage developed between the aluminum electrode and IN-800 substrate is independent of the electrolyte used. The organic materials and a single coating of photo-resist do not completely insulate the electrolytic action; apparently, some ion mobility through the coating is allowed. The ZrO_2 coating develops an electrolytic voltage which is different from that developed with the IN-800 substrate; the source of this voltage has not been determined.

7.2.5 High Voltage (Corona) Probe

Preliminary experiments have been performed using the vibrator-driven tesla coil in a corona-probe leak detector as the high voltage source. Through-hole defects in a dielectric coating were simulated by drilled holes in a plastic sheet on a grounded metal plate. The probe does preferentially arc at the drilled-hole sties (as we anticipated), however, the arc stretches out as the probe moves past the defect and may arc to two defects simultaneously. This effect destroys the resolution of defect position. We are investigating methods of eliminating this effect.

Calculations show that the dielectric strength of an air-filled through-hole in a ceramic coating will be less than the strength of the intact coating. This result indicates that preferential arcing to hole-type defects should occur when the test is performed in air. Thus, immersion of the test piece in a dielectric fluid should not be necessary.

7.3 Iron and Nickel Carbonyl Formation and Prevention

J. Byrnestad and J. H. DeVan

The atomic absorption spectrophotometer has been optimized for the direct monitoring of iron and nickel carbonyl in carbon monoxide and H_2 -CO- CH_4 (51/17/32 vol %) mixtures. The optical absorbance achieved for the 284.3 nm iron line was 0.25 for carbon monoxide with 48 ppb iron carbonyl, and 0.35 for the H_2 -CO- CH_4 gas mixture with 98 ppb carbonyl. The absorbance sensitivity of the spectrophotometer is about 0.004. This means that the detection limit for iron carbonyl in these gases is about 1 ppb with absorption spectrophotometry. The minimum flow rate of the gas required through the burner is 4 l/min for the mixed gas, and about 8 l/min for the carbon monoxide. These data allow an estimate of the sensitivity of the method. For a sample of about 1 m² area, and with a steady-state carbonyl content of about 10 ppb in the gas, the rate of carbonyl formation would be about 0.1 g Fe · m⁻² · year⁻¹. By increasing the sample surface area and allowing even lower carbonyl contents in the gas, at the expense of accuracy, a lower detection limit can be assumed to be about 0.01 g Fe · m⁻² · year⁻¹.

We were asked by Dr. McMichael at PERC to evaluate the efficiency and useful lifetime of the charcoal trap which will be used to purify the gas mixture fed into our reaction chamber. This information is of interest since charcoal is being used to purify the feed gas to the Synthane methanator. To increase the relevance of our evaluation, Dr. McMichael has supplied us with charcoal from the same stock as used in the Synthane Plant. We have designed a simple system to measure the carbonyl absorption capacity of our trap using the one-line atomic absorption spectrophotometer, which will not significantly interfere with the carbonyl formation study.

We expect to complete construction of the reaction rate apparatus and associated gas supply system by the end of July.

7.4 Failure Analysis of Materials and Components

D. A. Canonico, D. P. Edmonds, and T. K. Roche

This project is devoted to the posttest examination of components that have been removed from service in coal liquefaction processes. The goal is the avoidance of failures in commercial coal conversion systems.

The examination of the pressure let-down trim set from the Synthoil PDU at PERC is almost completed. We have made a plexiglass model of the valve and are studying the flow patterns of liquids past the trim set. This should give us more insight into the cause of premature failures of the trim sets and the effects of slight misalignments on flow patterns.

Investigations into the failure of the type 316 stainless steel heater tube from Project Lignite are underway. A report describing the work done at ORNL is being prepared and will be forwarded to Fossil Energy Research in August.

7.5 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.

During this period, refractory and liner design studies were intensified, and emphasis was given to obtaining improved data on temperatures and temperature distributions in the vessel-liner-refractory composite structures. Material selections and design details are being considered in greater depth and preparations are being made to do refined heat transfer calculations. In addition, the initial conceptual design for the HYGAS gasifier vessel was completed and an overall drawing was made.

Under Task 1, in addition to the completion of the initial preliminary vessel design, work was continued on the closure in the top head for the Synthane gasifier vessel. There are two designs being considered. One makes use of toggle bolts for hold-down and transfer of load to the vertical prestressing tendons. Disadvantages of this design are tendon configuration requirements and bearing limitations at the tendon bearing plate and concrete interface.

The second closure design makes use of an annular steel box beam around the opening, with the beam being cast integrally with the concrete vessel. This design reduces tendon bearing stress requirements, allows better manway positioning, and reduces the overall height of the vessel. The disadvantages in this case are construction difficulties and higher cost. Studies on the two closures are being continued; a selection is to be made during the next reporting period.

The finite element computer program described in the report for last period was used to examine two Synthane vessel configurations. A cold liner was assumed in each case. The configuration differences were associated with the two closure designs under consideration, and effects of internal pressure, external prestress, and changes in prestressing forces in the head region alone were calculated.

Other simplified computations were made using closed-form thick-walled cylinder equations to examine hot liner designs. Various thicknesses of refractory lining and insulating concrete between the steel liner and vessel were considered. Four loading cases were used; these were: (a) prestressing forces only; (b) prestress and internal pressure; (c) prestress and thermal stress; and (d) prestress, internal pressure, and thermal stress. These studies gave allowable overall thermal gradients, wall thickness requirements, and circumferential prestress force requirements.

Under Task 2, examinations were initiated on circumferential prestressing methods and configurations as well as preloads and preloading techniques. Both the wire winding and the segmented method are under current consideration. Interactions of manway penetrations and prestress arrangements are of particular concern.

Present investigations of discontinuities, as listed under Task 3, are being conducted in conjunction with Task 1 studies.

Studies of refractory lining materials and properties under Task 4, were initiated as mentioned above. Attachment methods and other factors are to be included in these studies. The thermal analyses being undertaken will address, in detail; temperatures, temperature distributions, and vessel cooling requirements in the region of the liner-vessel interface. The hot liner concept was selected as the reference design, although the cold liner concept will be also be analyzed.

7.6 Other Related Work

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

Nothing to report.

8. COAL-FUELED MIUS

A. P. Fraas and W. R. Nixon

This project for analysis, design, and demonstration of a concept utilizing a fluidized-bed coal combustion system as a heat source for a gas turbine generator suitable for applications in Modular Integrated Utility Systems (MIUS) is carried out under the ORNL-HUD-MIUS Program within the Energy Division. Work is supported by the U.S. Department of Housing and Urban Development under HUD Interagency Agreement No. 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.

8.1 Furnace Procurement

A request for bids on fabrication of the fluidized-bed furnace and tube bundles was sent to 17 vendors on June 10, 1976. Initial responses from these firms indicate that five are preparing bids, one is interested but might be unable to meet the July 22 deadline, and ten will not bid.

The specifications for the regenerator for transferring heat from the flue gas to the combustion air have been written and are ready for internal review.

8.2 Turbine-Generator Unit

Specifications for the recuperator modules were completed, but procurement of these modules and placement of the turbine modification contract with AiResearch are awaiting receipt of additional funds. Analysis of the turbine electronic control system was initiated as a preparatory step to instrumentation and control system design.

8.3 Site Preparation

Design of building modifications required for installation of the system continued. Interior partition wall design was completed and work began on drawings for relocation of existing equipment.

8.4 Cold Flow Tests of a Fluidized Bed

The natural frequency of a full-length furnace tube was found to be over 15 Hz. Comparison with vibration frequencies of 10 to 50 Hz over the operating range of fluidizing velocity (previously measured in the 4-ft. cold flow model) indicates that use of tube supports to dampen vibration would be advisable.

8.5 Coal Metering and Feed System

Endurance tests of the flow splitter feed system and on a prototype furnace coal feed nozzle were continued. No problems have been observed during total operating times of 133 hr on the feed system and 53 hr on the nozzle.

Disappointing results were obtained from coal feed tests with the Iron Fireman feeder. Maximum coal feed rate was only 250 lb/hr because of excessive recycling of transport air along the screw feeder. This feeder will not be adequate to deliver the maximum required furnace feed rate of 470 lb/hr. A Virba-screw coal feeder was received and is being installed for testing.

8.6 Coal Feed Preparation

Specifications for the coal preparation plant were reviewed and found suitable for system procurement. The system will operate on stoker-size coal but will retain the capability to prepare run-of-mine coal.

8.7 Supplemental Studies

The quantities of solid materials to be handled, and estimated handling costs, for a typical MIUS installation were found to be so small that it is questionable whether the economic, social, and environmental impact will be of enough significance to merit a study of the original scope. Further review of the scope of work and discussions with ERDA-FE are planned.

The subcontract with Fluidyne Engineering Corporation to conduct corrosion tests on air-cooled tubes has been approved. The test program is planned to obtain 500-hr exposure time at a bed temperature of 1650°F (900°C) on 8 tube materials. Each of the 16 tubes to be tested will consist of three 6-in. (153 mm) long specimens welded together. The 48 tubular specimens are on hand and ready to be shipped to Fluidyne.

The subcontract for heat transfer test in the Fluidyne furnace has been arranged as a supplement to the corrosion subcontract. The scope of the heat transfer test program includes 3 series of tests,

each with 8 values of fluidizing velocity from 0.8 to 2.2 ft/sec (0.24-0.67 m/sec). Fabrication of the instrumented tube is in progress at ORNL.

A draft of the report on the reliability of coal-fired gas turbine systems was completed and reviewed. Calculations for alternate operating conditions are nearly completed and preparation of the report on system performance with alternate cycles is underway.

Calculations on electrical heating and cooling loads and compilation of cost data are in progress for the economic comparison of the closed cycle gas turbine with two alternative energy systems.

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195. Robert Hangebrauck, National Environmental Research Center,
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201. S. G. Wellborn, Manager, Feedstocks Development, E.I. du Pont
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202. Tetra Tech, Inc., 1911 N. Ft. Myer Drive, Suite 601, Arlington,
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203-204. Jack Gillespie, UCCND, P.O. Box 1410, Paducah, Kentucky 42001

205. Jerry D. Kennedy, Sandia Laboratories, P.O. Box 5800,
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206. L. B. Batta, Coalcon, P.O. Box 44, Tonawanda, N.Y. 14150

207. Emmett J. Ferretti, Synthetic Fuels Department, Dravo Corp.,
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208. O. L. Anderson, Institute of Geophysics & Planetary Phys.,
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209. S. K. Chakrabartty, Alberta Research Council, Fuel Sciences Div.,
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210. E. H. Reichl, Vice-President, Consolidation Coal Co., Library,
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