FE-2722-6
Distribution Category UC-90i

THE EFFECTS OF MINERALS ON COAL BENEFICATION PROCESSES

QUARTERLY REPORT NO. 6

January 1 - March 31, 1979

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June 15, 1979

PREPARED FOR THE UNITED STATES
DEPARTMENT OF ENERGY

Under Contract No. EF-77-S-01-2722

ACKNOWLEDGMENTS

The authors would like to acknowledge the services and skills of a number of people whose efforts are not otherwise documented in this report: Charles R. McFadden for a number of the illustrations presented; Kenneth K. Humphreys who wrote the Calcomp Program used to produce the computer-drawn washability curves; Edward J. Simcoe who modified the program for our needs; Larry L. Nice and the analytical group at the Coal Research Bureau for extensive chemical testing; and Sally See and Martha Fekete who typed and proofed this report. The authors would also like to thank Dr. John J. Renton of the West Virginia Geological Survey for his assistance in verifying the feasibility of a special X-ray sample preparation technique.

OBJECTIVE AND SCOPE

The purpose of this research program is to examine the effect of coal cleaning and preparation on the distribution of mineral materials in coal and the influence of the mineral materials on the coal cleaning operation. The research program will involve the examination of, for coal mineral materials: (1) the natural occurrence and distribution of mineral materials in run-of-mine coal, (2) the changes in these characteristics during cleaning and preparation, (3) the specific effects of coal mineral materials on individual cleaning and preparation processes, and (4) improved methods for controlling their distribution.

In order to accomplish these objectives samples will be obtained from three commercial coal preparation plants which are: (1) handling coal from major (by volume) coal seams, (2) handling coal most likely to be used in future large scale coal conversion processes (for example, the BI-Gas process), and (3) using a range of different types of modern cleaning methods. At least one of these plants shall process a coal likely to be used as a feed to a D.O.E.-supported conversion process or similar to a type of coal likely to be used.

SUMMARY OF PROGRESS TO DATE

Samples of feed coal, clean coal product, and refuse from three commercial coal preparation plants have been taken, processed to yield samples for chemical and mineralogical analysis, physically characterized using screens and float-sink gravity separations, and submitted for extensive chemical analyses. A large amount of data has been collected from this work and is being reported, in update-fashion, as it is available.

This report presents a variety of data collected during characterization of the Pocahontas No. 3 sample (southern West Virginia) collected at the District Seven Preparation plant. Calcomp plots of washability data, the screen analysis of the raw coal, a large part of the chemical data obtained from the screen and gravity fractions, and the washability analysis itself are presented.

Further characterization of the Pittsburgh sample (see Quarterly Reports 3, 4, and 5) during the report period included correlations between maceral groups, macerals, and mineral matter in head and float-sink fractions. Qualitative mineral distribution trends were also identified for head samples from the Pittsburgh and Pocahontas No. 3 seams using IR, and a special die and pressing technique was developed for use with the Phillips* APD-3501 X-ray diffractor.

Additional work in the quarter was performed on equipment in the "pilot-scale" preparation plant to optimize jigging capability and is discussed later in this report. The plant is still not "on-line" but plant operating parameters are dependent on Task 2 (characterization) so this should not be a problem due to the built-in time differential.

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*The use of brand names in no way implies recommendation or endorsement of these products by the Coal Research Bureau, West Virginia University, or the Department of Energy.

DESCRIPTION OF TECHNICAL PROGRESS

Coal Preparation Plant Samples

With the collection of the third commercial coal preparation plant samples in Central Illinois (See Quarterly Report No. 5) and their subsequent processing this quarter, the objective of Facet I has been completed. A summary of the facilities sampled is as follows:

Commercial Facilities Sampled

Mining District*	State	Type of Plant	Coal Seam
3	Northern WV	Jig and Table	Pittsburgh
7	Southern WV	Heavy Media, Table, Froth Flotation	Pocahontas No. 3
1	Central IL	Jig	Illinois No. 6

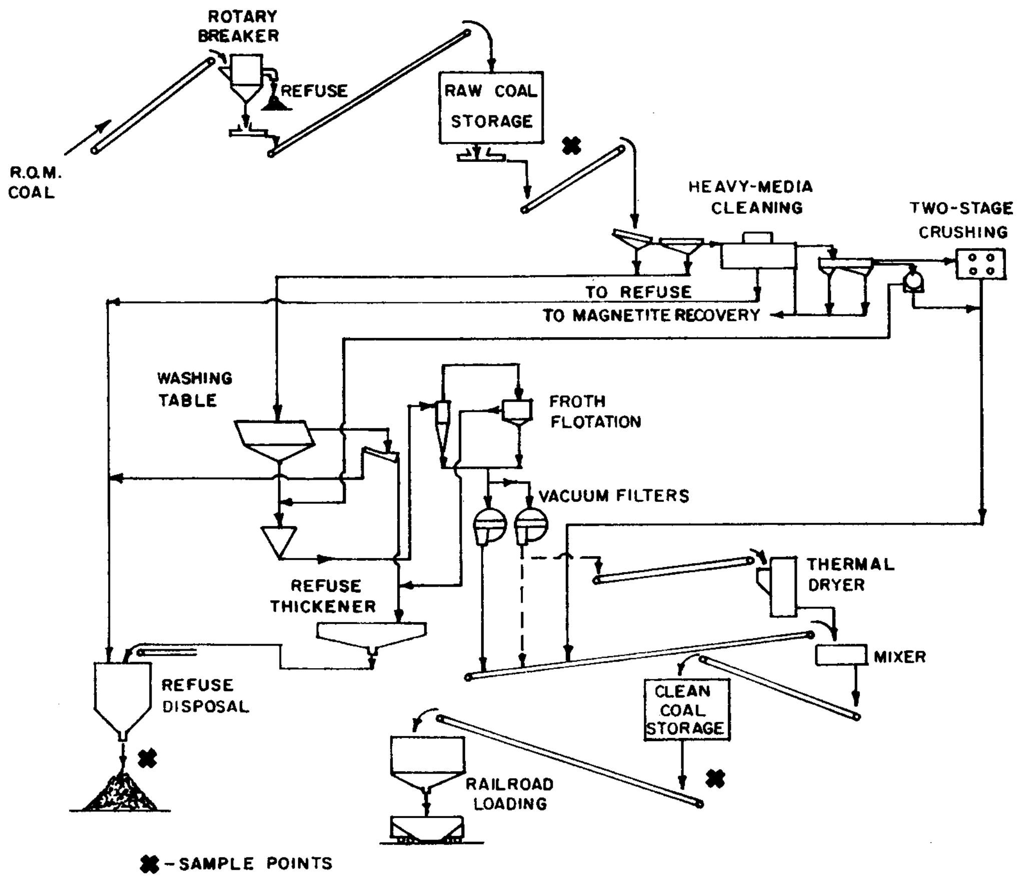
* Ref. Bituminous Coal Act of 1937

Samples of coal feeds, products, and refuse from the above sites are still being physically, chemically and mineralogically characterized as reviewed later in this report. Figure 1 shows the generalized flow diagram of the District 7 Preparation Plant and the sampling points for the run of mine (ROM) raw coal, the clean coal, and the refuse.

Bulk processing of preparation plant samples feed and product coals was completed during the quarter; representative samples prepared for characterization studies were from the Illinois No. 6 coal cleaning facility. Approximately 4,000 lbs. of raw coal were air dried and split (by cone and quartering) into equal increments, one of which was used for specific gravity separation and physical characterization studies in Task II of the subject contract. The remaining increment was crushed to 2" x 0 and a representative portion obtained by riffling was stored under nitrogen for pilot plant studies. The remaining

Figure 1

GENERALIZED FLOW SHEET OF DISTRICT SEVEN PREPARATION PLANT



2" x 0 fraction was further crushed and split to obtain head samples for chemical characterization.

Representative "as received" samples of washed coal as well as refuse were stored under nitrogen, and the remainder of the clean coal was processed by crushing and splitting to yield representative samples for analytical characterization. Additionally, approximately 90 gallons of coal slurry collected from the underflow surge tank were stored in plastic containers to prevent corrosive contamination prior to physical, chemical and mineralogical characterization.

Coal Preparation Pilot Plant

During the past quarter, the McNally-Pittsburg single-compartment, three cell jig was disassembled for a complete overhaul. This unit, built in 1947 is one of two such jigs produced by the company. All interior components of the jig which come in contact with water were refurbished. The bucket refuse elevator, hutch screw, perforated bed plate, refuse gate mechanism, journal bearings and underwater grease line were removed and the entire shell interior descaled by hand and sandblasted. To gain access to the back side of the jig, a six inch square opening was cut into each of the three cells to permit removal of interior scale and to allow sandblasting. Flanges were welded to the access plates for replacement by bolting after priming and painting the interior. The air cylinders which control pulsion and suction for each cell were dissassembled and reconditioned. The float mechanism which operates the refuse gate is also ready for re-assembly. The jig should be fully operational by the time the mineralogical and chemical characterization data from Facet II is completed.

Also during the quarter, an eight inch heavy media cyclone was made available for pilot plant testing work on the subject contract. A 200 gallon heavy media sump and pump system is being designed for this unit according to suggestions made by representatives of the manufacturer, Heyl-Patterson, during an on-site consultation visit. If possible, this equipment will be used in the pilot plant testing of 2.37 mm x 0.50 mm (3/8" x 35 mesh U. S.) raw coals.

Froth Flotation Tests

Froth flotation tests were performed on 2 samples of -100 mesh, Pittsburgh seam coal from North Central West Virginia during the sixth quarter. In both tests the 500 gram coal samples were conditioned for 15 minutes in 3000 ml. of distilled water (pH 6.2) at an impeller speed of 1500 rpm. MIBC was added during the conditioning period to allow optimum contact time between the coal and frother. Prior to aeration an additional 2000 ml. of distilled water was added to the slurry reducing the solids concentration in the slurry to 9 percent. The coal froth was then collected for a period of 1 minute. The froth product was filtered and dried to 100°C for 24 hours. This same procedure was followed for the refuse. Representative portions of both samples were submitted for analysis. The remaining portion of the froth product was retained for future reference.

Table 1 shows the results of the analysis of Pittsburgh seam tests # 1 and # 2. The results of these two tests indicate that an acceptable product can be produced from the -100 mesh fraction of the Pittsburgh coal seam by froth flotation. The total percent sulfur and percent ash in the clean coal from the preparation plant were 2.7 and 8.0, respectively. Test # 1 gave a product containing 3.14% sulfur and 11.3% ash while test # 2 gave a product of 2.85% sulfur and 8.6% ash. Future tests with the Pittsburgh sample will be directed at cleaning the coal to prepare a product that is either at or below the sulfur and ash levels of the commercially prepared sample.

TABLE 1

COAL FLOTATION TESTS

PITTSBURGH SEAM, NORTH CENTRAL WEST VIRGINIA

Test 1

	MIBC	% Yield	% Moisture	% Ash*	% Volatile Matter*	% FC*	% Total Sulfur
Feed Coal	-	-	0.7	19.6	34.1	46.3	3.26
Product	.56 lb. ton	72.0	0.4	11.3	34.9	53.9	3.14
Refuse	-----	28.0	0.3	40.1	28.3	31.6	3.65

Test 2

	MIBC	% Yield	% Moisture	% Ash*	% Volatile Matter*	% FC*	% Total Sulfur
Feed Coal	-	-	0.7	19.6	34.1	46.3	3.26
Product	.24 lb. ton	75.0	0.5	8.6	35.6	55.8	2.85
Refuse	-----	25.0	0.2	50.9	25.1	24.0	4.45

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*Whole coal, dry basis

Characterization of Coal Samples

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

Physical Characterization

A cumulative logarithmic plot of the screen analysis of the head sample of Pocahontas No. 3 sample, District Seven Preparation Plant, is shown in Figure 2. This ROM coal was crushed to approximately 5" x 0 prior to entering the preparation plant. Approximately 30 percent of the sample is greater than 1/4" (6.3 mm U. S. Mesh).

Bulk sample processing of the Pocahontas No. 3 raw coal head fraction and screen fractions followed the same procedure used for the Pittsburgh coal (See Quarterly Report No. 5). Each of 5 screened fractions obtained from the +100 mesh size range of the head sample were subsequently cleaned using laboratory float-sink tests while the -100 mesh portion was submitted for chemical analysis and froth flotation testing.

Table 2 presents the data from the float and sink specific gravity separations of the +100 mesh fractions, by fraction, for each of the following gravities: 1.3, 1.4, 1.6, and 1.8. An examination of the composite, +100 mesh, cumulative float data (1.8 S.G.) in Table 2 indicates that 75.84 weight percent can be recovered with an ash of 5.28 percent and 0.63 percent sulfur.

Figures 3-8 show the calcomp plots of the washability data from Table 2 and allow for interpolation between gravities. These plots will enable data from the laboratory cleaning studies to be more fully evaluated and compared to the products produced in both the pilot-scale preparation tests and the commercial plant.

FIG. 2

POCAHONTAS NO.3 RAW COAL (CUMULATIVE LOGARITHMIC PLOT OF SCREEN ANALYSIS)

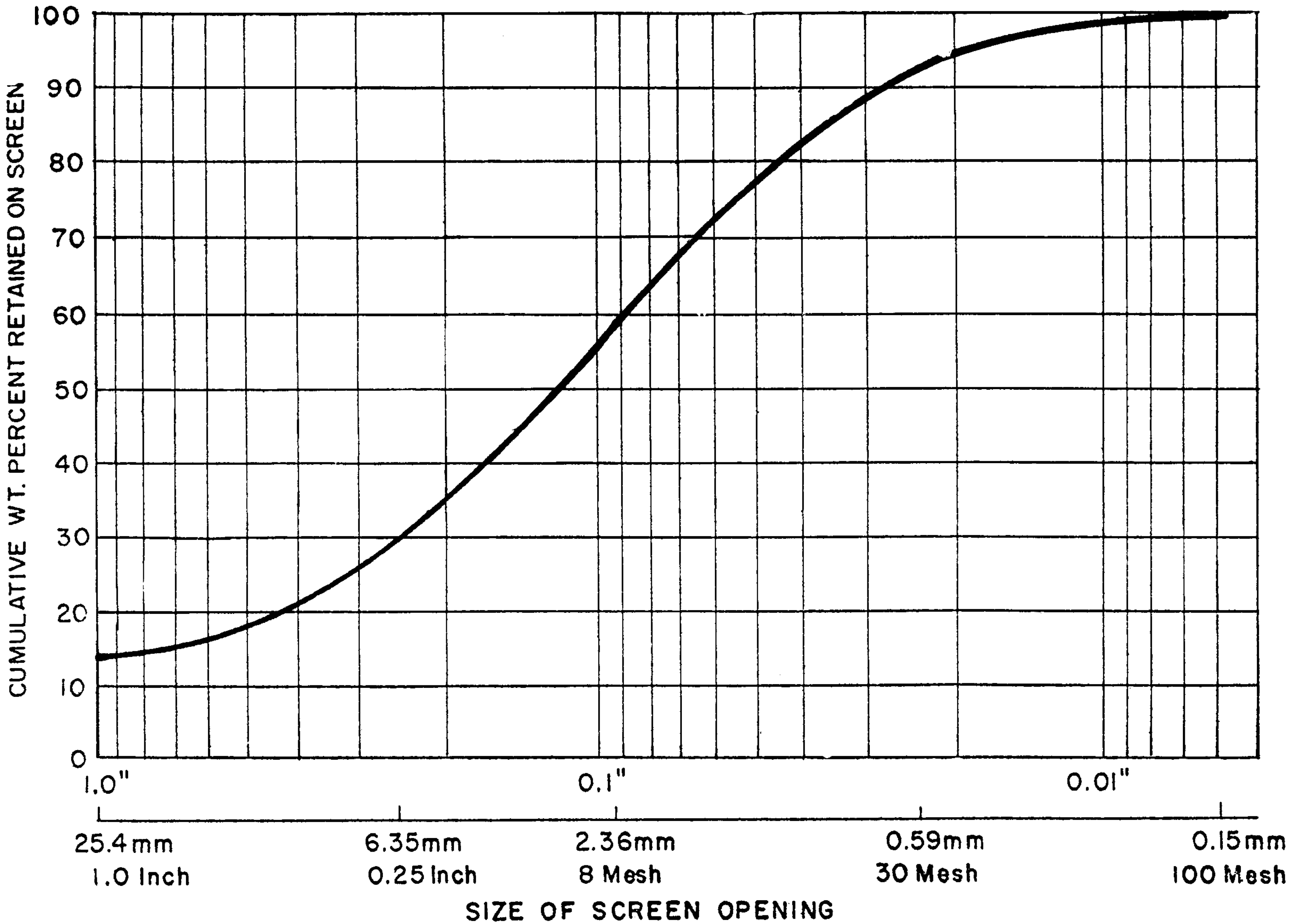


TABLE 2

FLOAT AND SINK DATA (+1" POCAHONTAS #3 HEAD SAMPLE)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
(1)	(2) Wt. %	(3) Ash %	(4) Ash Prod.	(5) Wt. %	(6) Ash Prod.	(7) Ash %	(8) Wt. %	(9) Ash Prod.	(10) Ash %	(11)
Float 1.3	14.04	2.6	36.50	14.04	36.50	2.60	100.00	5553.78	55.54	0.74
1.3 x 1.4	9.56	5.9	56.40	23.60	92.91	3.94	85.96	5517.27	64.18	0.69
1.4 x 1.6	5.51	19.6	108.00	29.11	200.90	6.90	76.40	5460.87	71.48	0.67
1.6 x 1.8	5.45	36.0	196.20	34.56	397.10	11.49	70.89	5352.87	75.51	0.63
Sink 1.8	65.44	78.8	5156.67	100.00	5553.78	55.54	65.44	5156.67	78.80	0.45

FLOAT AND SINK DATA (1" x 1/4" POCAHONTAS #3 HEAD SAMPLE)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
(1)	(2) Wt. %	(3) Ash %	(4) Ash Prod.	(5) Wt. %	(6) Ash Prod.	(7) Ash %	(8) Wt. %	(9) Ash Prod.	(10) Ash %	(11)
Float 1.3	21.28	2.4	51.07	21.28	51.07	2.40	100.00	4229.08	42.29	0.61
1.3 x 1.4	22.26	7.6	169.18	43.54	220.25	5.06	78.72	4178.01	53.07	0.60
1.4 x 1.6	8.16	21.3	173.81	51.70	394.06	7.62	56.46	4008.83	71.00	0.59
1.6 x 1.8	5.94	35.9	213.25	57.64	607.30	10.54	48.30	3835.03	79.40	0.57
Sink 1.8	42.36	85.5	3621.78	100.00	4229.08	42.29	42.36	3621.78	85.50	0.47

TABLE 2 (Cont'd)

FLOAT AND SINK DATA (1/4" x 8 M POCAHONTAS #3 HEAD SAMPLE)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
	Wt. %	Ash %	Ash Prod.	Wt. %	Ash Prod.	Ash %	Wt. %	Ash Prod.	Ash %	
Float 1.3	55.33	2.4	132.79	55.33	132.79	2.40	100.00	1624.21	16.24	0.64
1.3 x 1.4	22.34	7.9	176.49	77.67	309.28	3.98	44.67	1491.42	33.39	0.63
1.4 x 1.6	6.42	17.5	112.35	84.09	421.63	5.01	22.33	1314.94	58.89	0.63
1.6 x 1.8	2.63	32.6	85.74	86.72	507.37	5.85	15.91	1202.59	75.59	0.63
Sink 1.8	13.28	84.1	1116.85	100.00	1624.21	16.24	13.28	1116.85	84.10	0.59

FLOAT AND SINK DATA (8 x 28 M POCAHONTAS #3 HEAD SAMPLE)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
	Wt. %	Ash %	Ash Prod.	Wt. %	Ash Prod.	Ash %	Wt. %	Ash Prod.	Ash %	
Float 1.3	75.11	1.9	142.71	75.11	142.71	1.90	100.01	868.83	8.69	0.65
1.3 x 1.4	13.71	11.6	159.04	88.82	301.75	3.40	24.90	726.12	29.16	0.65
1.4 x 1.6	4.01	16.8	67.37	92.83	369.11	3.98	11.19	567.08	50.68	0.64
1.6 x 1.8	1.27	30.5	38.74	94.10	407.85	4.33	7.18	499.72	69.60	0.64
Sink 1.8	5.91	78.0	460.98	100.01	868.83	8.69	5.91	460.98	78.00	0.64

TABLE 2 (Cont'd)

FLOAT AND SINK DATA (28 x 100 M POCAHONTAS #3 HEAD SAMPLE)

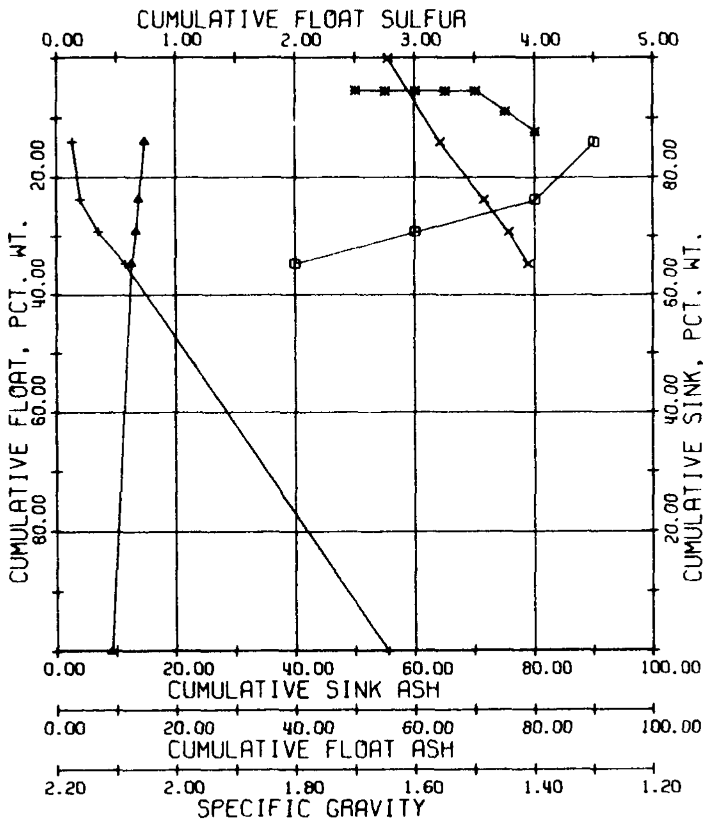
Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
(1)	(2) Wt. %	(3) Ash %	(4) Ash Prod.	(5) Wt. %	(6) Ash Prod.	(7) Ash %	(8) Wt. %	(9) Ash Prod.	(10) Ash %	(11)
Float 1.3	82.47	1.8	148.45	82.47	148.45	1.80	100.00	583.62	5.84	0.66
1.3 x 1.4	8.84	6.9	61.00	91.31	209.44	2.29	17.53	435.18	24.82	0.66
1.4 x 1.6	3.35	13.5	45.23	94.66	254.67	2.69	8.69	374.18	43.06	0.66
1.6 x 1.8	1.07	23.3	24.93	95.73	279.60	2.92	5.34	328.96	61.60	0.66
Sink 1.8	4.27	71.2	304.02	100.00	583.62	5.84	4.27	304.02	71.20	0.71

FLOAT AND SINK DATA (+100 M COMPOSITE HEAD SAMPLE)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
(1)	(2) Wt. %	(3) Ash %	(4) Ash Prod.	(5) Wt. %	(6) Ash Prod.	(7) Ash %	(8) Wt. %	(9) Ash Prod.	(10) Ash %	(11)
Float 1.3	53.49	2.0	106.98	53.49	106.98	2.00	99.98	2283.61	22.84	0.65
1.3 x 1.4	14.04	7.8	109.51	67.53	216.49	3.21	46.49	2176.63	46.82	0.64
1.4 x 1.6	5.20	17.3	89.96	72.73	306.45	4.21	32.45	2067.11	63.70	0.63
1.6 x 1.8	3.11	30.3	94.23	75.84	400.69	5.28	27.25	1977.15	72.56	0.63
Sink 1.8	24.14	78.0	1882.92	99.98	2283.61	22.84	24.14	1882.92	78.00	0.57

Figure 3

SULFUR AND ASH WASHABILITY
POCA.RAW COAL +1-IN.FRACTION
15.26 WT. PCT. OF TOTAL SAMPLE



KEY

✱ ± 0.1 SPECIFIC GRAVITY DISTRIBUTION
\+ CUMULATIVE FLOAT ASH
× CUMULATIVE SINK ASH
▢ SPECIFIC GRAVITY
△ CUMULATIVE FLOAT SULFUR

Figure 4

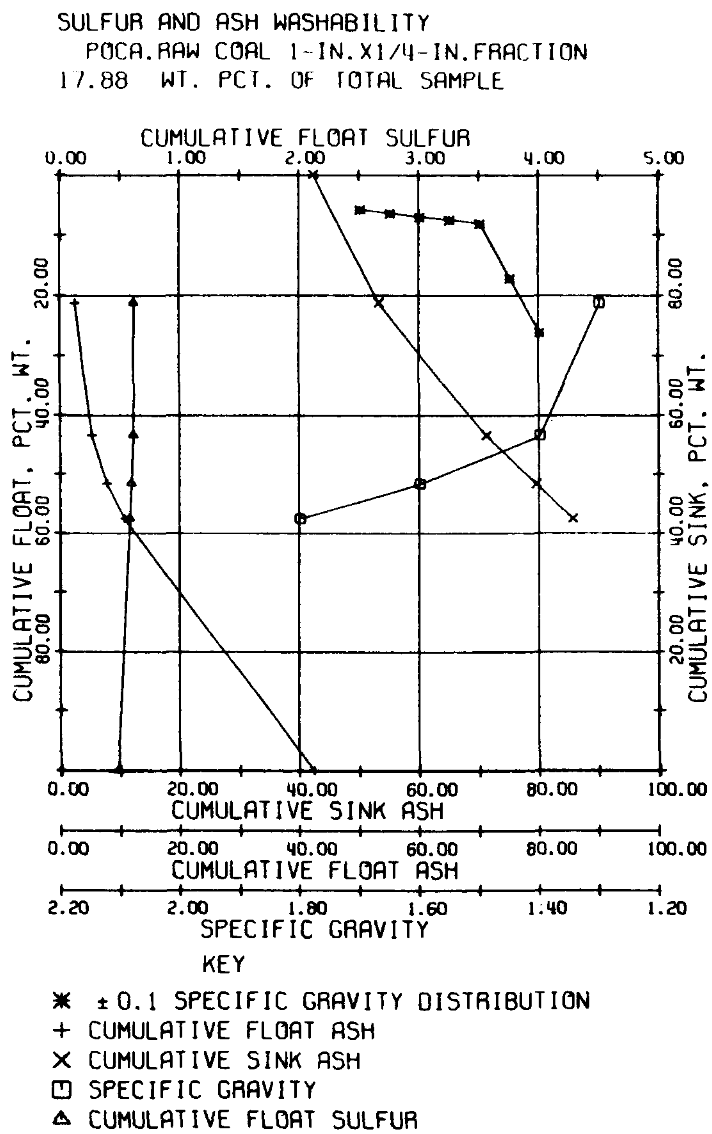
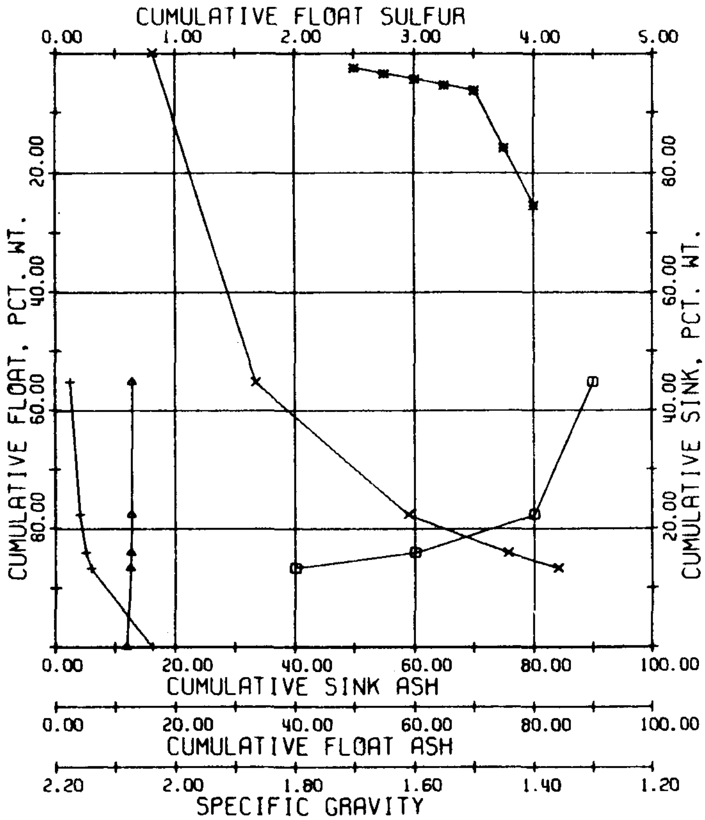

SULFUR AND ASH WASHABILITY
POCA.RAW COAL 1-IN.X1/4-IN.FRACTION
17.88 WT. PCT. OF TOTAL SAMPLE
CUMULATIVE FLOAT SULFUR
0.00
1.00
2.00
3.00
4.00
5.00
CUMULATIVE FLOAT, PCT. WT.
20.00
40.00
60.00
80.00
CUMULATIVE SINK, PCT. WT.
80.00
60.00
40.00
20.00
0.00
20.00
40.00
60.00
80.00
100.00
CUMULATIVE SINK ASH
0.00
20.00
40.00
60.00
80.00
100.00
CUMULATIVE FLOAT ASH
2.20
2.00
1.80
1.60
1.40
1.20
SPECIFIC GRAVITY
KEY
± 0.1 SPECIFIC GRAVITY DISTRIBUTION
CUMULATIVE FLOAT ASH
CUMULATIVE SINK ASH
SPECIFIC GRAVITY
CUMULATIVE FLOAT SULFUR

Figure 5

SULFUR AND ASH WASHABILITY
POCA.RAW COAL 1/4-IN.X 8MESH FRACTION
25.31 WT. PCT. OF TOTAL SAMPLE



KEY

✱ ±0.1 SPECIFIC GRAVITY DISTRIBUTION
\+ CUMULATIVE FLOAT ASH
× CUMULATIVE SINK ASH
⊡ SPECIFIC GRAVITY
△ CUMULATIVE FLOAT SULFUR

Figure 6

SULFUR AND ASH WASHABILITY
POCA.RAW COAL 8MESH X 28MESH FRACTION
35.02 WT. PCT. OF TOTAL SAMPLE

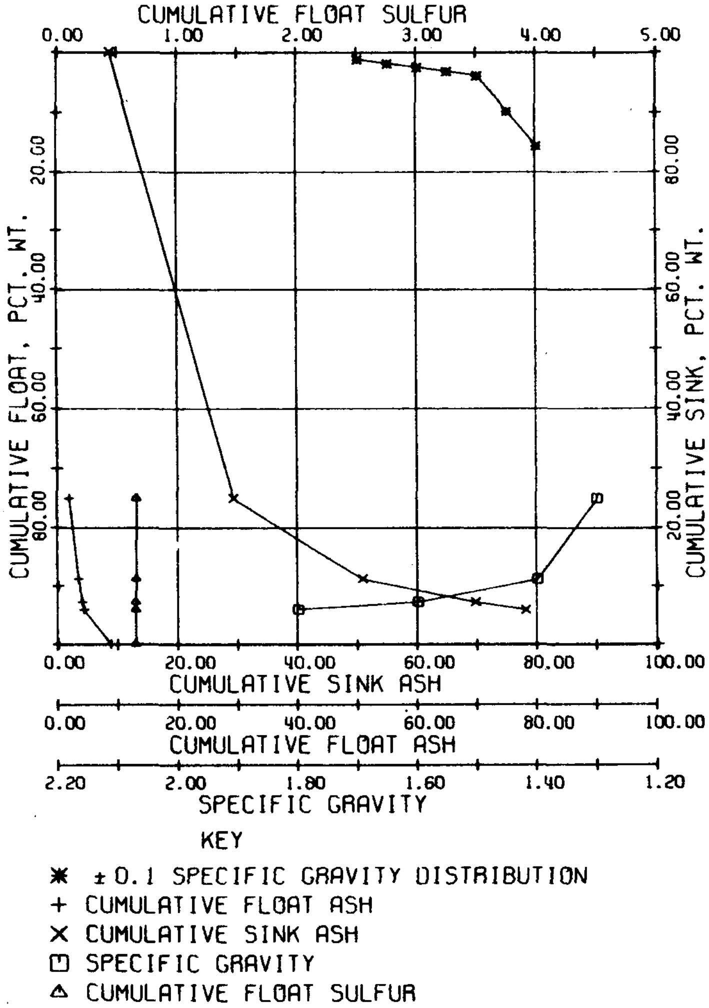
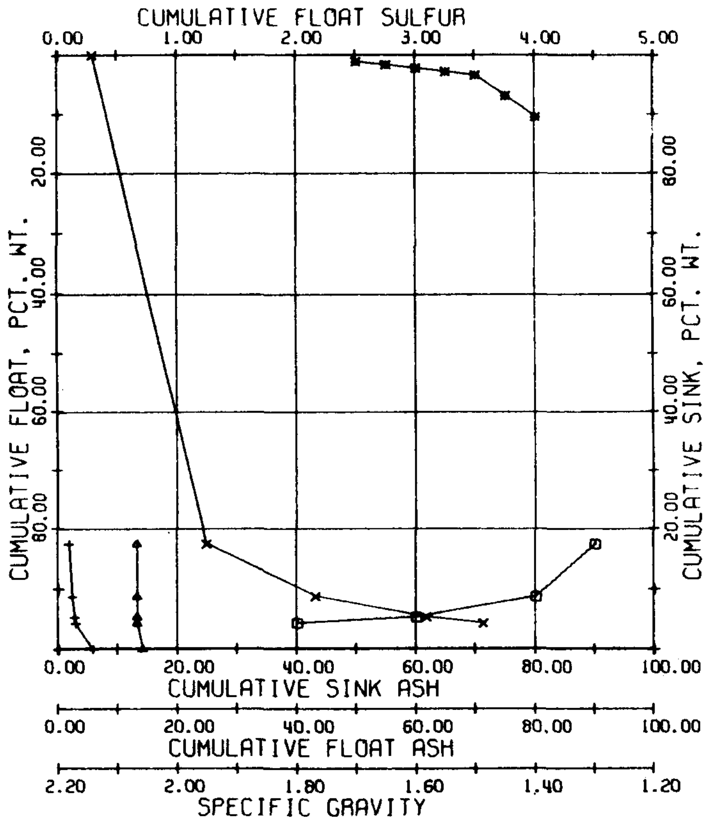


Figure 7

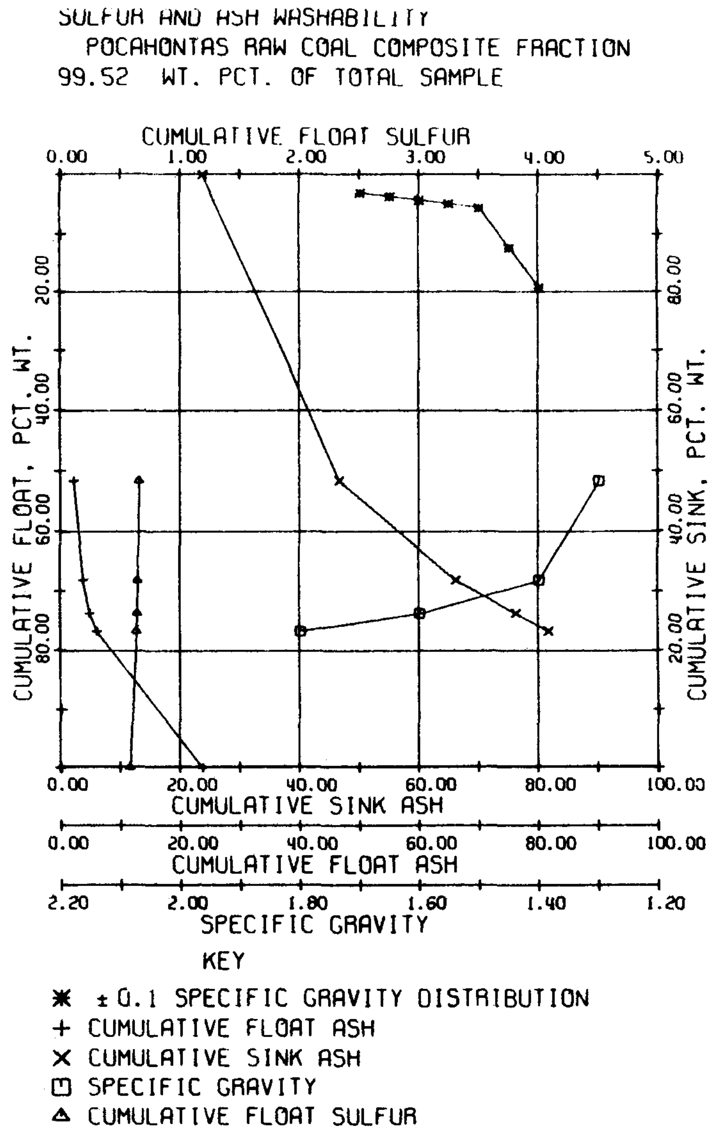
SULFUR AND ASH WASHABILITY
POCA.RAW COAL 28MESH X 100MESH FRACTION
6.05 WT. PCT. OF TOTAL SAMPLE



KEY

- ✱ ± 0.1 SPECIFIC GRAVITY DISTRIBUTION
- + CUMULATIVE FLOAT ASH
- × CUMULATIVE SINK ASH
- ⊡ SPECIFIC GRAVITY
- △ CUMULATIVE FLOAT SULFUR

Figure 8


SULFUR AND ASH WASHABILITY
POCAHONTAS RAW COAL COMPOSITE FRACTION
99.52 WT. PCT. OF TOTAL SAMPLE
CUMULATIVE FLOAT SULFUR
0.00
1.00
2.00
3.00
4.00
5.00
CUMULATIVE FLOAT, PCT. WT.
20.00
40.00
60.00
80.00
CUMULATIVE SINK, PCT. WT.
80.00
60.00
40.00
20.00
0.00
20.00
40.00
60.00
80.00
100.00
CUMULATIVE SINK ASH
0.00
20.00
40.00
60.00
80.00
100.00
CUMULATIVE FLOAT ASH
2.20
2.00
1.80
1.60
1.40
1.20
SPECIFIC GRAVITY
KEY
± 0.1 SPECIFIC GRAVITY DISTRIBUTION
CUMULATIVE FLOAT ASH
CUMULATIVE SINK ASH
SPECIFIC GRAVITY
CUMULATIVE FLOAT SULFUR

Chemical Characterization

Tables 3 and 4 contain detailed analytical data for those fractions of the Pocahontas No. 3 head coal which were prepared by sizing and gravity techniques into 25 fractions of the plus 100 mesh (U. S.) raw coal feed. Analyses of the minus 100 mesh feed coal and the raw coal head samples are also reported. Table 3 shows the proximate analysis and sulfur breakdown analysis of each fraction. About two-thirds of the S in the Pocahontas head sample is organic and therefore tends to be concentrated in the cleaner coal fractions. The sulfate S is evenly distributed and has no appreciable concentration in clean coal fractions. Pyrite S is increasingly concentrated in the finer coal fractions and at the higher gravities, in particular the 1.80 sink as would be expected.

Trends in the proximate analysis are typical, with ash increasing and fixed carbon and volatile matter decreasing as the gravity of separation increases.

Spectrographic analyses for Si, Al, Fe, Ti, Ca, and Mg and atomic absorption analyses for Na and K are shown in Table 4. Computer correlations of these elements and with other chemical and mineralogical data will be considered in future work. However, a preliminary examination of the data shows relative concentrations of Si, Al, Fe, Ti, Mg, Na and K in the heavier gravities within a given size fraction. Variations between fractions are not generally pronounced, with most of the variability apparently due to gravity and not size. Future analyses of the LTA of these fractions should relate these elemental data to clay minerals, quartz, and pyrite concentrations in the heavier fractions of a given size.

Mineralogical Characterization

Petrographic correlations between maceral groups, macerals and mineral matter in Pittsburgh feed coal fractions were investigated during the report period, and a relationship between LTA mineral matter and petrographically

TABLE 3

CHEMICAL CHARACTERIZATION DATA
(Pocahontas No. 3 Coal - Sized, Gravity Fraction)*

	Proximate Analysis				Sulfur Breakdown			
	Moisture	Ash	Volatile Matter	Fixed Carbon	Sulfate S	Pryite S	Organic S	Total S
+1" Coal								
Float 1.3	0.8	2.6	17.7	79.7	0.01	0.13	0.60	0.74
1.3 x 1.4	0.7	5.9	16.2	77.9	0.02	0.10	0.50	0.62
1.4 x 1.6	0.7	19.6	14.7	65.7	0.01	0.17	0.38	0.56
1.6 x 1.8	0.6	36.0	12.5	51.5	0.01	0.12	0.29	0.42
Sink 1.8	0.9	78.8	9.2	12.0	0.03	0.30	0.03	0.36
1" x 1/4" Coal								
Float 1.3	0.6	2.4	18.1	79.5	0.01	0.06	0.54	0.61
1.3 x 1.4	0.8	7.6	15.3	77.1	0.01	0.10	0.48	0.59
1.4 x 1.6	0.8	21.3	14.4	64.3	0.01	0.16	0.34	0.51
1.6 x 1.8	0.8	35.9	12.8	51.3	0.01	0.16	0.26	0.43
Sink 1.8	1.0	85.5	7.3	7.2	0.01	0.32	0.01	0.34
1/4" x 8 Mesh								
Float 1.3	0.8	2.4	18.1	79.5	0.01	0.10	0.53	0.64
1.3 x 1.4	0.9	7.9	15.5	76.6	0.01	0.13	0.48	0.62
1.4 x 1.6	0.9	17.5	13.9	68.6	0.01	0.21	0.36	0.58
1.6 x 1.8	0.9	32.6	13.2	54.2	0.01	0.25	0.28	0.54
Sink 1.8	1.1	84.1	7.3	8.6	0.01	0.35	0.01	0.37

*Date reported in percent, on the moisture free whole coal basis (for each fraction).

TABLE 3 (Continued)

CHEMICAL CHARACTERIZATION DATA
(Pocahontas No. 3 Coal - Sized, Gravity Fraction)*

	Proximate Analysis				Sulfur Breakdown			
	Moisture	Ash	Volatile Matter	Fixed Carbon	Sulfate S	Pyrite S	Organic S	Total S
8 x 28 Mesh Coal								
Float 1.3	1.0	1.9	18.4	79.7	0.01	0.08	0.56	0.65
1.3 x 1.4	0.9	11.6	15.2	73.2	0.01	0.17	0.44	0.62
1.4 x 1.6	1.0	16.8	13.8	69.4	0.01	0.19	0.37	0.57
1.6 x 1.8	1.2	30.5	13.2	56.3	0.01	0.42	0.21	0.64
Sink 1.8	1.6	78.0	8.7	13.3	0.01	0.56	0.02	0.59
28 x 100 Mesh								
Float 1.3	0.4	1.8	18.9	79.3	0.01	0.05	0.60	0.66
1.3 x 1.4	0.6	6.9	17.3	75.8	0.02	0.13	0.52	0.67
1.4 x 1.6	0.6	13.5	14.2	72.3	0.02	0.19	0.42	0.63
1.6 x 1.8	1.0	23.3	13.4	63.3	0.05	0.36	0.33	0.74
Sink 1.8	0.7	71.2	12.7	16.1	0.10	1.61	0.02	1.73
Minus 100 Mesh								
	1.1	13.8	16.8	69.4	0.02	0.21	0.48	0.71
Raw Coal Head								
	1.2	35.0	14.1	50.9	0.01	0.16	0.33	0.50

*Data reported in percent, on the moisture free whole coal basis (for each fraction).

TABLE 4

CHARACTERIZATION DATA
(Pocahontas No. 3 Coal - Sized, Gravity Fractions)

	Spectrographic Analysis*						Atomic Absorption**		LTA	BTU
	Si	Al	Fe	Ti	Ca	Mg	Na	K	%	
+1" Coal										
Float 1.3	0.31	0.24	0.64	0.01	0.08	0.03	0.03	0.01	3.6	15491
1.3 x 1.4	1.18	0.67	0.43	0.07	0.09	0.04	0.02	0.07	6.8	125055
1.4 x 1.6	4.55	2.27	0.42	0.37	0.49	0.06	0.02	0.07	21.8	12405
1.6 x 1.8	8.43	2.95	0.55	0.74	0.17	0.06	0.31	0.20	39.0	9557
Sink 1.8	19.67	8.22	2.98	0.51	0.37	0.59	0.33	0.30	83.7	2246
1" x 1/4" Coal										
Float 1.3	0.39	0.33	0.32	0.01	0.05	0.02	0.01	0.01	3.1	15650
1.3 x 1.4	1.49	1.03	0.52	0.09	0.11	0.05	0.04	0.03	8.6	14608
1.4 x 1.6	5.02	2.22	0.48	0.28	0.55	0.08	0.07	0.07	23.6	12250
1.6 x 1.8	9.89	3.50	0.60	0.87	0.65	0.09	0.12	0.89	39.1	9571
Sink 1.8	20.59	8.55	2.88	0.60	0.44	0.66	0.32	2.33	90.3	945
1/4" x 8 Mesh										
Float 1.3	0.46	0.37	0.23	0.02	0.04	0.02	0.03	0.01	3.2	15602
1.3 x 1.4	1.68	1.23	0.46	0.08	0.08	0.05	0.06	0.07	9.2	14633
1.4 x 1.6	3.54	1.99	0.68	0.22	0.34	0.09	0.10	0.11	20.0	12623
1.68 x 1.8	7.97	3.26	0.92	0.36	0.92	0.19	0.12	0.24	36.7	9942
Sink 1.8	19.97	8.19	3.32	0.54	0.44	0.65	0.26	2.03	89.5	1191

---

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

**Percent element in the ash.

TABLE 4 (Continued)

CHARACTERIZATION DATA
(Pocahontas No. 3 Coal - Sized, Gravity Fractions)

	Spectrographic Analysis*						Atomic Absorption**		LTA	BTU
	Si	Al	Fe	Ti	Ca	Mg	Na	K	%	
8 x 28 Mesh Coal										
Float 1.3	0.36	0.34	0.19	0.02	0.04	0.01	0.02	0.02	2.8	15683
1.3 x 1.4	2.87	1.95	0.65	0.12	0.17	0.09	0.07	0.12	13.0	14173
1.4 x 1.6	3.96	2.40	0.61	0.27	0.21	0.09	0.09	0.08	19.1	12831
1.6 x 1.8	7.46	3.81	1.19	0.46	0.78	0.14	0.13	0.19	35.4	10062
Sink 1.8	19.47	10.78	2.21	0.50	1.09	0.66	0.29	1.53	83.5	2260
28 x 100 Mesh Coal										
Float 1.3	0.33	0.24	0.13	0.02	0.04	0.01	0.02	0.02	2.7	15585
1.3 x 1.4	1.47	1.08	0.38	0.08	0.10	0.04	0.05	0.07	8.2	15040
1.4 x 1.6	3.28	2.19	0.73	0.18	0.14	0.06	0.09	0.09	15.5	13372
1.6 x 1.8	5.15	3.01	1.20	0.27	0.39	0.12	0.14	0.19	27.2	11393
Sink 1.8	13.81	5.69	3.37	0.37	4.13	0.45	0.23	1.49	80.8	3012
Minus 100 Mesh Coal										
	2.99	1.65	0.58	0.10	0.53	0.12	0.07	0.42	18.2	13573
Raw Coal Head										
	8.13	3.26	1.58	0.25	0.26	0.26	0.16	0.34	37.6	9949

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

**Percent element in the ash.

observed mineral matter was determined. Using IR, some mineral distribution trends were found for head samples of both the Pittsburgh and Pocahontas No. 3 coals. In the X-ray area of mineralogical characterization, attempts to install the Phillips APD unit were delayed by a generator malfunction which should be corrected in the next quarter. Preparatory X-ray work was devoted to the design of a special pellet die and pressing technique for use with the automatic sample feeder for the APD.

Petrographic Analysis - Analyses presented in Table 5 of Quarterly Report No. 5 established volume-percent concentrations of 14 macerals and mineral matter in float-sink fractions of the Pittsburgh coal. It was also established that these data were seldom normally distributed, and therefore only simple statistics could be applied to these data to aid in the interpretation of maceral trends in the float-sink fractions.

The statistical technique which was most applicable was Spearman rank correlation. Data compared by this technique were: amounts of the 14 macerals, 3 maceral group totals, mineral matter total, and the specific gravity and mean size of the particles in the 29 fractions. Results of Spearman rank correlation are presented in tabular form in Table 5. The correlation coefficient matrix was rearranged to separate the correlations between maceral groups and their member macerals from correlations occurring between macerals outside the member group. Maceral groups and member macerals are presented in Table 6 of Quarterly Report No. 5. Table 5, for example, shows that telinite correlates with vitrinite as a group at +0.56 (correlation coefficient), and telinite also correlates with micrinite from the inertinite group at +0.58. A correlation with a maceral outside the group is only included in Table 5 if it exceeds the correlation coefficient of the member maceral to the group.

Group Maceral		*Maceral within Group*	*Maceral to Group Correlation Coefficient*		*Maceral with Greatest Correlation Outside Group*	*C.C.*
VITRINITE	to:	Collinite	+0.99	to:	none	
		telocollinite	+0.99	to:	none	
		Telinite	+0.56	to:	Micrinite	+0.58
		desmocollinite	+0.39	to:	Semifusinite	+0.80
EXINITE	to:	Liptodetrinite	+0.86	to:	none	
		Sporinite	+0.64	to:	Mineral Matter	-0.74
INERTINITE	to:	Inertodetrinite	+0.84	to:	none	
		Macrinite	+0.75	to:	none	
		Semifusinite	+0.75	to:	desmocollinite	+0.80
		Fusinite	+0.75	to:	none	
		pyrofusinite	+0.68	to:	none	
		degradosemifusinite	+0.67	to:	desmocollinite	+0.76
		degradofusinite	+0.63	to:	none	
		Micrinite	+0.12	to:	telocollinite	+0.86

Table 5. Correlations between Maceral Groups, Macerals, and Mineral Matter in Head samples and float-sink fractions of the Pittsburgh District #3 Preparation Plant feed coal. The first set of correlations is between Maceral Groups and their member Macerals, and the second set includes the greatest correlation between that maceral and any other maceral which exceeds the Group-to-Maceral correlation. ($n=29$)

As expected, collinite and telocollinite (the major maceral and submaceral of the vitrinite group) correlate well (+0.99) with vitrinite. Telinite correlates moderately well with the vitrinite group, but also with the inertinite maceral micrinite. Desmocollinite correlates best with semifusinite, and this will be discussed in the following paragraph. Both macerals of the exinite group correlate well with that group. Sporinite inversely correlates with mineral matter, and this shows that sporinite is greatest in coal fractions lowest in mineral matter. Inertinite macerals and submacerals correlate well with the group, except for micrinite. Micrinite shows a closer affinity to vitrinite macerals than the inertinite group.

From the above correlations three types of coal fractions appear to have been formed by the float-sink separation: (1) a fraction high in the vitrinite macerals and submacerals (collinite, telocollinite, and telinite), and micrinite. This preference of the inertinite maceral, micrinite, for vitrinite has been previously documented (Stach et. al., 1975, p. 103). This fraction represents "bright" bands of the coal separated from the remainder of the coal through the float-sink technique; (2) a coal fraction including the macerals desmocollinite, semifusinite, and degradosemifusinite. These three macerals are common in "dull" bands of the seam, and this fraction represents the accumulation of these bands in certain float-sink fractions; (3) mineral-matter-rich layers.

Average particle size of the coal from the mine did not correlate strongly with any maceral or maceral group. The greatest correlation coefficient (+0.48) occurred between size and exinite amount. This weakly supports the theory proposed by others that exinite macerals toughen the coal, and thus increase particle size produced through mining. Relatively small amounts of exinite in these samples (6% maximum) produce the low correlation coefficient. Recalculations of exinite on a mineral-matter-free basis resulted in a better correlation (+0.72) between mineral-matter-free exinite and size.

Specific gravity of the coal fraction correlates negatively with most macerals (Table 6), and positively, as expected, with mineral matter. Macerals of the "bright" coal fractions correlate best, in a negative sense, with specific gravity. These include vitrinite, collinite, telocollinite (all -0.94), telinite (-0.56), sporinite (-0.79) and micrinite (-0.86). The maceral fusinite and its submacerals pyrofusinite and degradofusinite correlate positively with specific gravity along with mineral matter. This results from mineral matter inclusions which often fill cavities in this maceral. Mineral matter filled fusinite increases proportionally with increasing specific gravity of the fraction produced in float-sink tests.

It is highly important to this study that all or most of the mineral matter in these samples be recognized under the petrographic microscope. To evaluate this assumption, petrographic mineral matter (PMM-expressed as a volume percent of the coal) was plotted against the low temperature ash (LTA) value (assumed to be "true" mineral matter contents of the samples). The resulting plot, Figure 9, shows a good agreement which is confirmed by the correlation coefficient (+0.99). Linear regression applied to the values (Table 7) plotted in Figure 9 resulted in the following equation for petrographic mineral matter (PMM):

$$PMM = 1.074\,(LTA) - 5.819 \qquad \text{(eq. 1)}$$

The slope of the line (1.074) indicates that the difference between PMM and LTA is greatest in the low mineral matter (MM) samples. High MM samples (1.80 sink) generally contain more MM by volume than LTA by weight. This is as expected due to the difference in density between coal

	Maceral or Maceral Group	Correlation Coefficient
Specific Gravity Correlation to:	VITRINITE	-0.94
	Collinite	-0.94
	telocollinite	-0.94
	Telinite	-0.56
	desmocollinite	-0.36
	EXINITE	-0.41
	Sporinite	-0.79
	Liptodetrinite	-0.10
	INERTINITE	-0.02
	Micrinite	-0.86
	pyrofusinite	+0.55
	degradosemifusinite	-0.45
	degradofusinite	+0.42
	Fusinite	+0.40
	Semifusinite	-0.39
	Macrinite	+0.05
	Inertodetrinite	+0.02
	MINERAL MATTER	+0.94

Table 6. Correlations between specific gravity of the float-sink fraction and Maceral Groups, Macerals, and Mineral Matter. (n=29)

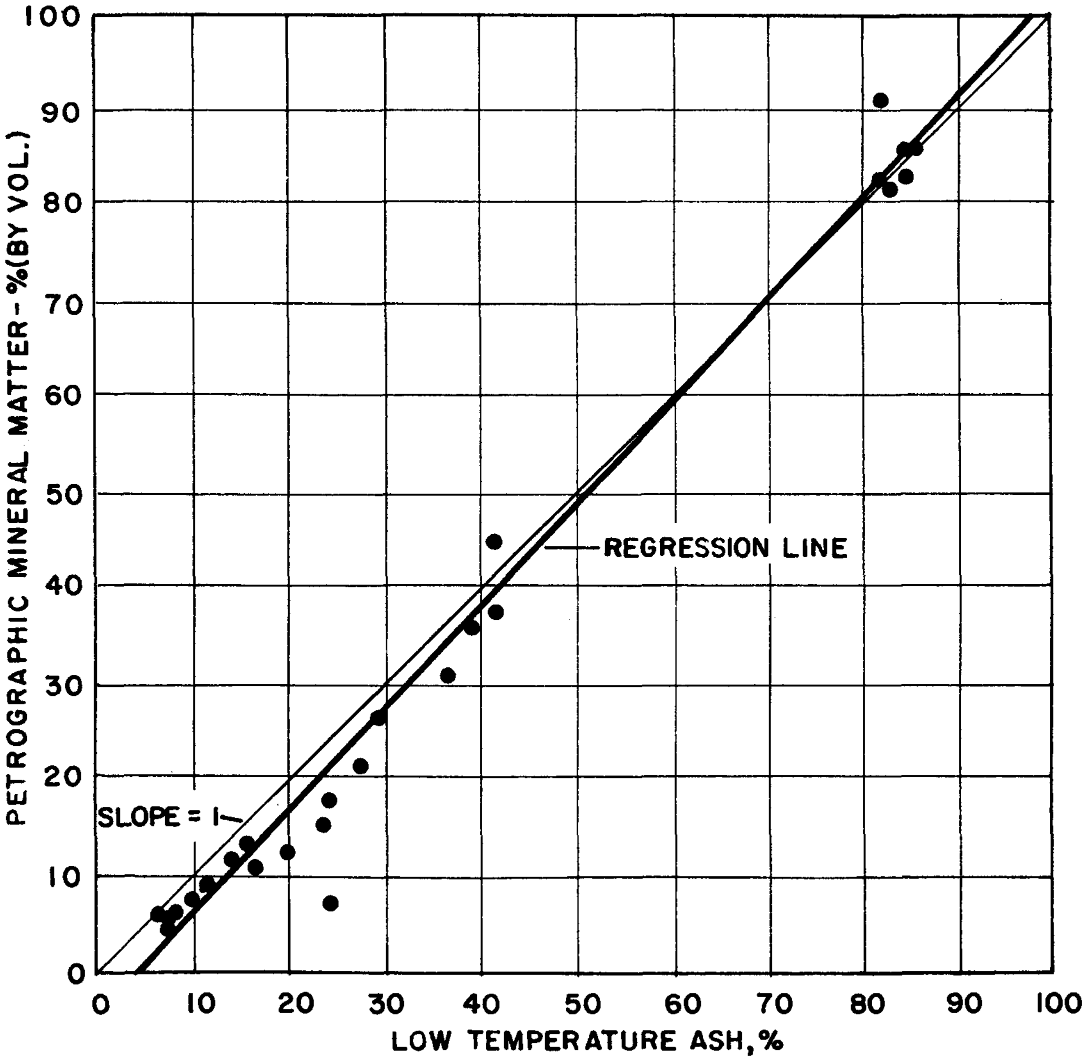


FIGURE 9

RELATIONSHIP BETWEEN TRUE MINERAL MATTER (LTA) AND PETROGRAPHICALLY OBSERVED MINERAL MATTER IN THE PITTSBURGH DISTRICT #3 PREPARATION PLANT FEED COAL

Table 7. A comparison of "true" mineral matter (LTA) and petrographic mineral matter (PMM) in the Pittsburgh District #3 preparation plant feed coal head samples and float-sink fractions.

Size	*Specific Gravity*	LTA *(weight percent)*	PMM *(volume percent)*
+1 inch	1.30 float	7.8%	5%
+1 inch	1.40 float	13.6	12
+1 inch	1.60 float	26.0	26
+1 inch	1.80 float	41.0	46
+1 inch	1.80 sink	82.5	91
1X¼ inch	1.30 float	7.2	6
1X¼ inch	1.40 float	14.8	13
1X¼ inch	1.60 float	28.7	26
1X¼ inch	1.80 float	42.4	37
1X¼ inch	1.80 sink	84.6	86
¼X8 mesh	1.30 float	7.1	4
¼X8 mesh	1.40 float	23.7	15
¼X8 mesh	1.60 float	27.7	21
¼X8 mesh	1.80 float	39.0	36
¼X8 mesh	1.80 sink	84.4	86
8X28 mesh	1.30 float	6.1	5
8X28 mesh	1.40 float	13.6	12
8X28 mesh	1.60 float	24.3	18
8X28 mesh	1.80 float	39.3	36
8X28 mesh	1.80 sink	82.4	83
28X100 mesh	1.30 float	7.0	5
28X100 mesh	1.40 float	11.4	8
28X100 mesh	1.60 float	19.9	12
28X100 mesh	1.80 float	35.9	30
28X100 mesh	1.80 sink	85.3	83
-100 mesh screen fraction		24.8	8
Clean Coal Head		10.1	7
Feed Coal Head		17.3	11
Refuse Head		83.2	82

and mineral matter. Using eq. 1, 0% PMM equates to 5% LTA. This 5% MM may be the so-called inherent mineral matter finely disseminated throughout the coal, especially in the vitrinite macerals. Since there is less vitrinite and less inherent mineral matter in the 1.80 sink fractions there is less disagreement between PMM and LTA in these fractions. Disagreement is greatest in fractions with intermediate amounts of MM (1.60 - 1.80 floats). These samples contain many finely dispersed maceral and mineral particles, making quantitative mineral measurements difficult. A similar problem was encountered with the -100 mesh screen fraction in which 8% PMM was observed as opposed to 25% LTA. Because the particle size was much smaller than the 20 mesh size recommended for petrographic work, much mineral matter was missed or unrecognizable through the microscope.

In summary, a limited statistical evaluation of data presented last quarter revealed that maceral trends exist in the 25 float-sink fractions, and that these are directly attributable to heterogeneous banding in the coal seam. It was also determined that most aggregated mineral matter in the Pittsburgh coal could be recognized through petrographic analysis, but that each fraction contains a small amount (about 5%) of inherent, and apparently unrecognizable, mineral matter dispersed throughout the coal matrix.

Infrared Analysis - During this report period a recirculating air dryer/CO_2 absorption unit was installed, resulting in much improved resolution over a broad spectral range (4000 - 180 cm^{-1}) utilizing both potassium bromide and cesium iodide matrix materials.

Mineral distribution trends were qualitatively determined for the feed coal, clean coal, and refuse fractions of both the Pittsburgh and Pocahontas seams based on comparison of relative peak intensities. Minerals studied included kaolinite, quartz, gypsum-hemihydrate, and carbonates in general.

Of the Pittsburgh coals, the feed coal fraction contained the highest concentration of kaolinite (Figure 10). The refuse fraction (Figure 11) contained less kaolinite than both the feed and clean coals. Two absorption bands were reported for gypsum-hemihydrate at 595 and 660 cm^{-1} in Quarterly Report No. 5. With the air dryer/CO_2 absorption unit in operation this quarter, additional bands for these species were observed at 3550, 3400, 1670, 1615, 1110, and 1090 cm^{-1} (Figures 10 through 12).[1] Gypsum-hemihydrate appeared to be highly concentrated in the Pittsburgh feed coal and significantly less concentrated in the refuse.

Carbonate minerals typically exhibit a broad absorption band at about 1400 cm^{-1}, with additional diagnostic peaks at 871 cm^{-1} (calcite) and 875 cm^{-1} (dolomite).[2] Infrared spectroscopy alone is unable to differentiate the various carbonate minerals in the mid-infrared region, and thus the term "carbonates" will be used to collectively include the major minerals in the group (eg. calcite and dolomite). The highest concentration of carbonates was found in the Pittsburgh refuse as shown by the broad band at approximately 870 cm^{-1}. The feed coal was moderately concentrated with respect to carbonates while the clean coal was very low in carbonates (Figure 12). The sharp band at 1400 cm^{-1} in the spectrum of the Pittsburgh clean coal was the ammonium complex $(NH_4)^+$ (discussed in Quarterly Report No. 5, pp 29-30).

Quartz concentration was highest in the Pittsburgh refuse, but was also evident to a lesser degree in the feed and clean coal fractions.

Kaolinite concentration appeared to be highest in the Pocahontas clean coal fraction (Figure 13) with significantly lower levels in the feed coal and refuse fractions (Figures 14 and 15). This trend is in contrast to the Pittsburgh seam in which the kaolinite concentration was highest in the feed coal.

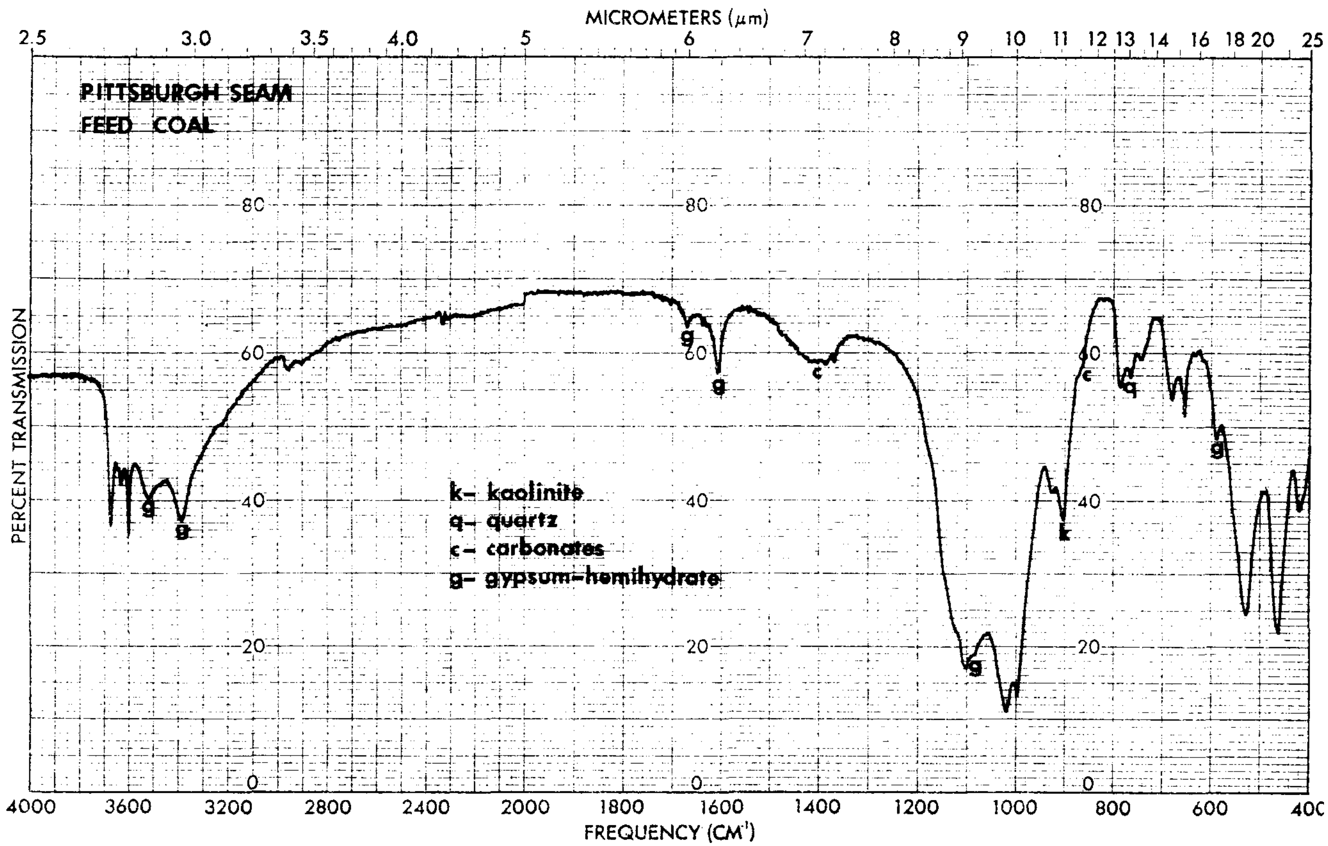


FIGURE 10

INFRARED SCAN OF PITTSBURGH SEAM FEED COAL HEAD SAMPLE

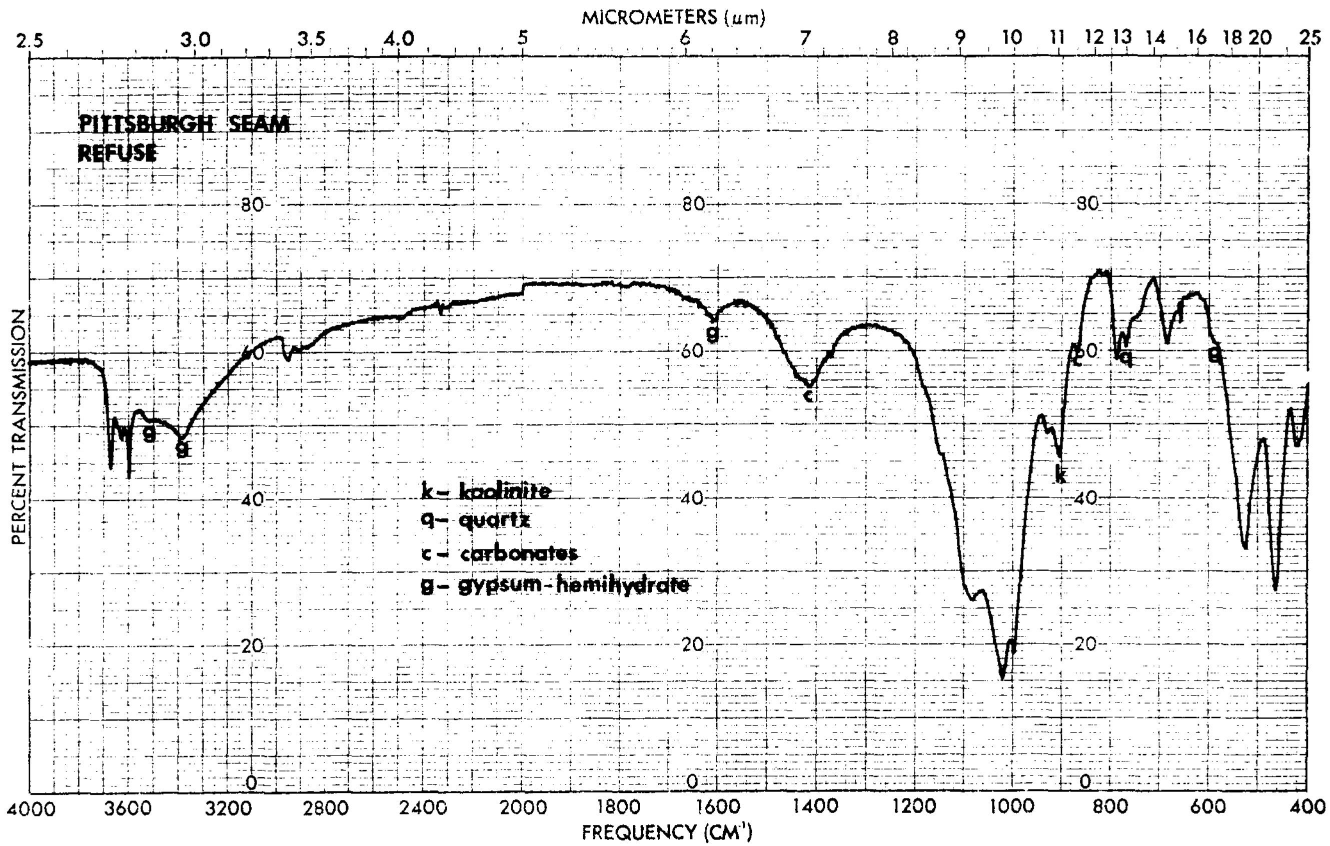


FIGURE 11

INFRARED SCAN OF PITTSBURGH SEAM REFUSE HEAD SAMPLE

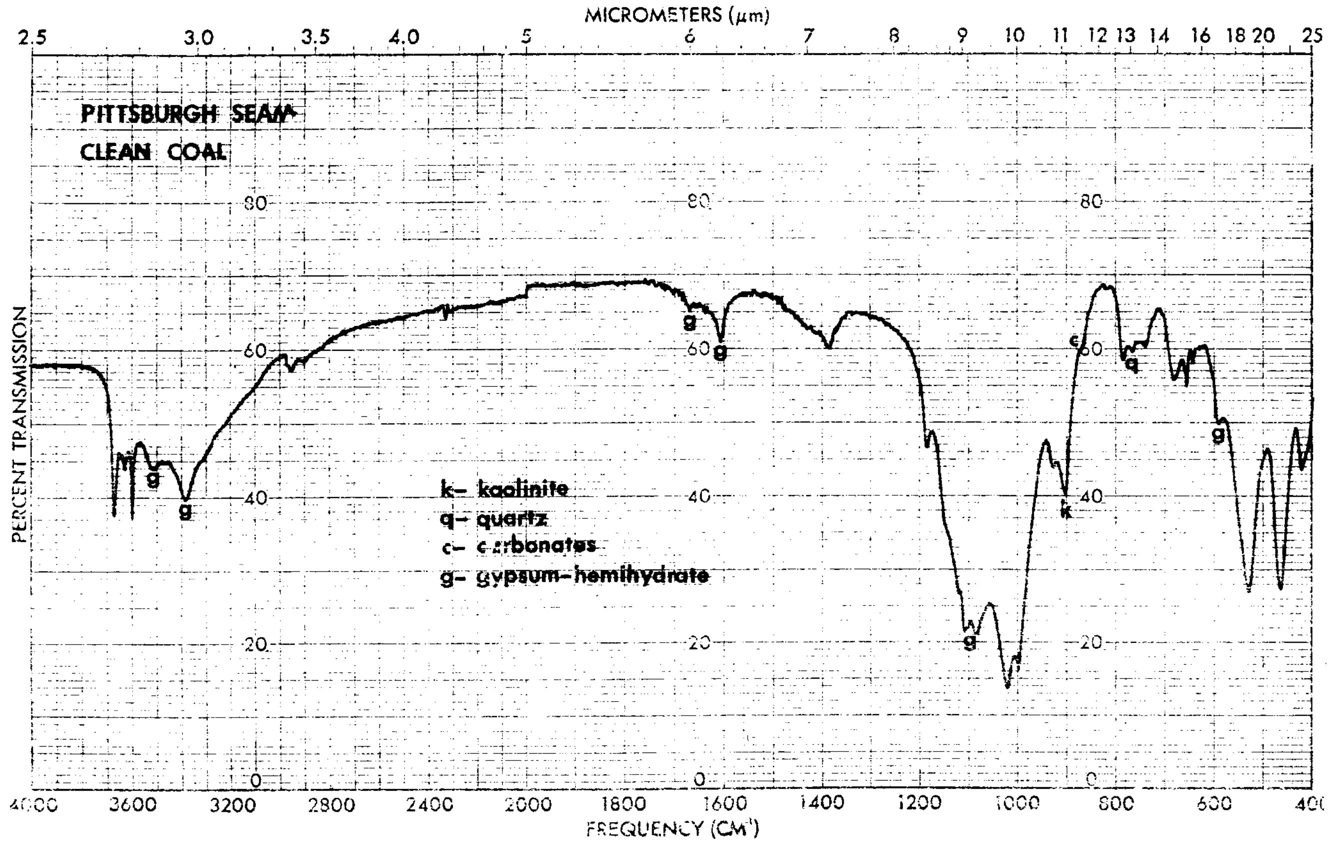


FIGURE 12

INFRARED SCAN OF PITTSBURGH SEAM CLEAN COAL HEAD SAMPLE

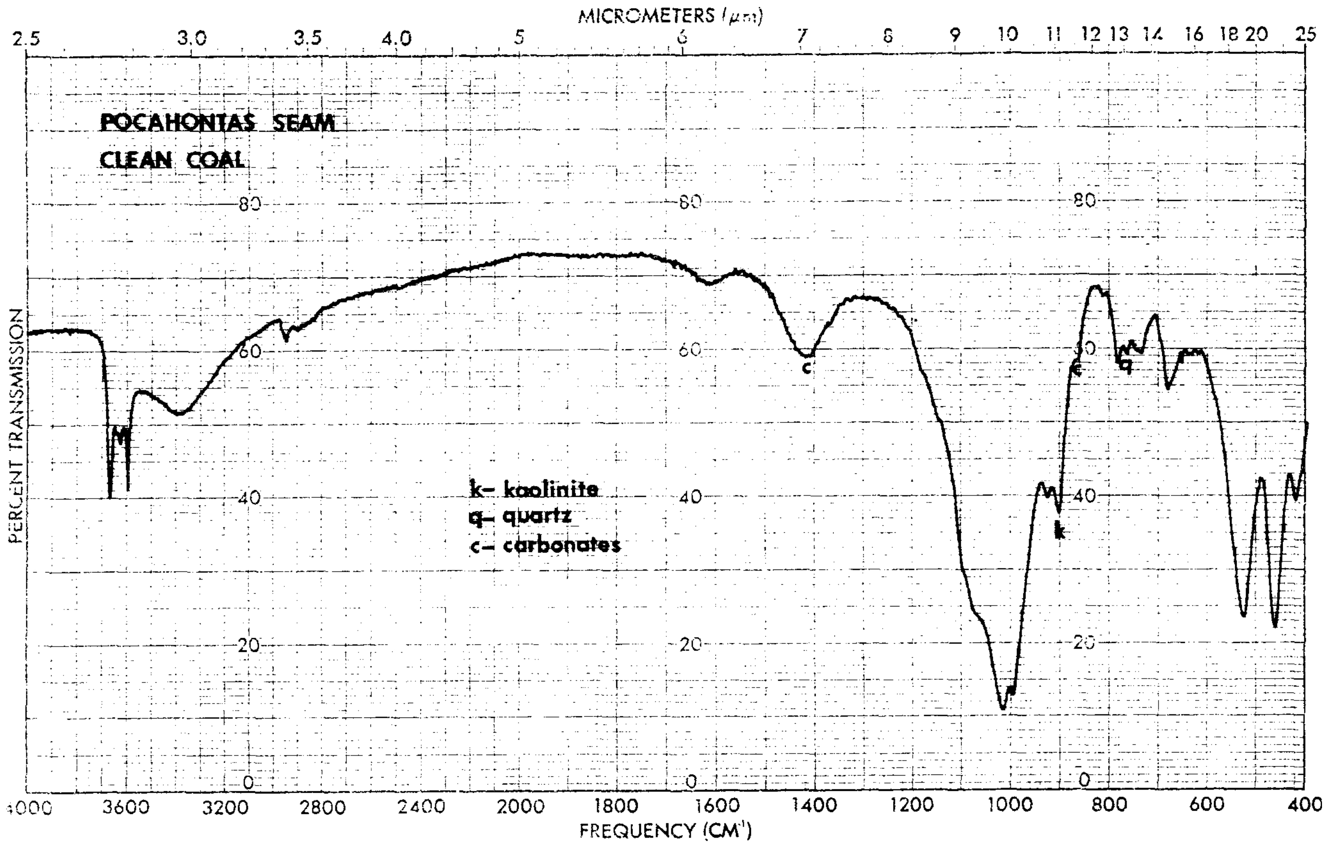


FIGURE 13

INFRARED SCAN OF POCAHONTAS NO. 3 SEAM CLEAN COAL HEAD SAMPLE

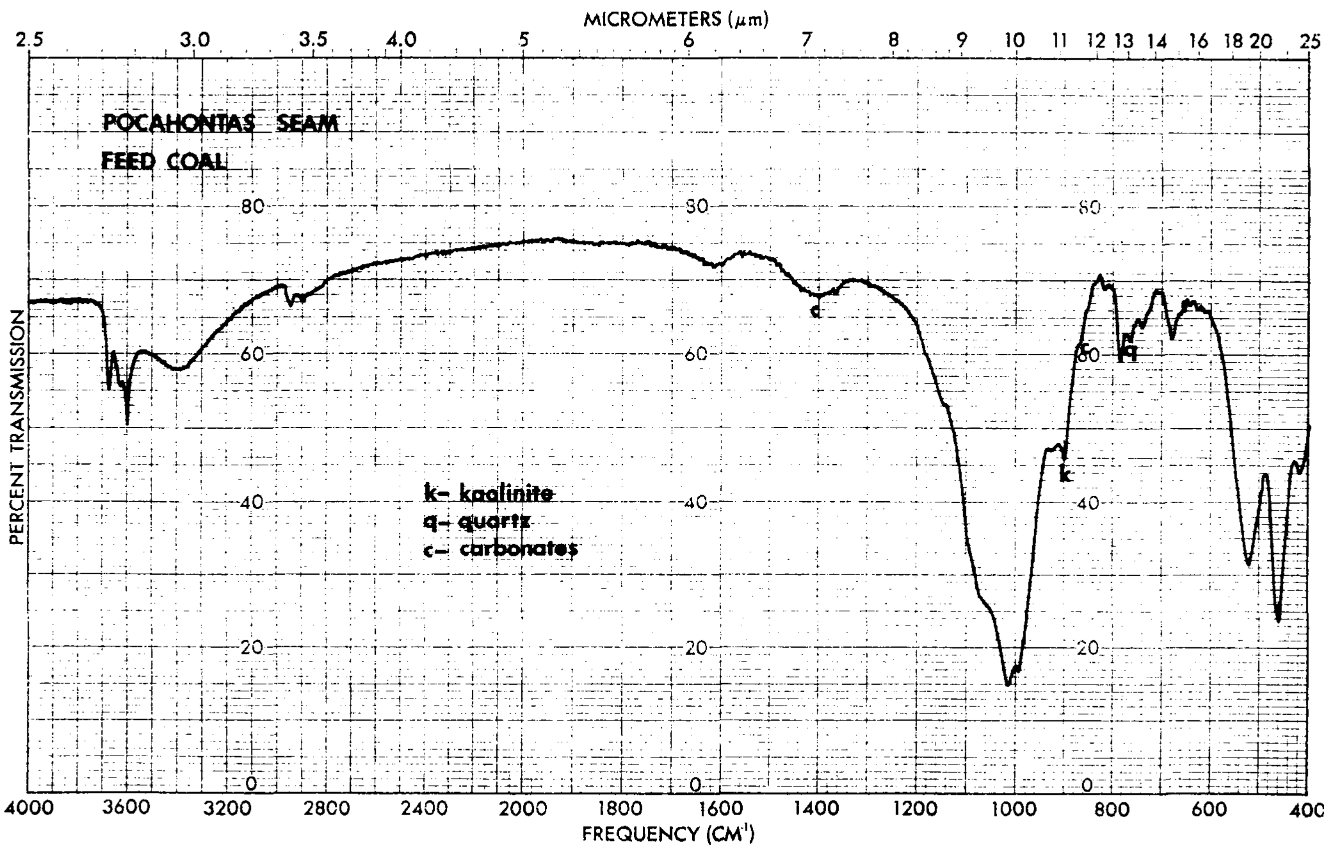


FIGURE 14

INFRARED SCAN OF POCAHONTAS NO. 3 SEAM FEED COAL HEAD SAMPLE

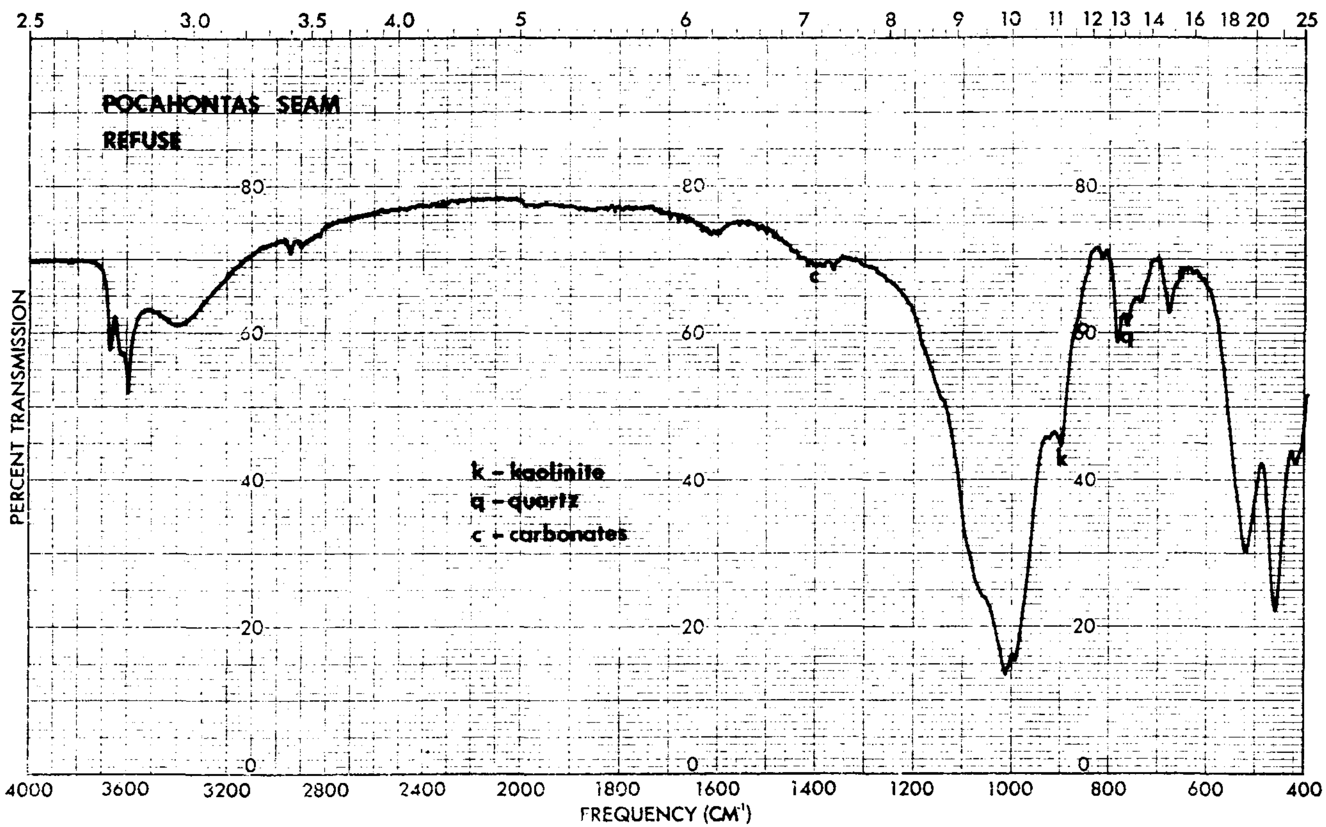


FIGURE 15

INFRARED SCAN OF POCAHONTAS NO. 3 REFUSE HEAD SAMPLE

While all spectra of the Pittsburgh coals studied contained gypsum-hemihydrate, no definitive bands for these species were observed in the spectra of the Pocahontas coals. Carbonates were highest in the Pocahontas clean coal, in contrast to the Pittsburgh seam in which the refuse fraction contained the highest carbonate concentration.

The amount of quartz appeared to be greatest in the refuse fractions of both of the coals studied.

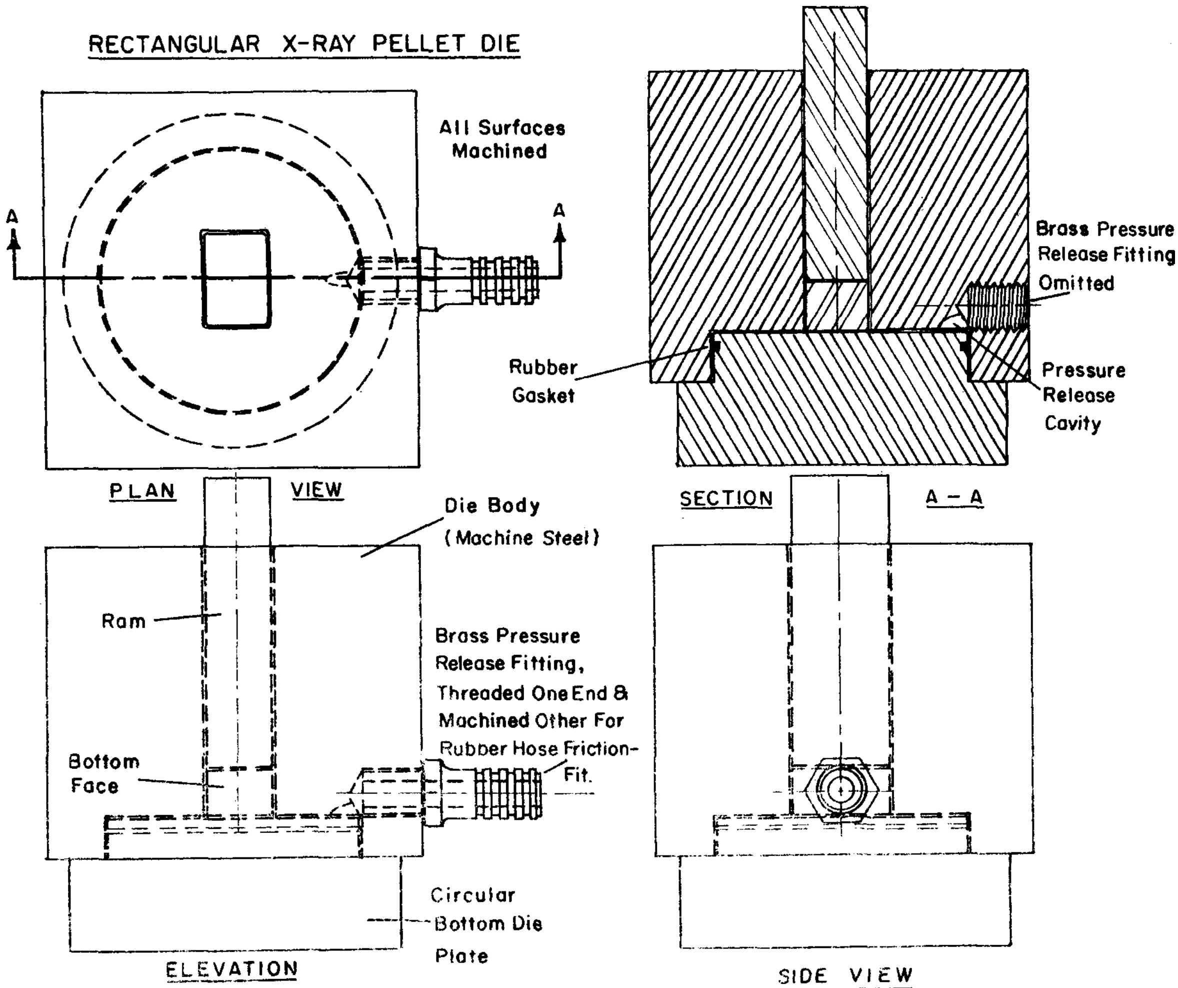
X-Ray Powder Diffraction (XRPD) - A rectangular X-ray pellet die was designed to press pellets to fit the sample holder of the Phillips APD-3501 X-ray diffraction unit. The die was designed to produce pellets slightly smaller than the internal dimensions of the sample holder to insure both a secure fit and prevent pellet breakage.

The pellet die consists of four fundamental parts: steel die body equipped with a brass pressure release fitting; steel circular plate; hardened steel bottom face; and hardened steel ram. (See Figure 16). The pressing surfaces of the ram and bottom face were polished to assure a smooth pellet surface. Pellets were prepared using a range of preweighed samples of LTA (100 mg - 250 mg) with methyl cellulose, 400 cp (400 mg - 600 mg) and with gum arabic (100 mg) as a binder for the lower-temperature-ashed coal.

Two methods of pellet preparation were attempted. Using the first technique, the backing or binder for a layered (LTA/binder) pellet was uniformly dispersed on the polished bottom face within the die and compressed by hand. The LTA was then evenly distributed over the binder. In the second, or dispersion method, the weighed portions of LTA with methyl cellulose and/or gum arabic were placed into the die as a mixture.

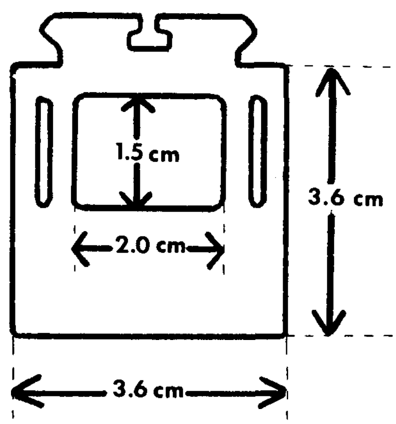
After positioning the ram (polished face down) into the body cavity, the die was placed on a hydraulic press and the samples were pressed

FIGURE 16


RECTANGULAR X-RAY PELLET DIE
All Surfaces Machined
A
A
PLAN VIEW
Brass Pressure Release Fitting Omitted
Rubber Gasket
Pressure Release Cavity
SECTION A - A
Die Body (Machine Steel)
Ram
Brass Pressure Release Fitting, Threaded One End & Machined Other For Rubber Hose Friction-Fit.
Bottom Face
Circular Bottom Die Plate
ELEVATION
SIDE VIEW

at 51,500 psi for two minutes. The pellets were removed from the die and placed face down on a glass plate. The sample holder was positioned over the pellet and pressed lightly to mount the pellet into the sample holder (Figure 17). Mounting in this manner allowed the pellet face to be flush with the sample holder surface.

Figure 17.



APD SAMPLE HOLDER

Initial tests of several dispersed-mixture pellets and layered pellets using a Phillips APD 3501 diffractometer were run at the West Virginia Geological Survey by Dr. John J. Renton. These preliminary tests indicated that the layered-pellet technique using a carefully dispersed layer of coal LTA backed by a layer of methyl cellulose will provide very acceptable X-ray scans with sharp peaks and a uniform, low background. Further investigation of this method of sample preparation and mounting (which is a modification of the LTA/coal layered pellet technique of Hildalgo and Renton, 1970[3]) using LTA/methyl cellulose will be presented in future reports.

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1. Nyquist, R. H., R. O. Kagel, "Infrared Spectra of Inorganic Compounds (3800-45 cm^{-1})," New York, 1971, p. 269.

2. O'Gorman, J. V., "Studies of Mineral Matter and Trace Elements in North American Coals," Ph.D. Dissertation, Pennsylvania State University, 1971, p. 44.

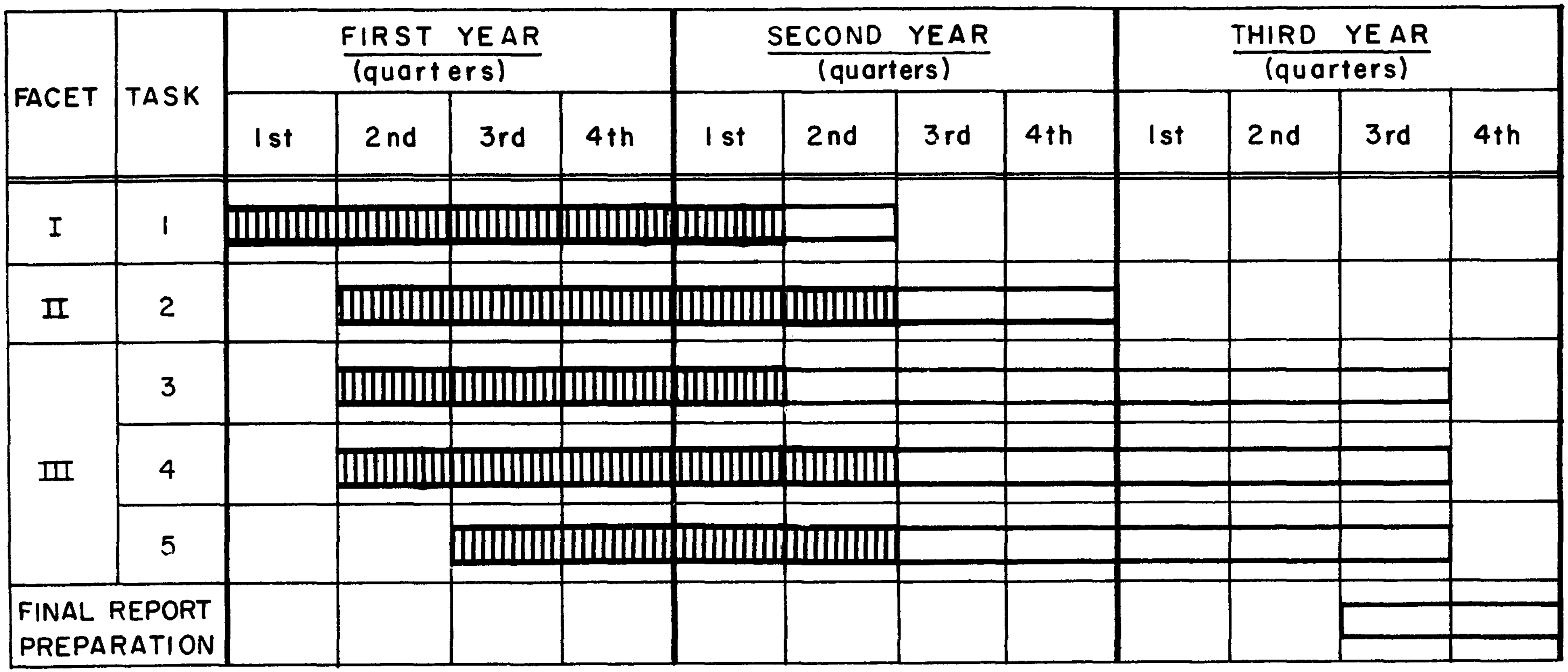
3. Hidalgo, R. V., J. J. Renton, "The Use of Pelletized Samples for X-ray Diffraction Analysis of Clay Minerals in Shale," West Virginia Geological and Economic Survey, Circular 12, 1970.

APPENDIX A

Milestone Chart

THE EFFECTS OF MINERALS ON COAL BENEFICIATION PROCESSES

West Virginia University



Legend

Scheduled

Progress

Figure I - WORK SCHEDULE

☆U.S. GOVERNMENT PRINTING OFFICE: 1980-640-258-1762