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UTILIZATION OF COAL ASSOCIATED MINERALS

Quarterly Report No. 7

April 1 - June 30, 1979

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OBJECTIVE AND SCOPE

The purpose of this research program is to examine the effects of coal mineral materials on coal waste by-product utilization and to investigate new and improved methods for the utilization of waste by-products from the cleaning, combustion and conversion processing of coal. The intermediate objectives include: (1) the examination of the effects of cleaning, gasification and combustion on coal mineral materials; and (2) the changes which occur in the coal wastes as a result of both form and distribution of mineral materials in feed coals in conjunction with the coal treatment effects resulting from coal cleaning or either gasification or combustion.

SUMMARY OF PROGRESS TO DATE

At this time, approximately half-way through the contract period, progress has been as scheduled in most areas although somewhat behind in a few. The net effect does not appear to preclude meeting schedule deadlines, although that assumes that no major problems occur with equipment or instrumentation necessary for the remainder of the contract.

This report continues the documentation of detailed characterization of both raw and prepared fractions of coal feeds, products, and refuse from the preparation plant in northern West Virginia. It also documents the installation and initial operation of two recently acquired instruments, i. e. a Scanning Electron Microscope and an X-Ray Powder Diffractomer. These units are both operable at this time, although quantitative/semi-quantitative capabilities are still being refined.

Other areas where significant work was achieved include froth flotation of the Pittsburgh sample and the quantitative determination of kaolinite using IR. This work is documented herein. Major and minor elemental analysis of samples is on-going and should be reported in the next report.

Work was continued in the quarter to determine the utilization of Pope, Evans and Robbins fluidized bed combustion by-products in fired structural materials.

Arrangements were made during the quarter to obtain samples of the coal used in the solvent refined coal conversion process (SRC II). The coal samples will be obtained from the District 4 coal mine and cleaning plant which produces the coal used in the SRC II conversion process. Arrangements were also made during the quarter to obtain samples of the waste by-product produced by the SRC II conversion process at Fort Lewis, Washington.

DESCRIPTION OF TECHNICAL PROCESSES

Coal Preparation Pilot Plant

Bids for the construction of the heavy media sump have been opened during the quarter. It is estimated that the sump will be completed by the end of October. This reservoir is designed to provide for the recirculation of media for both the Wemco drum heavy media separator and the 8" cyclone.

Operational tests were performed on the jig during the past quarter. Good separation was obtained; however, the refuse gate control device would not function properly. While not absolutely essential to the satisfactory operation of the jig, the control device allows the variation of effective gravities of separation without altering other operating parameters. Work is continuing to restore proper function to the gate control.

Also during the past quarter, floor drains were installed for the jig and the concentrating table. A slurry/distributor tank and platform were also constructed to feed the table. Water supply to the distributor and table are provided by a hose from the elevated head tank. The following photograph (Figure 1) shows the present condition of the pilot plant and sample storage area. In the near foreground crushing and sampling equipment can be seen and the coal cleaning equipment is shown in the background.

Froth Flotation Tests

Work on the froth flotation section of the contract continued with the bench-scale flotation testing of samples from the District 3 (Pittsburgh

Seam) preparation plant. The results of these tests are reported in Table 1. The flotation test parameters, proximate analysis, sulfur breakdown and Btu are presented in the table. From this data, at least one sample will be chosen from the district to be submitted for mineralogical analysis. The criteria used for sample selection will be based on the commercial value of each separation.

Representative 500 gram portions of coal were used in all flotation tests. These samples represented a screen fraction of the raw feed to the preparation plant. The percentage of solids in the flotation feed cell was held constant in the individual trials at 9.1%. The pH of the slurry ranged from 5.5 to 5.8 and the slurry temperature was 23°C ± 2°C. A 15 minute conditioning period was followed by air induction and a 1 minute collection of the froth overflow. Both the product and refuse were vacuum-filtered and oven-dried at 100°C for 24 hours. The weights of the product and refuse fractions were recorded and a fraction of each was submitted for proximate analysis, sulfur forms, and Btu.

In Table 1 the results of the froth flotation tests on the -50 mesh Pittsburgh seam samples are reported. The best overall product was obtained at an MIBC level of 0.23 pounds per ton. At a higher level of MIBC (0.53 pounds per ton) the product yield decreased but the ash and total sulfur increased relative to the product produced using 0.23 pounds per ton of MIBC. The use of either a pyrite depressant or a wetting agent appear to produce no beneficial effect in froth flotation of the Pittsburgh sample.

During the next quarter, chemical analyses will be performed on a few remaining samples and mineralogical evaluation will begin on selected product and refuse fractions from the froth flotation tests.

Characterization of Coal Samples

The following section is a review of progress in the chemical, physical, and mineralogical characterization of samples from Facet I.

Chemical Characterization

Additional chemical characterization data have been determined for each group of twenty-five size and gravity fractions of the Pittsburgh coal (see Table 2). This data, which includes the recently completed ultimate analyses for the Pittsburgh Seam (Table 2) is being added to the computerized data file for subsequent data processing in combination with other sample parameters.

Sample fractions were prepared for analysis using the conventional procedure previously described (Quarterly Report No. 5), producing twenty-five individual components from the plus 100 mesh coal. The minus 100 mesh sample and the raw coal head sample were also analyzed and are reported in Table 2 to facilitate comparisons.

Table 2 contains the ultimate analyses for the Pittsburgh sample. For this data several trends are noticeable within size fractions, i.e. C, H, and N decrease as gravity increases while S increases. The O content does not show consistent trends with higher concentrations occurring somewhat at random. Further examination of this data will be reported in the future.

Mineralogical Characterization

During this quarter a Cambridge Stereoscan 150 Scanning Electron Microscope was acquired by the Coal Research Bureau. This instrument, operated in conjunction with a Kevex X-Ray Dispersion Unit (acquired but not yet operational), will be utilized to study the nature and distribution of coal-associated minerals. The capability provided by the S.E.M. will enable the detailed study of the morphology (size, shape, etc.) of single particles (crystals) within the coal matrix, and together with the Kevex Unit, elemental ratio data may be determined. The elemental concentrations obtained may then be utilized to formulate mineralogical composition on a semi-quantitative basis (eg. high concentrations of iron and sulfur indicative of pyrite occurrence).

The S.E.M. has been set up and is now functional. Personnel are in the process of becoming familiar with its routine operational parameters. The Kevex is expected to be installed and in operation in the next quarter.

Other areas of this section report progress achieved in kaolinite determination using IR and continued progress in achieving X-ray Powder Diffraction capabilities.

Petrographic Analysis - Work showing the mass balance of both macerals and minerals through the preparation plant studied is nearly complete and will be reported next quarter. The important contribution of this study will be to follow mineral occurrences such as microscopic pyrite types or carbonate types (fracture filling, cell filling, etc.) through the plant. This will provide valuable information to be coupled with the mineral species determination by X-ray powder diffraction and I. R. spectroscopy.

Infrared Analysis - This quarter the quantitative distribution of kaolinite was determined in the float-sink fractions of the Pittsburgh coal. Kaolinite concentration in the low-temperature ash (LTA) of the coal studied was determined from infrared spectra utilizing the baseline method.[1] The absorption band chosen for quantitative analysis occurred at 910 cm^{-1}. The incident radiation (I_o) was measured by drawing a straight line tangential to the shoulders of the band. The transmitted radiation (I) was measured at the point of greatest absorbance (Figure 2). The value of log I_o/I, or absorbance, was then plotted against kaolinite concentration. A kaolinite calibration curve was thus prepared utilizing five synthetic mixtures containing various concentrations of American Petroleum Institute kaolinite standard No. 5 (Bath, S.C.), (Figure 3). Percentages of kaolinite in the LTA samples studied were then calculated from this curve.

For the Pittsburgh coal, kaolinite appeared to be concentrated in the 1.30 float of each size fraction. See Table 3. In the 1.30 float group, as well as in the other gravity fractions of the Pittsburgh coal, the percentage of kaolinite appeared to be a function of specific gravity and not particle size. Kaolinite concentration is shown to successively decrease with increasing specific gravity, with the lowest concentrations occurring in the 1.80 float and 1.80 sink fractions.

A general trend for the qualitative distribution of quartz in the float-sink fractions of the coal studied was also observed. Quartz was not detectable by IR spectroscopy in the 1.30 and 1.40 float fractions of the Pittsburgh coal, but was concentrated in the 1.80 float and 1.80 sink fractions of the coal.

In summary, kaolinite was generally concentrated in the lighter gravity fractions (1.30 and 1.40 floats), with the lowest concentrations occurring in the sink fractions. Distribution did not appear to be a function of particle size. Quartz, as expected, was concentrated in the heavier gravity fractions (1.80 float and 1.80 sink), with minimal concentrations in the ligher fractions.

X-Ray Powder Diffraction (XRPD) - The installation of the Philips APD 3501 X-Ray unit was completed during quarter seven.

Calibration of the APD is currently in progress. Standard mineral samples (calcite, pyrite, quartz, gypsum, illite, montmorillonite, rutile, hematite, and other coal associated minerals) are being analyzed on the APD and the results compared with mineralogical data available from the JCPDS Inorganic Powder Diffraction File.

Combined samples using various weight percentages of the standard minerals will also be analyzed to enable the preparation of standard mineral curves which will show the percent mineral occurring in the sample in accordance with the peak intensities produced. These curves will be utilized in determining the percent mineral present in the low temperature ashed coal, on a whole coal bais.

Analyses of low-temperature ashed coal using the standard mineral curves will be presented in future reports.

References

1. O'Gorman, J. V., "Studies of Mineral Matter and Trace Elements in North American Coals," Ph. D. Dissertation, Pennsylvania State University, 1971, p. 122.

Sample Collection

Arrangements were made during the quarter to obtain samples of coal feed and by-products from the solvent refined coal conversion process pilot plant (SRC II) of the Pittsburgh and Midway Coal Mining Company at Fort Lewis, Washington for evaluation under the subject contract. Tentative sampling dates are November 13 and 14. Through conversations with plant officials it has been determined that "vacuum bottoms" waste materials are the only significant waste by-product from the SRC II process. Therefore, it is anticipated that approximately four 55 gallon steel drums of coal feed will be obtained as well as eight 55 gallon steel drums of the waste material. Sample containers are being fitted with valves and seals to allow evacuation of air and re-filling with nitrogen after filling with sample to reduce oxidation of the coal materials during transit and storage.

The District 4 coal mine and cleaning plant which produces the coal used in the SRC II conversion process at Fort Lewis has been contacted and arrangements made to obtain representative samples of raw coal, refuse and cleaned product. The tentative sampling date for this preparation facility is the second week in October. It is anticipated that this preparation plant will be sampled in the same manner as the previous plant under the subject contract. Approximately eight 55 gallon steel drums of raw coal and refuse as well as 4 barrels of cleaned coal will be obtained. Flow sheets for both the preparation plant and the conversion facility will be provided when available.

Coal By-Product Utilization Studies

Samples of spent limestone bed ash and flyash from the carbon burn-off cell were obtained from the fluidized bed combustion process at Rivesville, West Virginia. The samples were taken during a steady state run and to insure representativeness the samples were taken at intervals over a four hour period. During this steady state run, the feed coal size was -1/2 inch +1/4 inch and the feed limestone size was -6 +16 mesh. It is thought that representative samples of the by-products were obtained during this steady state run. However, different operating conditions may produce by-products having slightly different characteristics.

The chemical analysis of the spent limestone bed ash and flyash from the carbon burn-off cell are presented in Table 4.

The median particle size of the spent limestone bed ash is around 10 mesh (1.7 mm) and the median particle size of the flyash from the carbon burn-off cell is approximately 200 mesh (0.08 mm).

In order to determine the suitability of the spent limestone bed ash as an aggregate in fired structural materials, test specimens in the form of a standard building brick were chosen for this study. The composition and physical properties of the flyash brick are presented in Table 5. Control flyash brick were produced using compositions which had been determined previously during pilot scale operations conducted by the Coal Research Bureau. The ingredients were mixed in a mix-muller, formed in a hydraulic toggle press, dried in a humidity drier, and fired in a gas-fired shuttle kiln. Firing conditions were carefully monitored both by thermocouples and also pyrometric cones which indicate the combined effect of temperature and time sometimes called "heat work".

All of the flyash brick were tested in accordance with American Society for Testing and Materials designation C67 "Methods of Sampling and Testing Brick" and evaluated in accordance with ASTM designations C62 "Specifications for Building Brick" and C216 "Specifications for Facing Brick."

The flyash was selected as being representative of a major coal seam and is designated as Southern West Virginia flyash.

The spent limestone bed ash was crushed and screened through a 20 mesh sieve. This is the same sieve that the standard bottom slag was screened through. The minus 20 mesh bed ash was utilized in this study.

The composition and properties of the flyash brick are presented in Table 5.

The standard bottom slag was replaced with bed ash at levels of 11.24, 21.24, and 31.24 percent when using the Southern West Virginia flyash.

Upon completion of the drying cycle the flyash brick containing bed ash exhibited cracks and fissures. It is thought that the cracks and fissures in the flyash brick were caused by the free lime in the bed ash hydrating and expanding after being wetted during the mixing cycle.

Sodium hydroxide which is frequently beneficial in the flyash brick process was tried but did not reduce or eliminate the cracks and fissures.

A batch was wetted and left standing for ten (10) minutes in order to allow the free line in the bed ash to hydrate and expand before being pressed but this did not reduce or eliminate the cracks and fissures.

The standard bottom slag was used with an equal percentage of bed ash in order to minimize the effects of the bed ash; however, cracks and fissures were still observed upon completion of the drying cycle.

From Table 4 it can be noted that the Pope, Evans and Robbins flyash has a loss on ignition (LOI) of 36.0 percent. Previous research, performed by the Coal Research Bureau under contract number 14-01-0001-483 to the Office of Coal Research, has indicated that a flyash having a loss on ignition (LOI) in excess of 10-12 percent would require some special modifications, such as calcination, to eliminate the weak porous structure which commonly results from bulk weight loss after carbon burn-off in high carbon flyashes. Since calcining the PER flyash would be energy-intensive as well as uneconomical, research on utilizing PER flyash in fired structural materials was not investigated.

The utilization potential of Pope, Evans and Robbins spent limestone bed ash as an aggregate in fired structural products has been shown to be unfeasible because of cracks and fissures in the structural products. The cracks and fissures are caused by the free lime in the bed ash hydrating and expanding after being wetted during the mixing cycle.

TABLE 1

FROTH FLOTATION RESULTS
-50 M Pittsburgh

Sample Description	Reagents[1]	Yield	Moisture	Ash[2]	Volatile[2] Matter	Fixed[2] Carbon
Head Sample	---------	---	0.7	19.6	34.1	46.3
Flotation Product	MIBC-0.53	72%	0.4	11.3	34.9	53.9
Flotation Refuse	MIBC-0.53	28%	0.3	40.1	28.3	31.6
Flotation Product	MIBC-0.23	75%	0.5	8.6	35.6	55.8
Flotation Refuse	MIBC-0.23	25%	0.2	50.9	25.1	24.0
Flotation Product	MIBC-0.23 Pyrite Depressant	70%	1.0	9.3	33.7	57.0
Flotation Refuse	MIBC-0.23 Pyrite Depressant	30%	1.0	37.2	28.3	34.5
Flotation Product	MIBC-0.23 Wetting Agent	76%	0.9	9.9	36.0	54.1
Flotation Refuse	MIBC-0.23 Wetting Agent	24%	1.0	35.6	28.4	36.8

1. lb./ton
2. percentage of whole coal, dry basis

TABLE 1 (Continued)

FROTH FLOTATION RESULTS
-50 M Pittsburgh

Sample Description	Total[2] Sulfur	Sulfate[2] Sulfur	Pyritic[2] Sulfur	Inorganic[2] Sulfur	Organic[2] Sulfur	BTU
Head Sample	3.26	----	----	----	----	------
Flotation Product	3.14	0.02	1.60	1.62	1.52	13,350
Flotation Refuse	3.65	0.03	2.63	2.66	0.99	------
Flotation Product	2.85	0.02	1.37	1.39	1.46	13,776
Flotation Refuse	4.45	0.05	3.78	3.83	0.62	------
Flotation Product	2.95	0.05	1.31	1.36	1.59	13,359
Flotation Refuse	3.95	0.08	2.83	2.91	1.04	------
Flotation Product	3.01	0.10	1.47	1.57	1.47	13,568
Flotation Refuse	3.78	0.11	2.67	2.78	1.00	8,845

2. percentage of whole coal, dry basis

TABLE 2

ULTIMATE ANALYSIS
(PITTSBURGH SEAM - SIZED, GRAVITY FRACTIONS)

Sample	C	H	O	N	S
+1" Coal					
Float 1.3	79.70	5.28	4.17	1.51	2.45
1.3 x 1.4	74.69	4.97	3.60	1.25	3.72
1.4 x 1.6	66.86	4.38	0.51	1.09	3.80
1.6 x 1.8	52.64	3.35	1.21	0.81	3.10
Sink 1.8	17.72	1.24	2.39	0.27	7.55
1" x 1/4" Coal					
Float 1.3	78.39	5.35	7.34	0.65	2.22
1.3 x 1.4	72.54	4.94	5.86	0.54	3.92
1.4 x 1.6	62.42	4.13	2.53	0.41	5.62
1.6 x 1.8	51.59	3.16	1.38	0.04	6.64
Sink 1.8	15.64	1.25	3.44	0.10	6.49
1/4" x 8 Mesh Coal					
Float 1.3	78.41	5.28	7.36	0.76	2.15
1.3 x 1.4	62.29	4.42	4.99	0.52	4.72
1.4 x 1.6	62.60	4.15	3.49	0.49	5.85
1.6 x 1.8	54.06	3.28	0.96	0.20	6.89
Sink 1.8	15.78	1.19	1.11	0.10	8.67
8 x 28 Mesh Coal					
Float 1.3	78.09	5.29	9.37	0.48	2.04
1.34 x 1.4	72.19	4.86	7.51	0.34	3.43
1.4 x 1.6	65.46	4.46	3.40	0.48	5.49
1.6 x 1.8	54.03	3.10	0.45	0.75	6.82
Sink 1.8	15.83	1.21	0.29	0.24	9.92
28 x 100 Mesh Coal					
Float 1.3	78.80	5.19	5.95	1.78	2.11
1.3 x 1.4	76.83	4.99	4.09	1.30	2.76
1.4 x 1.6	70.31	4.47	2.71	1.24	4.09
1.6 x 1.8	58.70	3.26	0.34	0.71	5.99
Sink 1.8	17.25	0.94	2.30	0.26	11.13
Minus 100 Mesh Coal					
	64.36	4.14	5.29	0.24	3.60
Raw Coal Head					
	72.38	4.68	3.38	1.25	3.01

*Percent element on a moisture free whole coal basis (for each fraction).

TABLE 3

PITTSBURGH COAL

Sample Description	Absorbance ($\log I_o/I$)	Percent Kaolinite
+1", 1.30 Float	.166	22
1 x 1/4, 1.30 Float	.194	26
1/4 x 8 Mesh, 1.30 Float	.168	22
8 x 28 Mesh, 1.30 Float	.201	27
28 x 100 Mesh, 1.30 Float	.219	29 AVE = 25%
+1", 1.40 Float	.107	14
1 x 1/4, 1.40 Float	.161	21
1/4 x 8 Mesh, 1.40 Float	.125	16
8 x 28 Mesh, 1.40 Float	.185	24
28 x 100 Mesh, 1.40 Float	.184	24 AVE = 20%
+1", 1.60 Float	.066	9
1 x 1/4, 1.60 Float	.099	13
1/4 x 8 Mesh, 1.60 Float	.097	13
8 x 28 Mesh, 1.60 Float	.131	17
28 x 100 Mesh, 1.60 Float	.170	22 AVE = 15%
+1", 1.80 Float	.085	11
1 x 1/4, 1.80 Float	.083	11
1/4 x 8 Mesh, 1.80 Float	.073	10
8 x 28 Mesh, 1.80 Float	.078	10
28 x 100 Mesh, 1.80 Float	.101	13 AVE = 11%
+1", 1.80 Sink	.114	15
1 x 1/4, 1.80 Sink	.116	15
1/4 x 8 Mesh, 1.80 Sink	.088	12
8 x 28 Mesh, 1.80 Sink	.079	10
28 x 100 Mesh, 1.80 Sink	.060	8 AVE = 12%

TABLE 4

CHEMICAL ANALYSIS OF FLUIDIZED BED PROCESS
(POPE, EVANS AND ROBBINS) BY-PRODUCTS

	Bed Ash	Flyash
SiO_2	22.40	31.20
Al_2O_3	5.97	8.36
Fe_2O_3	3.00	11.70
TiO_2	0.35	0.44
CaO	37.10	3.89
MgO	1.25	0.97
Na	0.16	0.21
K	0.68	0.64
S	8.63	3.87
LOI	4.4	36.0
C	2.99	32.18

TABLE 5

COMPOSITION AND PROPERTIES OF SOUTHERN WEST VIRGINIA FLYASH BRICK
WITH POPE, EVANS AND ROBBINS BED ASH

	1	2	3	4	5	6	7	8	9
Southern West Virginia Flyash	76.66	86.66	76.66	66.66	86.66	76.66	66.66	76.66	76.66
Bottom Slag - 20 Mesh	21.24	-	-	-	-	-	-	-	10.62
PER Bed Ash - 20 Mesh	-	11.24	21.24	31.24	11.24	21.24	31.24	21.24	10.62
Silicate	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1
Silicate Grade	47	47	47	47	47	47	47	47	47
Water	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0
Sodium Hydroxide	-	-	-	-	1.0	1.0	1.0	-	-
Firing Rate, °F./Hr.	119	119	119	119	119	119	119	119	119
Final Firing Temp., °F	2100	2100	2100	2100	2100	2100	2100	2100	2100
Cone	3	3	3	3	3	3	3	3	3
% Abs. 24 Hr. Soak	11.30	15.85	20.10	23.96	16.22	19.08	25.64	19.05	16.12
% Abs. 5 Hr. Boil	14.98	19.38	23.58	27.68	20.02	22.40	27.72	22.62	19.67
Saturation Coefficient	0.75	0.82	0.85	0.86	0.81	0.85	0.92	0.84	0.81
Apparent Porosity, %	26.58	32.08	36.99	41.31	32.43	35.55	40.95	35.54	32.18
Bulk Density, Lbs./Cu. Ft.	110.8	103.3	97.8	93.1	101.1	99.0	92.2	98.0	102.1
% Shrinkage	3.2	3.8	1.3	-1.4	3.1	0.7	-2.2	1.7	1.7
Unfired Compressive Strength, P.S.I.	304	66	40	55	96	50	55	41	93
Fired Compressive Strength, P.S.I.	7164	4363	1519	691	3806	1679	785	1760	2404
Suction, gms. H_2O/30 Sq. In./Min.	103	126	178	268	146	185	292	184	163

Note: Batch 8 had a ten (10) minute interval between wetting and pressing.

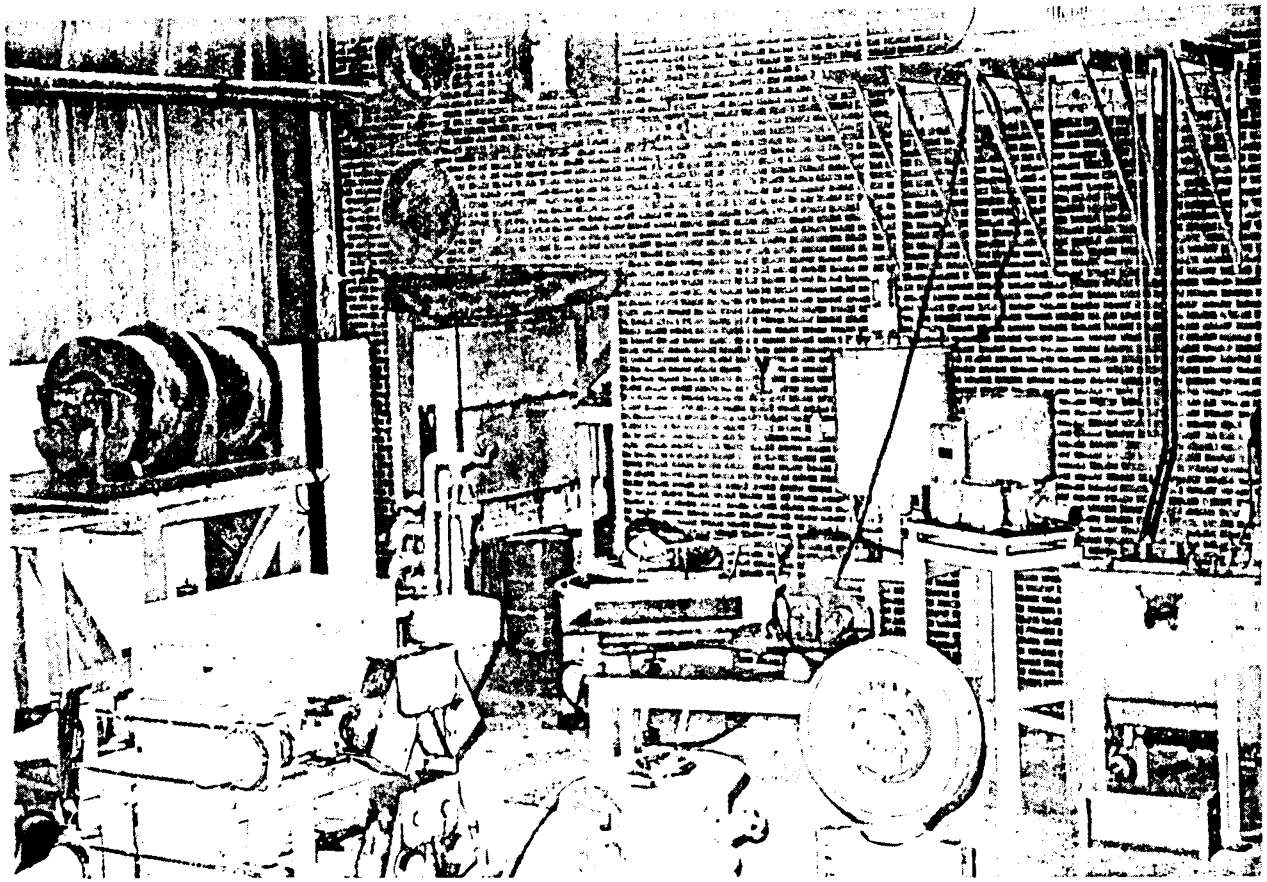
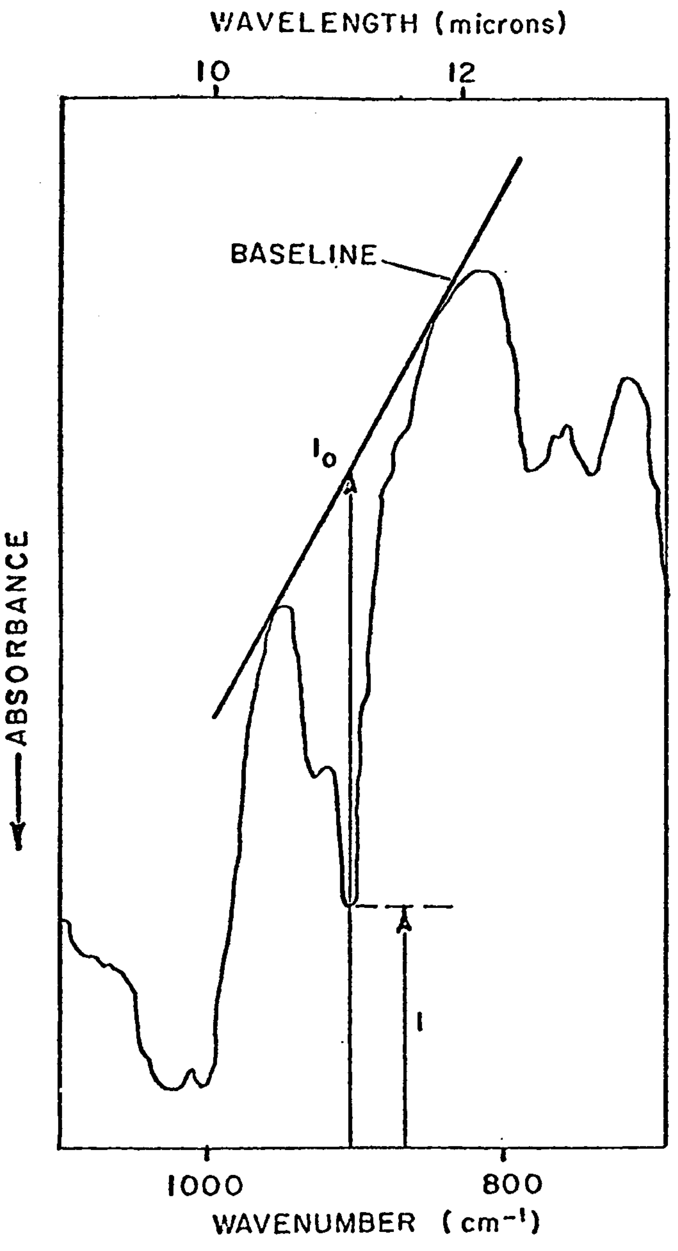


FIGURE 1. Coal Preparation Equipment - left to right: heavy media drum separator, 3" cyclone, concentrating table and distributor, elevated head tank and jig.

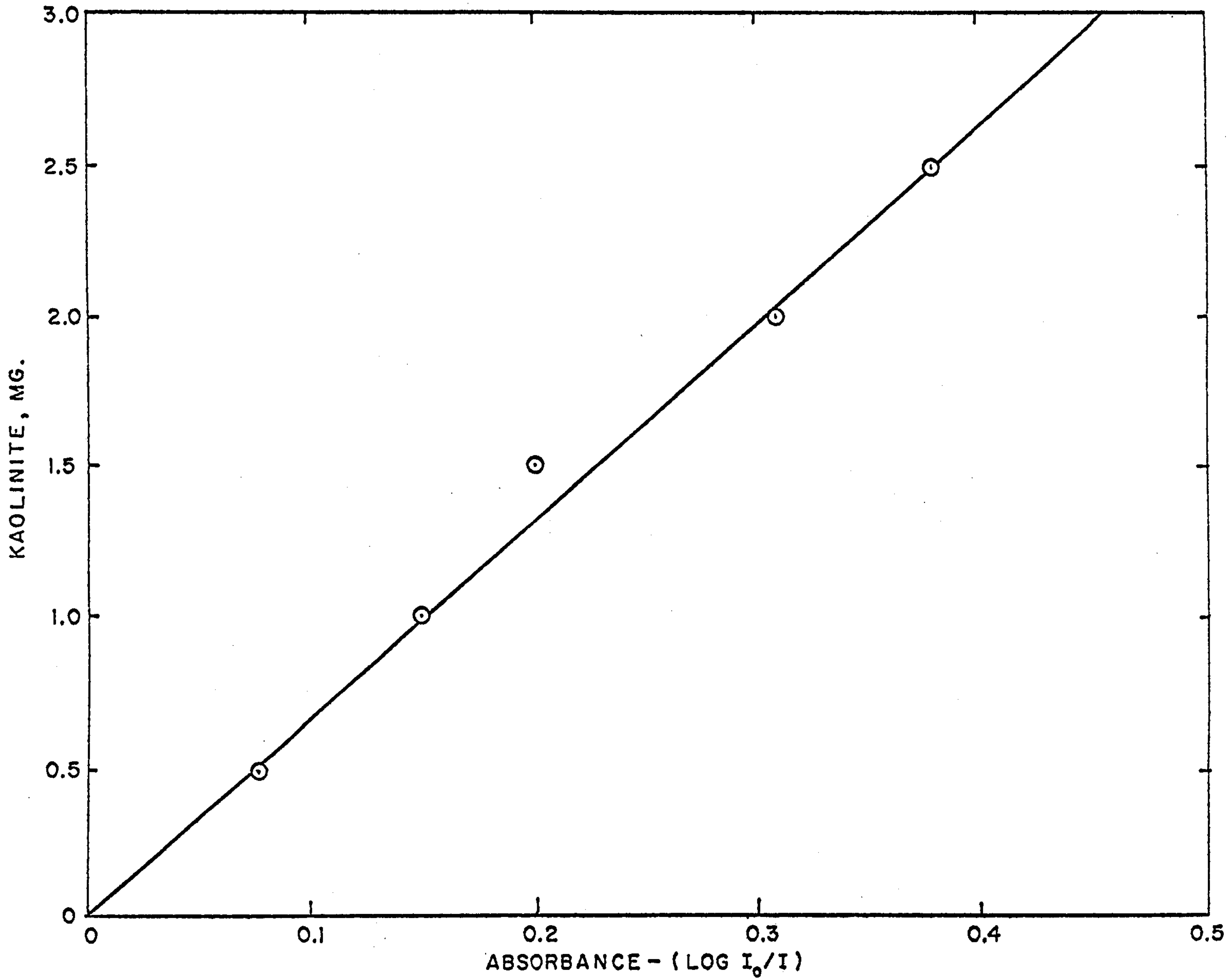
FIGURE 2



METHOD OF DRAWING BASELINE
FOR PLOTTING COMPOSITION VERSUS LOG I_o/I

FIGURE 3

Kaolinite Calibration Curve Utilizing The 910 CM^{-1} Absorption Band



APPENDIX A

UTILIZATION OF COAL ASSOCIATED MINERALS

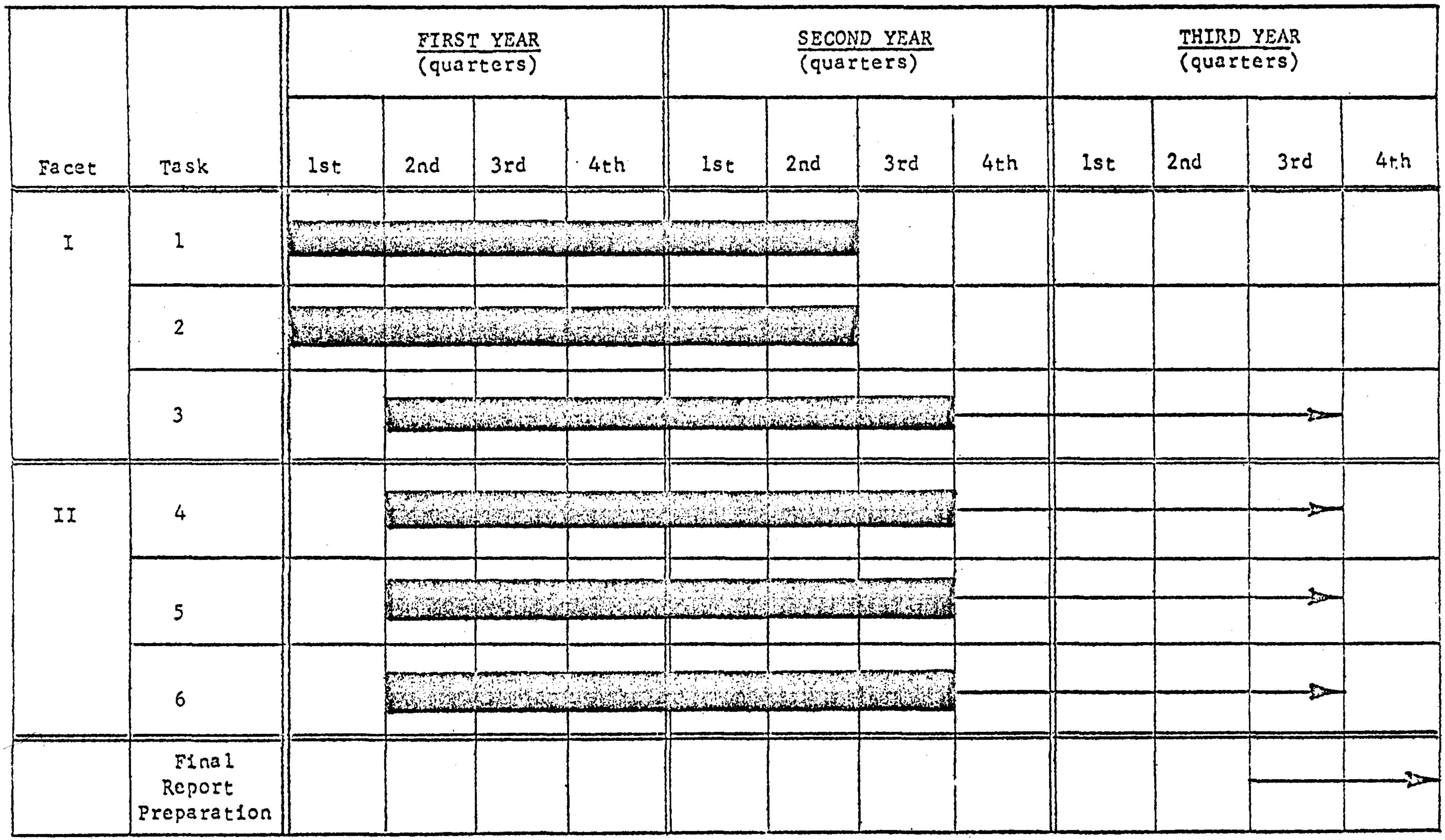
FINANCIAL REPORT

7TH QUARTER, APRIL 1, 1979 - JUNE 30, 1979

Expenditures This Quarter	
Personal Services	$6,195.40
Equipment, R & A	198.13
Current Expense	
Overhead	3,440.62
Supplies	2,112.03
Travel	450.00
Benefits	944.80
TOTAL EXPENDITURES 7TH QUARTER	$13,340.98
TOTAL EXPENDITURES TO DATE	$178,299.34
TOTAL CONTRACT AWARD TO 9/24/80	$378,000.00
CONTRACT BALANCE	$199,700.66

UTILIZATION OF COAL ASSOCIATED MINERALS

West Virginia University



Work Schedule

☆U.S. GOVERNMENT PRINTING OFFICE: 1980—640-258/2036/ REGION NO. 4