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THE EFFECTS OF MINERALS ON COAL BENEFICIATION PROCESSES

QUARTERLY REPORT NO. 5

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OBJECTIVE AND SCOPE

The purpose of this research program is to examine the effects 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

During quarter five, significant progress was achieved in a number of areas. Bulk amounts of raw coal, clean coal and refuse were collected on a field trip to a preparation plant in District 10, central Illinois. All samples were sealed in barrels and transported to Morgantown where sample processing was begun. Additional characterization of samples from the Pittsburgh and Pocahontas No. 3 preparation plants was completed and some of this data is discussed in the text. A number of modifications to the "pilot-scale" preparation plant were performed during the reporting period and some new equipment was added to the facility. Calcomp plots of washability data from static tests of the Pittsburgh Seam raw coal were made, and computerization of chemical and physical variables for Facet II was begun. Included in this computer data base are significant petrographic data on Pittsburgh Seam macerals which are discussed in the text. Financial and milestone data are reported in Appendix A.

DESCRIPTION OF TECHNICAL PROGRESS

Coal Preparation Plant Sampling

Bulk samples of a midwest preparation plant (feed, product, and refuse streams) were taken during one daylight shift. The plant is located in central Illinois, District 10, and processes the deep-mined Illinois No. 6 seam. The in-situ coal is bounded by a shale (overlain by limestone) roof and a fire clay floor.

Eight 55 gallon drums of raw (6 inch x 0) coal were collected using an "in-house" automatic beltline sampler. Samples were taken in barrel increments during about 3 hours of stream flow into the plant. Four 55 gallon drums of cleaned (2 inch x 0) coal, approximately 2000 pounds, were sampled directly from the load out facility.

Approximately 5600 pounds of coarse refuse were obtained directly from the load out hopper and transferred into eight 55 gallon drums. Five refuse samples were obtained from a three inch sampling pipe at the surge tank as the fine refuse slurry was discharged from the plant and prior to pumping to the disposal site. Six five gallon samples as well as two thirty gallon drums of fine refuse slurry were taken. The refuse slurry (about 25 percent solids) is composed of -28 mesh coaly solids and clay slimes. Due to boggy conditions around the settling ponds, no dewatered fines samples were collected.

All sample containers were sealed, labelled, and transported to Morgantown for processing.

Coal Preparation Pilot Plant

Work is continuing to make all modifications necessary for operation of the pilot scale, batch operation, coal cleaning facility. The facility consists of a crushing and screening operation, a single compartment jig washer, a lab scale tabling operation, a heavy media magnetite drum separation (HMS) unit and a cycloning stage which can be used as either a heavy media or hydrocyclone operation. This pilot plant will be used to study various methods of coal cleaning in order to produce a tailor-made product and to study the effects of the coal minerals inherent in the samples on coal beneficiation processes.

At this stage of the contract sufficient information from Facet II, Task 2 (Mineral Matter Distribution Data) has not been obtained to warrant full-scale pilot plant testing. Presently each unit operation is being optimized and the collection of "base line data" is underway. As soon as sufficient mineral matter distribution data is available the pilot plant will be used to reproduce on a pilot scale those conditions found to be desirable from the evaluation of bench-scale testing results.

The construction of an elevated platform was initiated during the last quarter of work. This platform will provide space for a 200 gallon head tank to supply water at high volume rates to the McNally-Pittsburg jig and space for a heavy media slurry tank for the Wemco heavy media drum separator. A six inch laboratory jaw crusher was also installed and electrical wiring for all plant equipment checked for code uniformity and upgraded where necessary.

Jig Tests

Parts necessary to repair the float mechanism of the McNally-Pittsburg "Baum" type jig were received during the quarter. The mechanism operates the refuse discharge gate which controls the effective gravity of separation.

It was determined that to produce a steady state condition of jig operation, where the coal feed rate, air volume, discharge rate and water flow are permitted to reach equilibrium, a much larger water supply was needed. To insure sufficient time to reach steady state, an elevated 200 gallon head tank will be connected to the jig by a 3" diameter line. Replenished by a 1" water line, this tank will permit operation at larger volumes of water flow for longer periods of time than the present system. The raised platform which holds the 200 gallon tank is being constructed to hold two such units to double the capacity if necessary, and/or to provide water for other equipment operating simultaneously.

Wemco Heavy Media Drum Separator

A new pump for the recirculation of the magnetite heavy-media slurry was procured during the quarter. A 3 horsepower motor-driven pump replaced two 3/4 horsepower units used previously. Using the platform constructed for the head tank, an agitated-media make-up sump/surge tank is to be elevated above the drum separator permitting more rapid adjustments in media make-up than could be achieved with the pump alone. A second dewatering screen has been ordered to permit the simultaneous sampling of both product and refuse streams from the heavy media separator.

Concentrating Table

A preliminary cleaning efficiency test was performed to ensure the proper operation of the pilot scale concentrating table prior to testing for effects on mineral concentration and distribution. Test results reported in Table 1 indicate that satisfactory operating parameters for this unit have been determined. The starting size of samples to be fed across the table in future tests will be 3/16 inch x 100 mesh. As can be seen in Table 1, ash was reduced while volatile matter and fixed carbon were increased by the tabling process. The sulfur reduction for the Pittsburgh seam sample and slight increase for the Pocahontas seam sample reflect the basic difference in sulfur forms in the two samples. Tabling is most effective in removing pyritic sulfur impurities prevalent in the Pittsburgh coal but present in much smaller amounts in the Pocahontas coal. By simply removing ash, the sulfur content increases as the inherent organic sulfur is increased along with the coal. The simultaneous removal of pyritic sulfur with the ash helps to keep the overall sulfur increase small in this case, i.e., from 0.68 to 0.70 percent.

Cyclone Tests

Preliminary results of the cyclone efficiency tests performed last quarter revealed that correct operating parameters had not been determined for the 3-inch heavy media cyclone. A larger 8-inch heavy media cyclone as well as an 8-inch water washing cyclone will be evaluated for use in the contract studies. Results from additional tests to determine operating parameters for all three units will be reported as they become available.

TABLE 1

PRELIMINARY CONCENTRATING TABLE TEST RESULTS
CLEANING EFFICIENCY

Pittsburgh Seam Coal

	Wt. %	Ash %	Sulfur %	Volatile Matter %	Fixed Carbon %
Feed	100.0	11.0	2.82	37.9	51.1
Clean	86.6	6.0	2.29	39.6	54.4
Reject	13.4	43.5	6.27	26.6	29.9

Pocahontas No. 3 Seam Coal

	Wt. %	Ash %	Sulfur %	Volatile Matter %	Fixed Carbon %
Feed	100.0	16.9	0.68	17.0	66.1
Clean	84.1	5.2	0.70	17.3	77.5
Reject	15.9	78.6	0.56	15.7	5.7

Froth Flotation Tests

In the fifth quarter, plans were reviewed for the initiation of froth flotation testing, and some initial laboratory work was performed. Preliminary plans for the integration of froth flotation into the Coal Research Bureau's coal preparation pilot plant (Quarterly Report No. 4, Contract EF-77-S-01-2722), call for the removal of -100 mesh raw feed coal at points in the flow scheme for beneficiation using froth flotation. Froth flotation is the only method of treatment in the pilot-scale plant not dependent upon specific gravity differences in the individual particles for a separation.

In flotation, the separation of coal and non-coal material depends upon the attachment of an air bubble to the surface of a coal particle immersed in water. To be effective, it is usually necessary to alter the surface of the coal particle and enhance bubble attachment by the addition of a chemical to the treatment cell. However, depending on the method employed, the surfaces of both coal and non-coal particles may be altered to achieve a sharper separation.

At this time, plans call for the use of a Denver Model D-12* laboratory flotation machine (See Figure 1). The impeller speed of this machine is variable and a variety of sample sizes (250-2000 gram) may be tested in the apparatus. Methylisobutylcarbinol (MIBC) will be used to alter the surface of the coal particles and float a coal product. The following parameters have been selected for the pilot-scale froth flotation tests. A 500 gram sample of fine feed coal will be combined with 2500 ml of distilled water in the flotation cell (the pH and temperature of the water will be recorded prior to mixing with the fine-coal). MIBC will be added to this mixture to form concentrations ranging from 0.01 to 1.0 percent, depending on the nature of the coal tested. At this point, the coal will be allowed to condition in the water and MIBC at an impeller speed of 1500 rpm. After 15 minutes, an additional 2500 ml of water will be added to the cell and an air valve will be opened to force air through the cell and promote froth formation. The froth will be collected as overflow from the cell for one minute. Both the froth product and the remaining solids in the cell will be filtered, dried, and submitted for standard chemical and mineralogical analyses.

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* The use of brand names in no way implies recommendation or endorsement of these products by the Coal Research Bureau or the U. S. Department of Energy.

Figure 1. Froth flotation cell, lab scale.

A preliminary test on a 500 gram sample of -100 mesh Pittsburgh seam coal was performed during the fifth quarter to check the operation of the flotation equipment. The results (See Table 2) indicated that the equipment was functioning properly.

TABLE 2

PRELIMINARY FROTH FLOTATION TEST RESULTS

Pittsburgh Seam Coal

	Moisture %	Ash %	Volatile Matter %
Feed	1.2	21.2	33.1
Float	0.9	8.9	34.9
Refuse	0.9	34.6	27.9

	Fixed Carbon %	Total Sulfur %	Btu
Feed	45.7	3.60	11,901
Float	56.2	2.97	13,969
Refuse	37.5	4.13	-

Characterization of Coal Samples

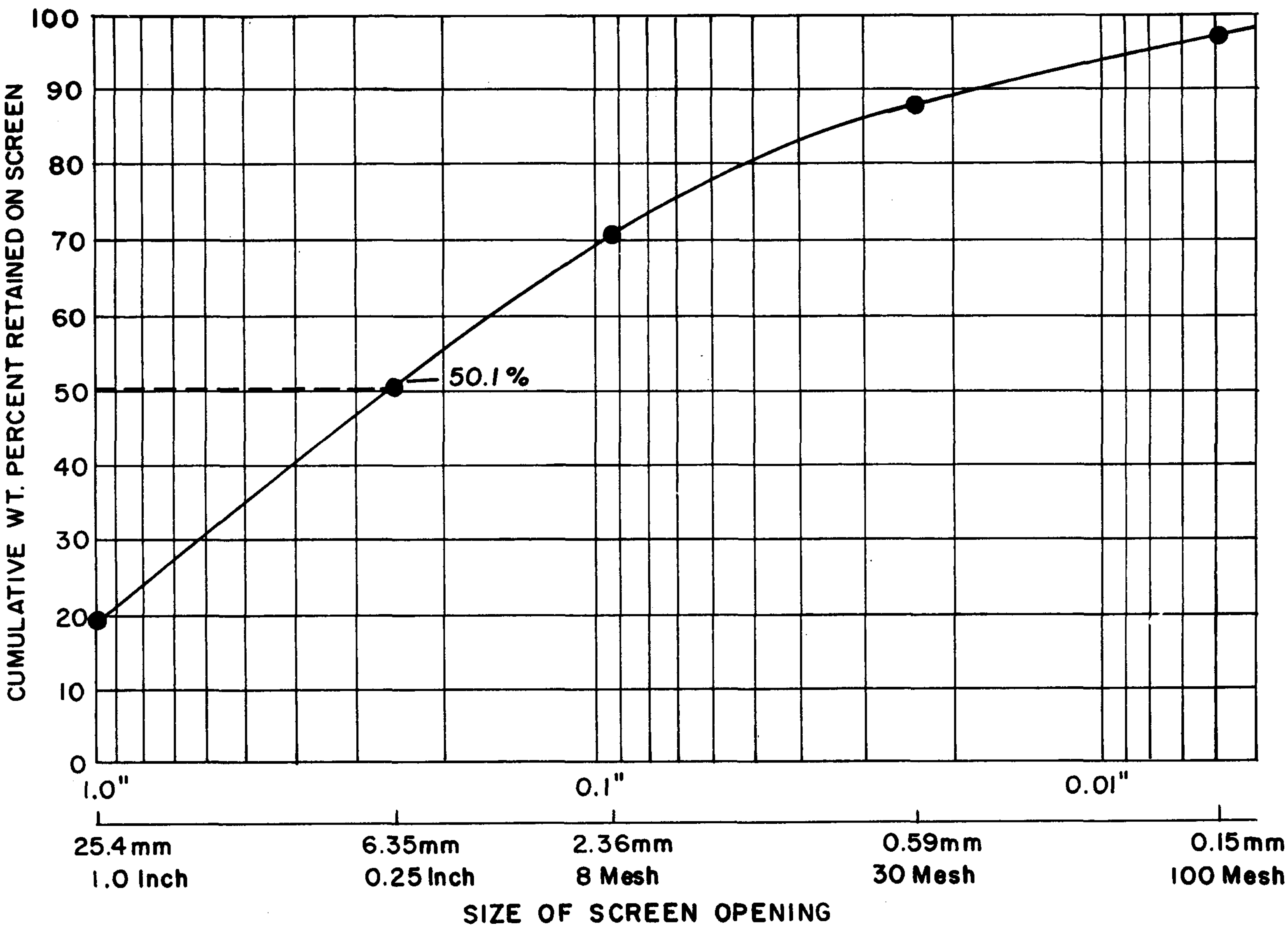
The following section is a review of progress in the physical, chemical, and mineralogical characterization of samples from Facet I.

Physical Characterization

A cumulative logarithmic plot of the screen analysis of a head sample of Pittsburgh Seam bituminous coal is presented in Figure 2. This coal is crushed to 5" x 0 size prior to cleaning at the preparation plant. As can be seen from the figure approximately 50 percent of this material is greater than 1/4" in size.

After coning and quartering, the Pittsburgh raw coal head fraction was divided into representative sizes using the following screens: 1 inch (25.4 mm), 1/4 inch (6.35 mm), 8 mesh U.S.S. (2.36 mm), 30 mesh (0.59 mm) and 100 mesh (0.15 mm). These screen sizes were selected as they represent approximate size limitations for the various pilot plant coal cleaning operations, and because they provide sufficient data for plotting purposes. The following raw coal screen fractions were subsequently laboratory cleaned using static float-sink lab tests: +1 inch, 1 inch x 1/4 inch, 1/4 inch x 8 mesh, 8 mesh x 30 mesh U.S.S. (28 mesh Tyler) and 30 mesh x 100 mesh. The test fractions were then collected and analyzed with special emphasis on ash and sulfur distribution. A portion of the minus 100 mesh fraction was submitted for chemical analysis and a large amount was reserved for froth flotation studies.

FIG. 2 - PITTSBURGH RAW COAL (CUMULATIVE LOGARITHMIC PLOT OF SCREEN ANALYSIS)



Figures 3-7 represent a cal-comp plot of the sulfur and ash washability data reported last quarter and Figure 8 is a plot of the composite (+100 mesh) sample data.

This information in conjunction with other data obtained from Facet II will be used to describe what effects laboratory coal cleaning processes, such as specific gravity separation, etc. have on the distribution of mineral constituents originally present in the coal.

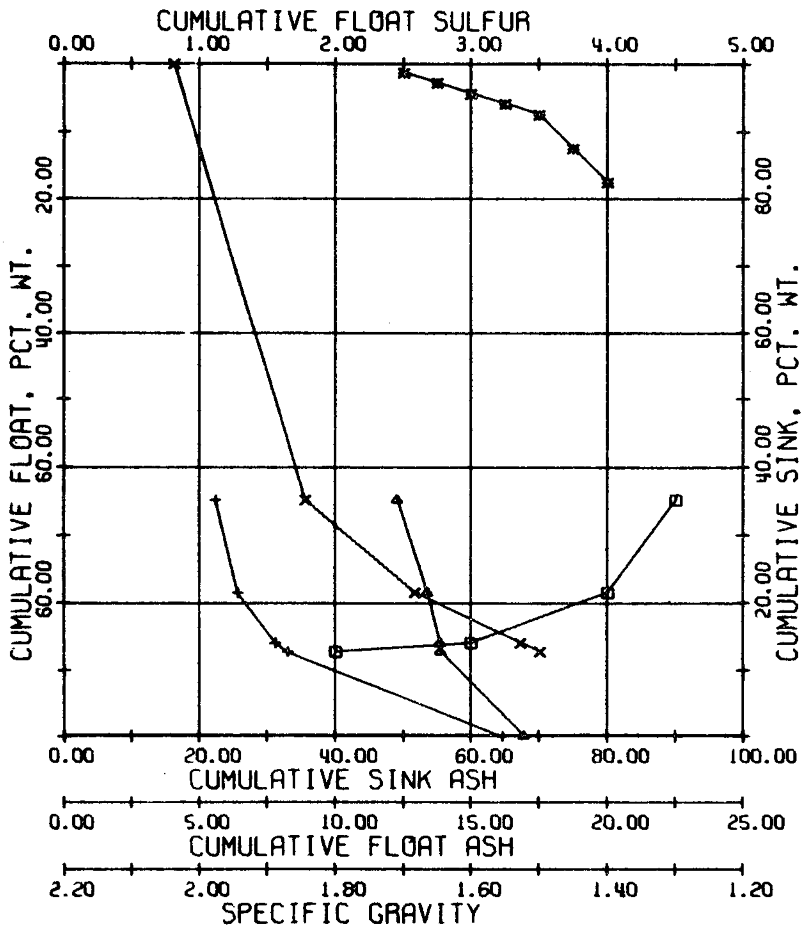
Chemical Characterization

A comparative tabulation of the chemical characterization data for Pittsburgh Seam "Head Samples" (raw preparation plant feed coal, a recombined "composite" raw coal head sample, the clean coal plant stream, and the pulverized refuse) is presented in Table 3. To simplify this data comparison, the ratios of the elemental concentrations (for six elements and for sulfur forms) in the Pittsburgh Head Samples are presented in Table 4. For example, the ratio of silicon content in the raw coal head sample to the amount found in the refuse head sample is 2.98 to 14.77 percent Si respectively (or a ratio of 0.20). In this manner the Table 3 data was used to generate Table 4. The ratio data point out the relative concentrations of these elements in the raw coal vs. refuse, clean coal vs. refuse, and the ability of the cleaning facility to change these concentrations indicated in the clean coal vs. raw coal comparison.

Table 3 also shows relative levels of sulfur forms in the coal and refuse components, and points out the reverse levels of pyrite S (found in large amounts in the refuse) and organic S (which is more concentrated in the coal).

FIGURE 3

SULFUR AND ASH WASHABILITY
PGH.RAW COAL +1-IN.FRACTION
19.63 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
PGH. RAW COAL 1-IN.X1/4-IN. FRACTION
30.44 WT. PCT. OF TOTAL SAMPLE

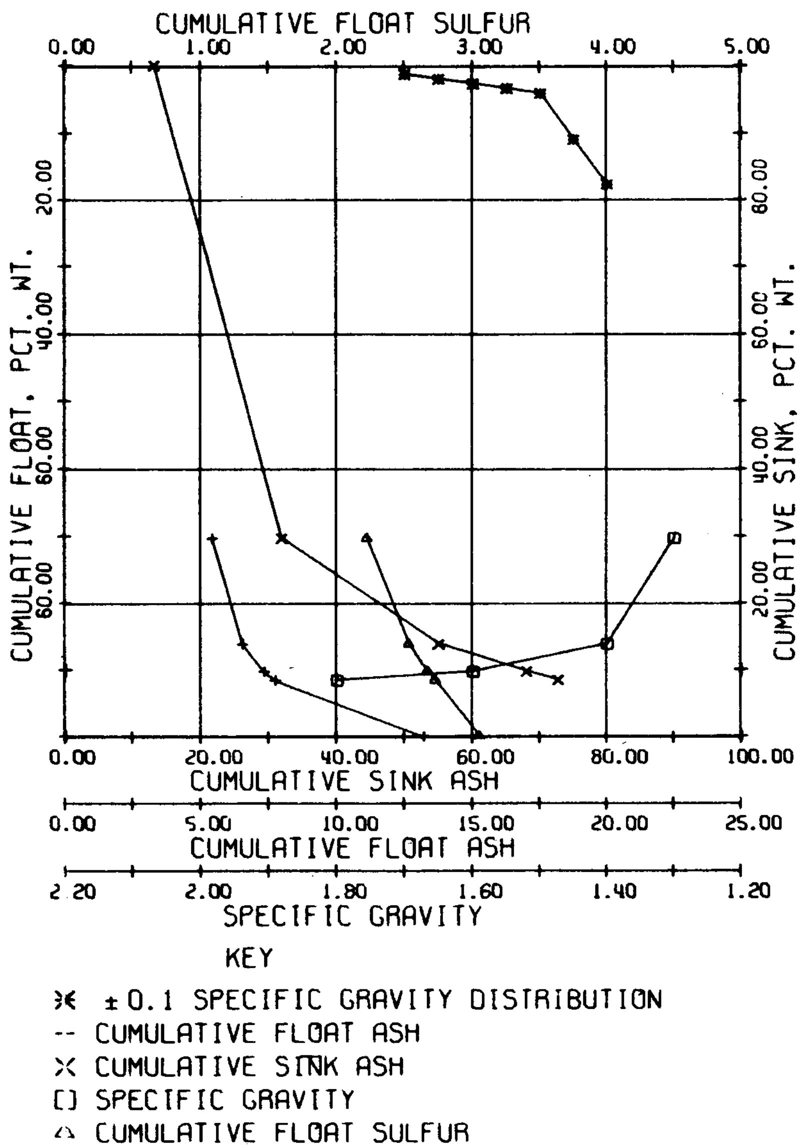
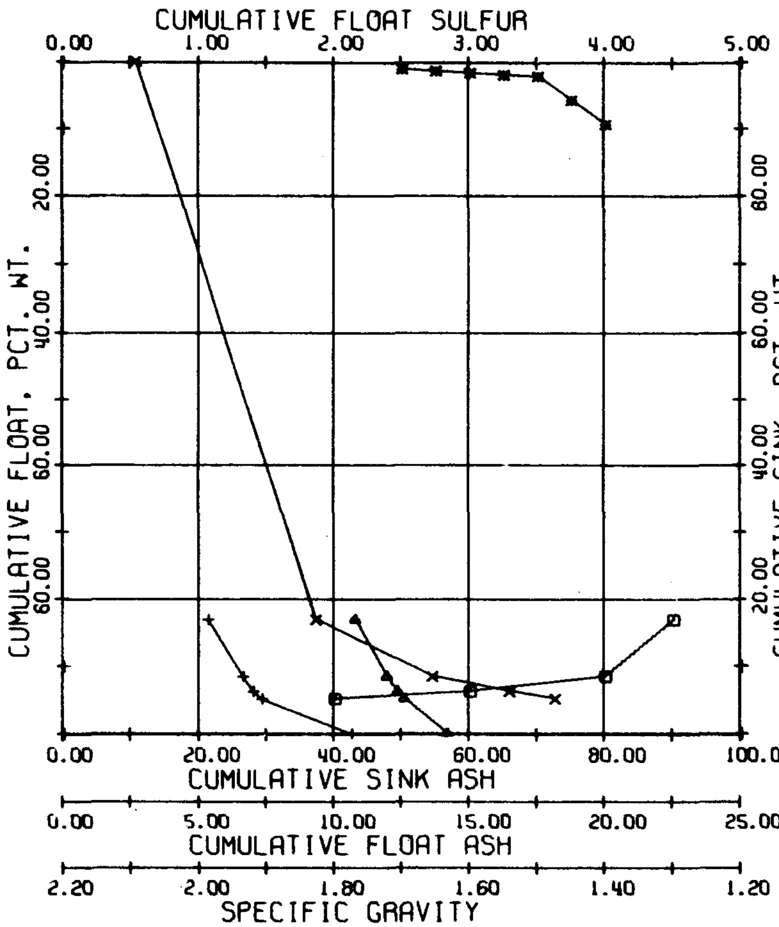


FIGURE 5

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

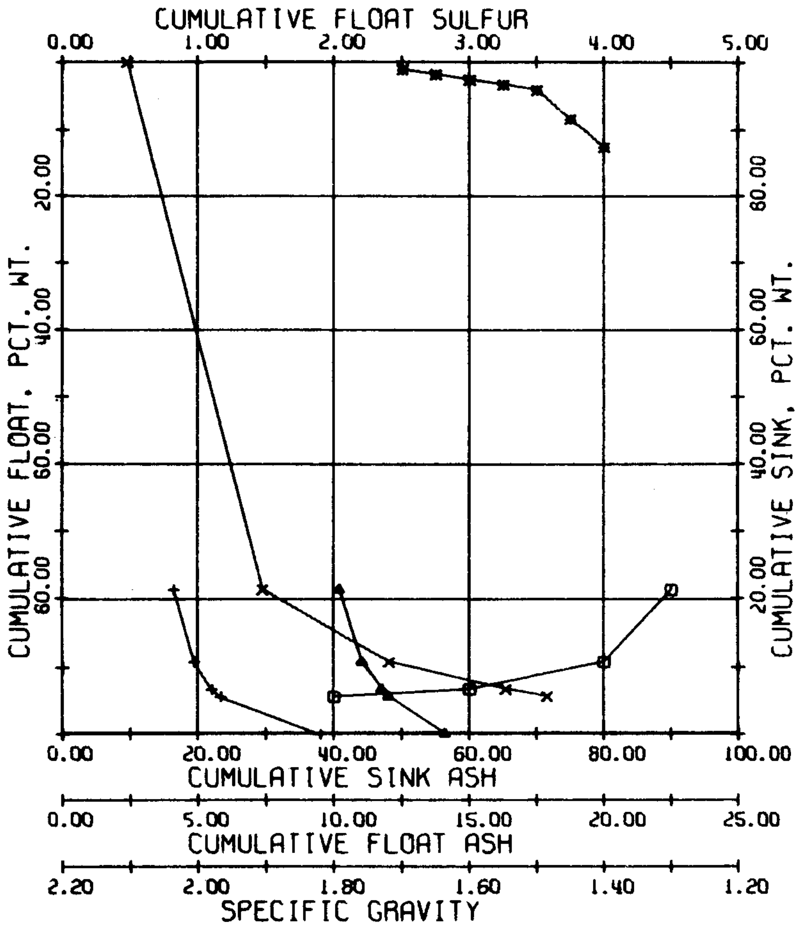


KEY

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

FIGURE 6

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

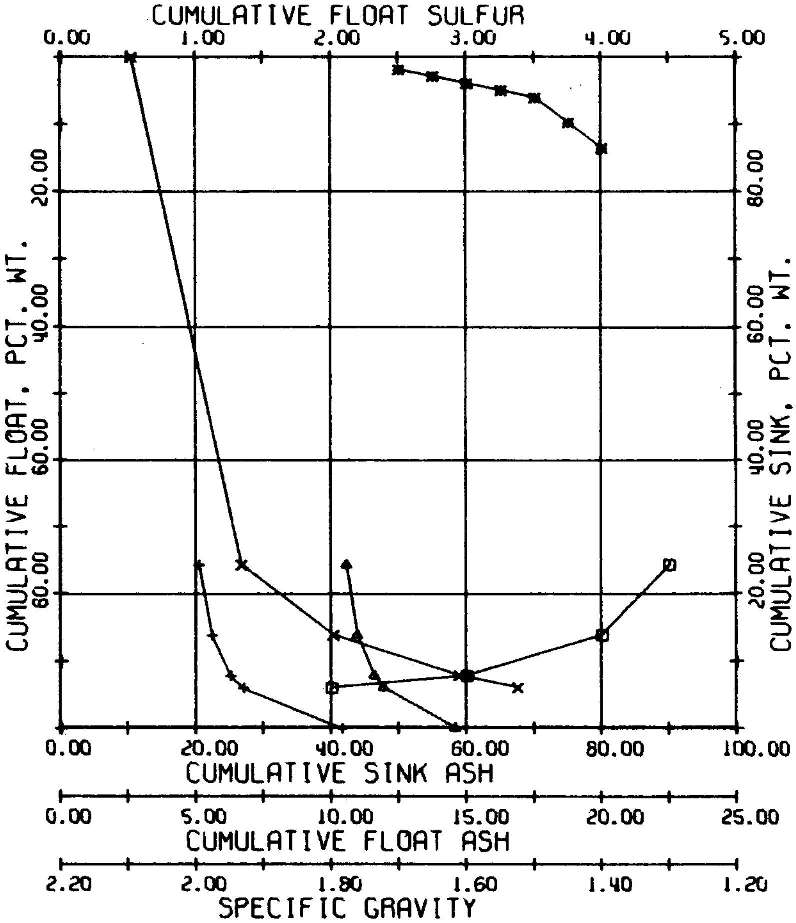


KEY

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

FIGURE 7

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

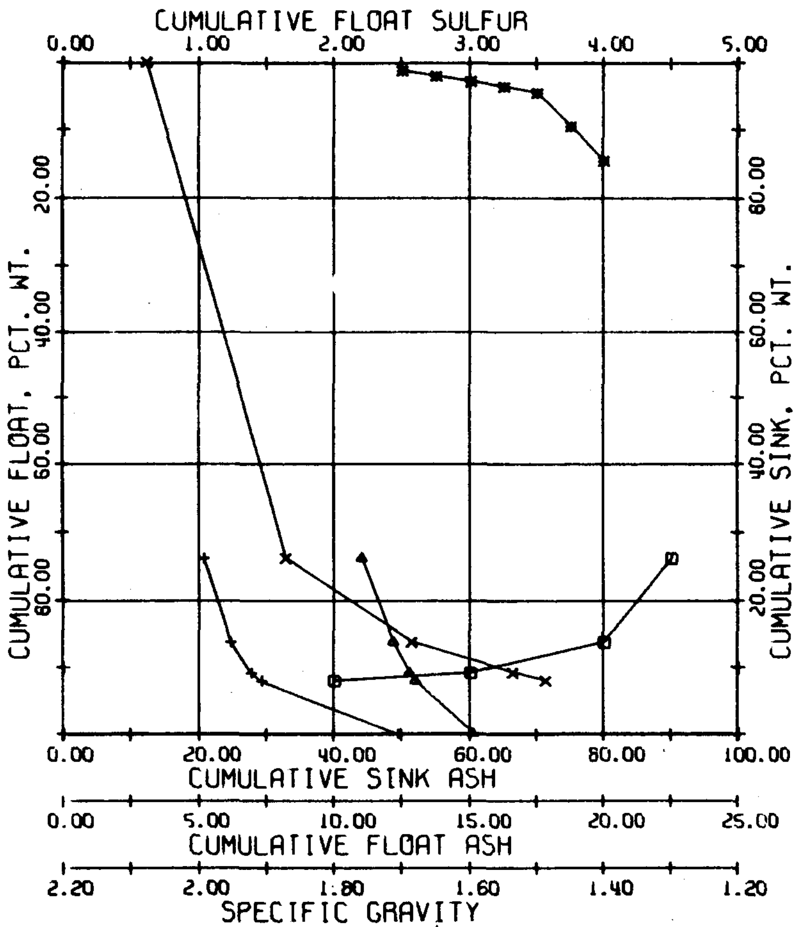


KEY

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

FIGURE 8

SULFUR AND ASH WASHABILITY
PITTSBURGH RAW COAL COMPOSITE FRACTION
96.73 WT. PCT. OF TOTAL SAMPLE



KEY

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

TABLE 3

CHEMICAL CHARACTERIZATION DATA*
(Pittsburgh Head Samples)

	Spectrographic Analysis**						Atomic Absorption***			
	Si	Al	Fe	Ti	Ca	Mg	Na	K	LTA	Btu
Raw Coal	2.98	1.56	1.83	0.08	0.63	0.10	0.07	0.34	17.3	13,102
Raw Coal Composite+	2.65	1.29	1.33	0.06	0.59	0.10	0.08	0.15	15.8	13,318
Clean Coal	1.66	1.07	1.20	0.05	0.40	0.06	0.07	0.08	10.1	14,020
Refuse	14.77	6.00	9.18	0.29	4.77	0.52	0.31	0.52	83.2	4,403

	Proximate Analysis				Sulfur Breakdown			
	Moisture	Ash	Volatile Matter	Fixed Carbon	Sulfate S	Pyrite S	Organic S	Total S
Raw Coal	1.0	13.9	36.5	50.4	0.04	1.57	1.40	3.01
Raw Coal Composite	0.9	12.9	37.9	49.2	0.01	1.50	1.50	3.01
Clean Coal	1.2	8.0	39.5	52.5	0.01	1.09	1.60	2.70
Refuse	0.7	71.3	19.5	9.2	0.11	8.35	0.37	8.83

* Data reported on moisture free basis.

** Percent element on a moisture free whole coal basis.

*** Percent element in the ash.

\+ Recombined head sample pro-rated on weight percent of component gravity fractions.

Additional characterization work is continuing in this area on both the Pocahontas No. 3 and Illinois No. 6 seams and will be reported in the near future.

TABLE 4

RATIO OF ELEMENTAL CONCENTRATIONS IN HEAD SAMPLES

Pittsburgh Head Samples	Si	Al	Fe	Ti	Ca	Mg
Raw Coal/Refuse	0.20	0.26	0.20	0.27	0.13	0.19
Clean Coal/Refuse	0.11	0.18	0.13	0.17	0.08	0.12
Clean Coal/Raw Coal	0.56	0.69	0.66	0.63	0.63	0.60

	Sulfur Forms			
	Sulfate S	Pyrite S	Organic S	Total S
Raw Coal/Refuse	0.36	0.19	3.78	0.34
Clean Coal/Refuse	0.09	0.13	4.30	0.31
Clean Coal/Raw Coal	0.25	0.69	1.10	0.89

* Based on Table 3 data.

Mineralogical Characterization

Although problems in installing the X-ray unit have continued to delay this phase of the contract, significant work in both the petrographic maceral analysis of the Pittsburgh Seam, and in establishing working curves for the infrared unit has been accomplished.

Petrographic Analyses- Petrographic analyses of the District 3 Pittsburgh coal identified 15 measurable constituents in amounts of 2 percent by volume or greater. Fourteen of these are macerals and submacerals presented in Table 6 of Quarterly Report No. 4, and the fifteenth constituent comprises the total mineral matter. Mineral species within the mineral matter have yet to be quantitatively distinguished. Results of the petrographic analyses of the 29 Pittsburgh coal samples are presented in Table 5. Each data point presented in Table 5 has an average expected error of $\pm$ 3 percent (ref. Equations in Quarterly Report No. 3). Because of the magnitude of this error, it was decided that any variable not present in an amount greater than or equal to 2 percent in at least one sample would be excluded from further examination. Macerals and submacerals observed but deleted in this manner included vitrodetrinite, cutinite, pyrosemifusinte, and sclerotinite.

Prior to the application of statistical tests to the data, the normality of the distributions of the 29 values for each variable (maceral group, maceral, and submaceral) was inspected. This was accomplished through the use of frequency distributions shown in Figure 9. It was anticipated that

TABLE 5

PETROGRAPHIC ANALYSES OF PITTSBURGH COAL FLOAT-SINK FRACTIONS AND HEAD SAMPLES PRESENTED AS VOLUME PERCENT OF THE WHOLE COAL

	+1"					1" x 1/4"					1/4 x 8 Mesh				
	1.3F	1.4F	1.6F	1.8F	1.8S	1.3F	1.4F	1.6F	1.8F	1.8S	1.3F	1.4F	1.6F	1.8F	1.8S
VITRINITE	78	66	54	27	6	80	64	48	32	8	79	66	52	31	8
Telinite	1	0	1	0	0	0	1	0	0	0	1	1	1	1	0
Collinite	77	66	53	27	6	80	63	47	31	7	78	65	51	30	8
Telocollinite	76	62	50	26	5	77	57	42	27	7	75	61	44	26	7
Desmocollinite	1	3	2	1	1	2	5	5	5	0	2	4	6	4	1
EXINITE	4	6	4	5	1	4	5	6	6	1	5	4	4	6	1
Sporinite	2	2	1	0	0	2	2	2	1	0	3	1	1	1	0
Cutinite	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Liptodetrinite	1	4	3	4	1	2	4	3	5	1	2	2	3	5	1
INERTINITE	13	16	16	22	2	10	17	21	24	5	12	15	23	27	6
Fusinite	1	1	3	3	0	1	1	4	8	1	1	2	4	9	2
Pyrofusinite	0	1	2	2	0	0	0	3	5	1	0	1	2	4	1
Degradofusinite	1	0	1	2	0	1	0	1	3	0	0	0	2	5	1
Semifusinite	4	6	3	3	0	4	7	7	4	0	3	6	9	4	1
Pyrosemifusinite	0	0	0	0	0	0	0	0	1	0	0	1	1	1	0
Degradosemifusinite	4	5	3	3	0	4	6	6	3	0	3	6	8	3	1
Macrinite	1	1	3	5	1	1	1	1	4	0	1	2	2	5	0
Micrinite	3	2	2	1	0	3	3	1	1	0	2	1	1	1	0
Inertodetrinite	4	5	5	9	1	3	6	8	8	3	3	4	6	7	3
MINERAL MATTER	5	12	26	46	91	6	13	26	37	86	4	15	21	36	86

TABLE 5 (Continued)

PETROGRAPHIC ANALYSES OF PITTSBURGH COAL FLOAT-SINK FRACTIONS AND HEAD SAMPLES PRESENTED AS VOLUME PERCENT OF THE WHOLE COAL

	8 x 28 Mesh					28 x 100 Mesh					-100 Mesh Screen Fraction	Raw Coal Head	Clean Coal Head	Refuse Head
	1.3F	1.4F	1.6F	1.8F	1.8S	1.3F	1.4F	1.6F	1.8F	1.8S				
VITRINITE	83	67	58	31	10	85	73	64	41	10	75	75	77	13
Telinite	2	1	0	0	0	1	0	0	0	0	1	0	1	0
Collinite	82	67	58	31	9	85	72	64	41	10	74	74	76	12
Telocollinite	78	59	55	28	9	81	66	58	38	10	72	70	71	12
Desmocollinite	3	7	3	3	1	3	7	5	3	0	2	5	4	1
EXINITE	3	3	2	2	1	3	3	4	3	0	1	3	4	0
Sporinite	2	2	0	1	0	1	2	1	0	0	0	1	2	0
Cutinite	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Liptodetrinite	1	1	2	2	0	1	2	3	2	0	1	2	2	0
INERTINITE	9	18	22	30	7	7	16	20	27	7	15	11	12	5
Fusinite	1	2	4	9	2	1	2	3	12	4	1	1	1	1
Pyrofusinite	0	1	2	6	1	0	1	2	8	3	1	1	1	1
Degradofusinite	0	1	3	5	2	1	1	2	4	2	0	0	1	0
Semifusinite	3	7	9	5	1	2	6	10	6	1	3	4	4	1
Pyrosemifusinite	0	1	1	1	0	0	1	1	1	0	0	0	0	1
Degradosemifusinite	3	7	8	4	1	2	5	9	5	1	3	4	4	1
Macrinite	0	2	2	6	1	1	1	1	4	0	0	0	0	0
Micrinite	3	1	1	0	0	2	2	1	1	0	2	1	2	0
Inertodetrinite	2	5	6	8	2	2	5	4	5	1	9	5	4	2
MINERAL MATTER	5	12	18	36	83	5	8	12	30	83	8	11	7	82

through sizing and specific gravity fractionation, resulting in the deliberate separation of minerals and macerals, that these values would be non-normally distributed. This type of fractionation should result in the values being skewed to both the high and low ends of the frequency distributions for some variables. Figure 9 illustrates this inherent problem with the data. Only inertinite, semifusinite, exinite, and liptodetrinite appear to be normally or near-normally distributed. The number of cells used in the frequency distributions was chosen based upon the empirical equation derived by Sturges:[1]

$$K = 1 + 3.3 \log n$$

where K = the optimum number of cells or classes,

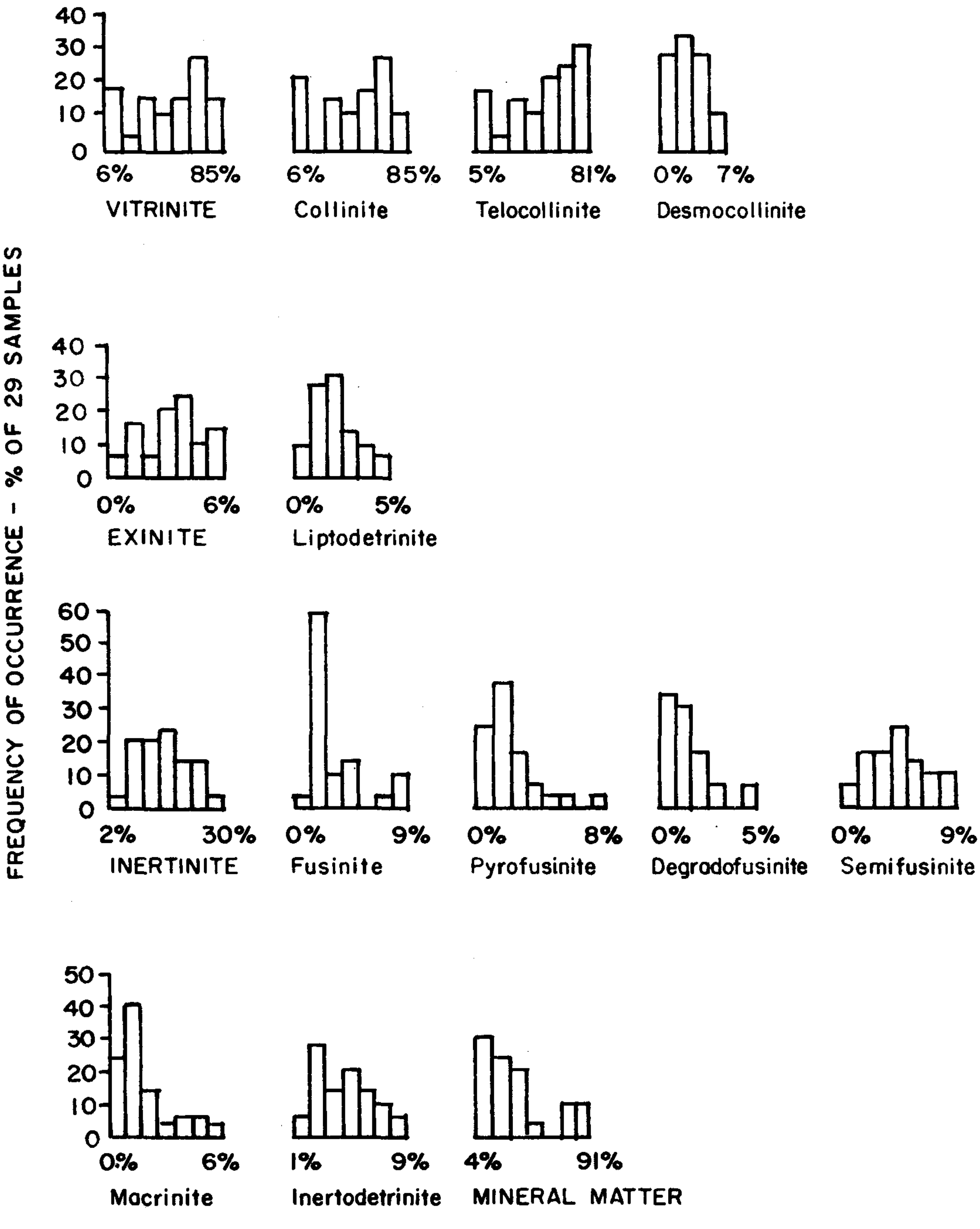
n = the number of values (samples).

For 29 samples the optimum number of classes was 6, but the distributions were constructed using 5 principal classes with one-half of a class interval added to each tail.

The range of the values of the variables was important in the interpretation of the petrography of these samples. Because the values are non-normally distributed, the mode, median, or mean values were inaccurate and even the range containing 90 percent or 95 percent of the sample values does not represent the distribution. Maximum and minimum values for each variable (maceral group, maceral, and submaceral) are presented in Table 6.

Distribution of the macerals within the float-sink fractions was as expected. Vitrinite macerals are present in greatest amounts in the clean coals (1.30 float), and minerals are concentrated in the 1.80 sink fractions. Correlation of the coal material (macerals) with minerals in the float-sink fractions will be possible when mineral values are available.

Fig.9 – MACERAL FREQUENCY DISTRIBUTIONS[a] OF THE PITTSBURGH COAL



[a] FREQUENCY DISTRIBUTIONS OF MACERAL GROUPS, MACERALS, AND SUBMACERALS OCCURRING IN THE PITTSBURGH COAL FLOAT-SINK FRACTIONS AND HEAD SAMPLES.

TABLE 6

MACERALS PRESENT IN THE PITTSBURGH DISTRICT 7 COAL SAMPLES IN AMOUNTS GREATER THAN 2 PERCENT BY VOLUME WITH THEIR MINIMUM AND MAXIMUM OBSERVED VALUES

GROUP MACERAL Maceral submaceral	Minimum	Maximum
VITRINITE	6% (48%)*	85% (89%)*
Telinite	0%	2%
Collinite	6%	85%
telocollinite	5%	81%
desmocollinite	0%	7%
EXINITE	0% (0%)	6% (11%)
Sporinite	0%	3%
Liptodetrinite	0%	5%
INERTINITE	2% (7%)	30% (48%)
Fusinite	0%	9%
pyrofusinite	0%	8%
degradofusinite	0%	5%
Semifusinite	0%	9%
degradosemifusinite	0%	9%
Macrinite	0%	6%
Micrinite	0%	3%
Inertodetrinite	1%	9%
MINERAL MATTER	4%	91%

* Values in parentheses are values of the maceral group recalculated on a mineral-matter-free basis.

Infrared Analysis - Spectra have been compiled this quarter for all size and gravity fractions of the Pittsburgh coal under investigation, and analysis by infrared spectroscopy revealed definite trends in mineral distribution with respect to both size and gravity fractions. The highest kaolinite concentrations were generally found in the 1.30 float fractions, while the lowest levels of the mineral occurred in the 1.80 float and 1.80 sink fractions. Kaolinite concentration vs. specific gravity can be ranked as follows: 1.30 float $>$1.40 float $>$1.60 float $>$1.80 float, 1.80 sink. In the 1.40 and 1.60 float fractions there was an obversed increase in kaolinite concentration with decreasing particle size, i.e., the highest concentrations were attained in the 28 x 100 mesh size fractions in both cases. Quartz concentrations were generally higher with higher specific gravities and its distribution can be ranked as follows: 1.80 sink $>$ 1.80 float $>$1.60 float $>$1.40 float $>$1.30 float. Thus a decreasing kaolinite concentration is accompanied by a corresponding increase in quartz concentration with increasing specific gravity.

IR scans of the Pittsburgh feed and clean coals (Figures 10 and 11) each showed two shallow bands at approximately 660 and 594 cm^{-1} which may possibly be indicative of a mixture of gypsum and gypsum-hemihydrite. The characteristic absorption bands for quartz (approximately 790 and 760 cm^{-1}) in the spectra of the Pittsburgh feed and clean coals were less intense than in the refuse (Figure 12), while the peaks at 660 and 594 cm^{-1} (gypsum-hemihydrite) were considerably more intense. Spectra of both the feed and clean coals in addition to several of the 1.30 and 1.40 float fractions

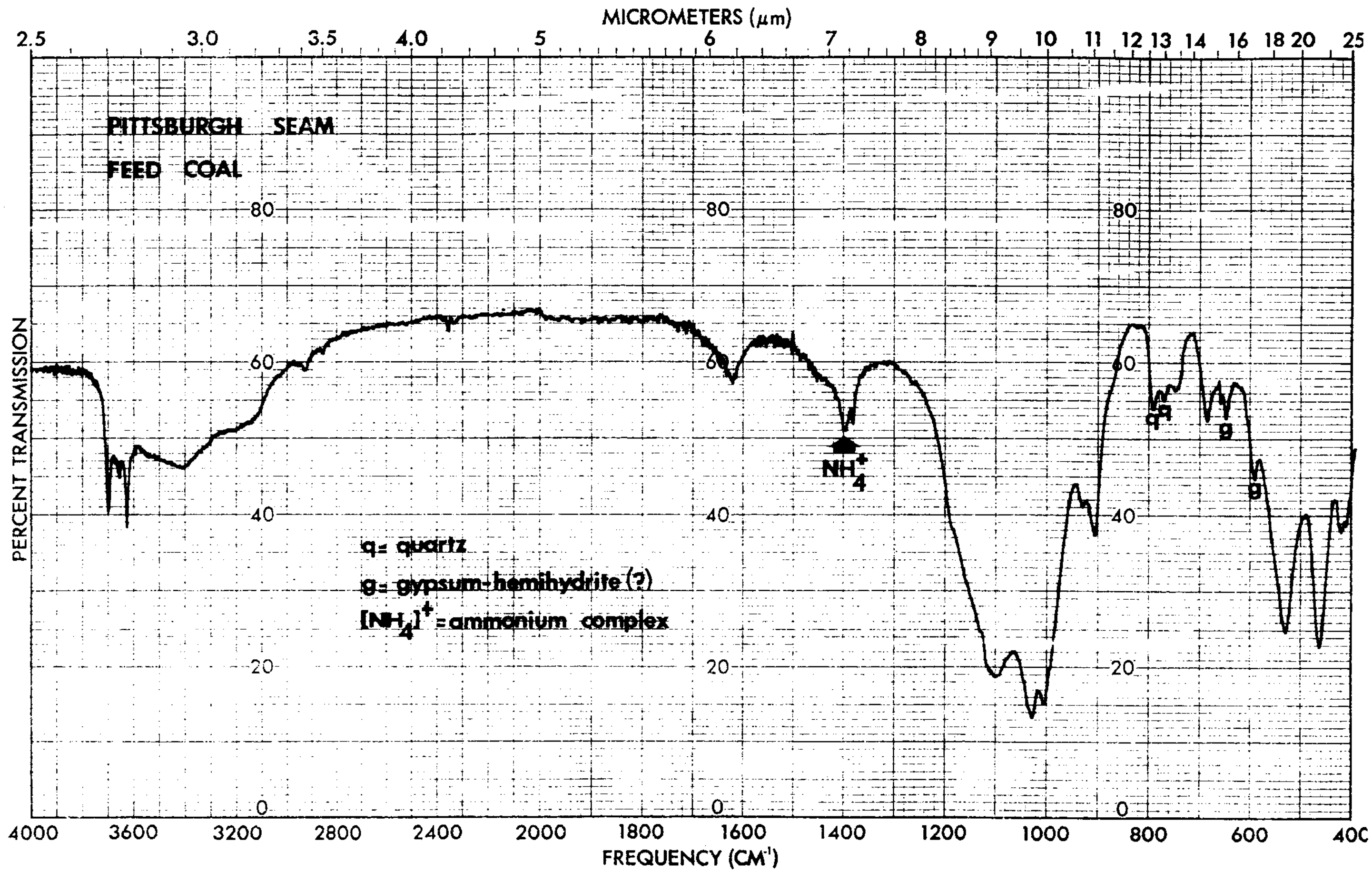


FIGURE 10

INFRARED SCAN OF PITTSBURGH SEAM FEED COAL HEAD SAMPLE

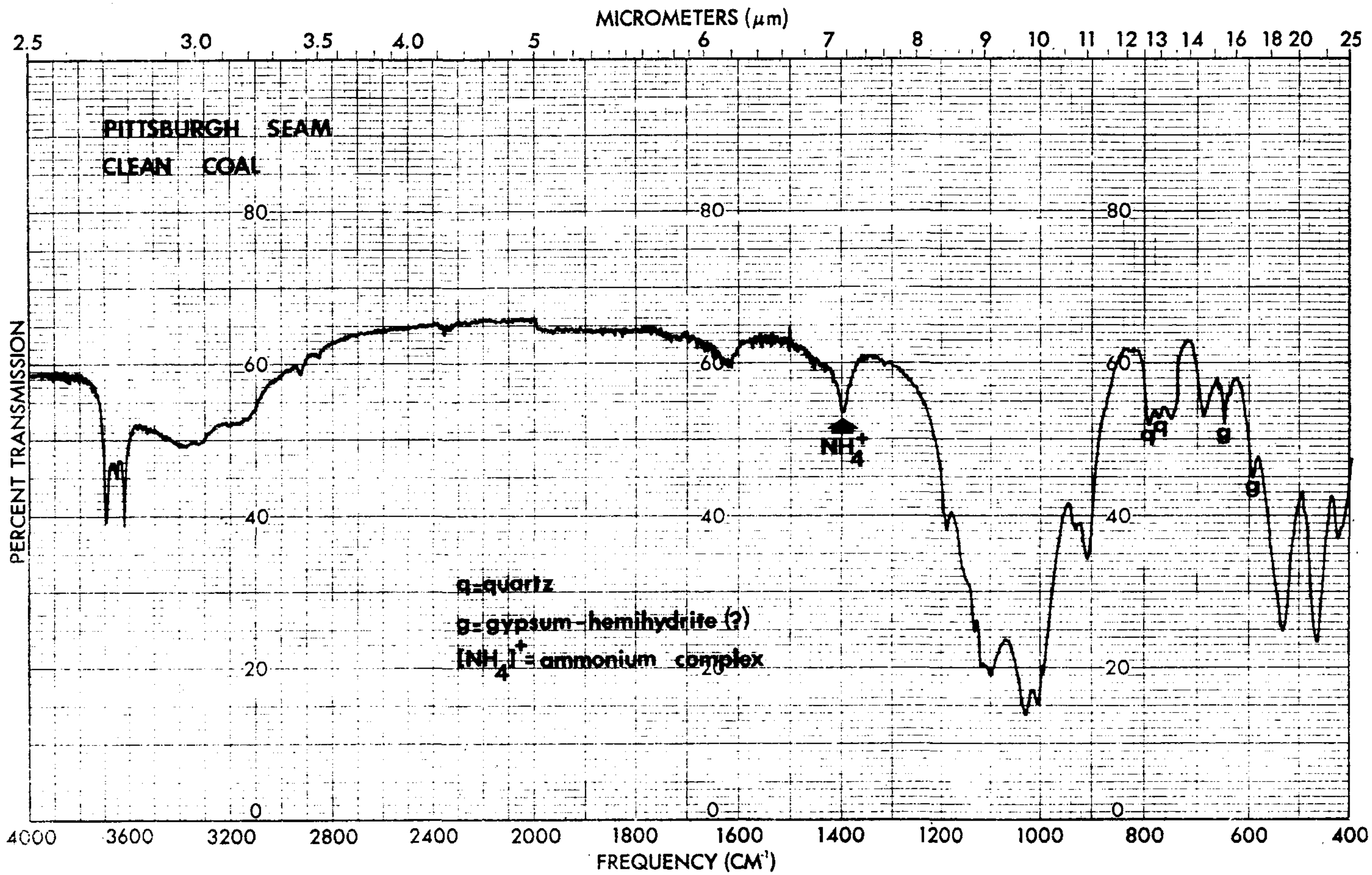


FIGURE 11

INFRARED SCAN OF PITTSBURGH SEAM CLEAN COAL HEAD SAMPLE

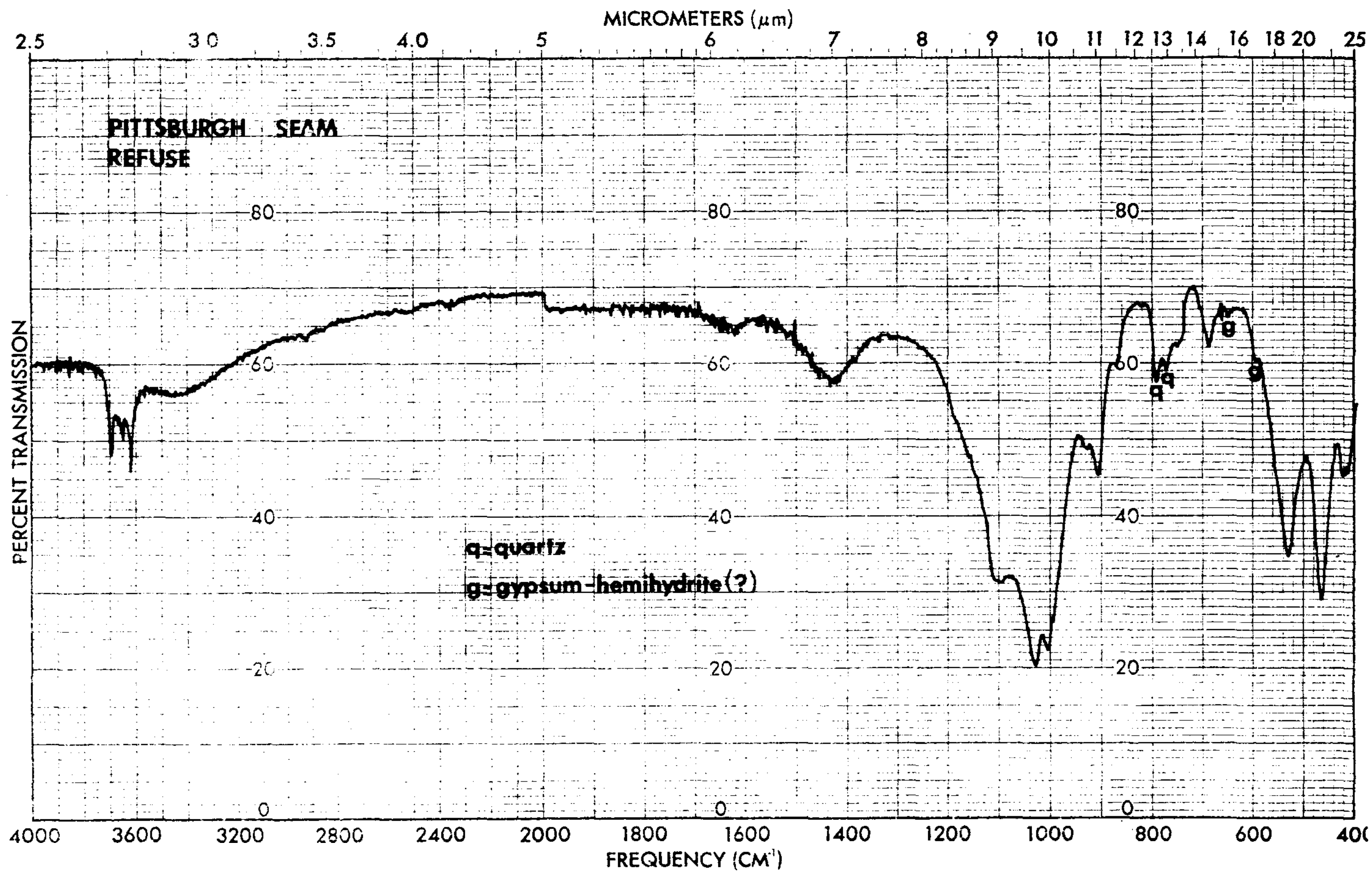


FIGURE 12

INFRARED SCAN OF PITTSBURGH SEAM REFUSE HEAD SAMPLE showed a sharp, well-defined band at 1400 cm^{-1} indicating the presence of the ammonium complex, $[NH_4]^+$, probably occurring as a reaction by-product resulting from nitrate formation during the low-temperature ashing process.[2]

Certain minerals were not detected by IR analysis in the coals studied which one would expect to occur in the LTA of bituminous coals, eg. illite, certain carbonates, and pyrite. Because of the high sensitivity of kaolinite to infrared, even at low concentrations, the strong absorption bands for this mineral had a tendency to mask the diffuse bands of both poorly ordered species (eg. illite) and the strong, well-defined peaks of carbonate minerals in the 900 to 700 cm^{-1} region (eg. calcite). High concentrations of illite, and carbonates to a lesser degree, were effectively masked by lower concentrations of kaolinite in the LTA of the Pittsburgh coals studied. The diagnostic bands for the identification of pyrite at 411 and 340 cm^{-1} were obscured in all of the spectra, possibly because of the inherent difficulties encountered in this spectral region with the use of the potassium bromide matrix. During the next quarter a recirculating air dryer and CO_2 absorption unit will be installed which will facilitate better resolution in the 600 to 200 cm^{-1} spectral region. Both cesium iodide and polyethylene matrix materials will be used to expand the useable spectral range below 600 cm^{-1}.

Certain minerals identified by petrographic methods were not detected by IR analysis because of their very low concentrations in the LTA material (eg. rutile and hematite).

X-ray Powder Diffraction (XRPD) - Facility modifications necessary for installing the Phillips APD-3501 have been completed (the installation of special 220 v. circuits and necessary plumbing). Other modifications necessary for proper operation of the unit are in progress and will be completed in the near future.

Investigations of various sample preparation techniques are continuing, and are being combined with a survey of the literature with respect to the most appropriate method of preparing low temperature ashed coal samples for analysis. Details of this work will be reported when available.

REFERENCES

1. Sturges, H. A., 1926, The Choice of Class Interval: Journal of Am. Stat. Assoc., March, 1926.

2. Kovach, J. J., Morgantown Energy Technology Center, Morgantown, West Virginia, private communication, November, 1978.

APPENDIX A

Financial Reports
and
Milestone Chart

THE EFFECTS OF MINERALS ON COAL BENEFICIATION PROCESSES

West Virginia University

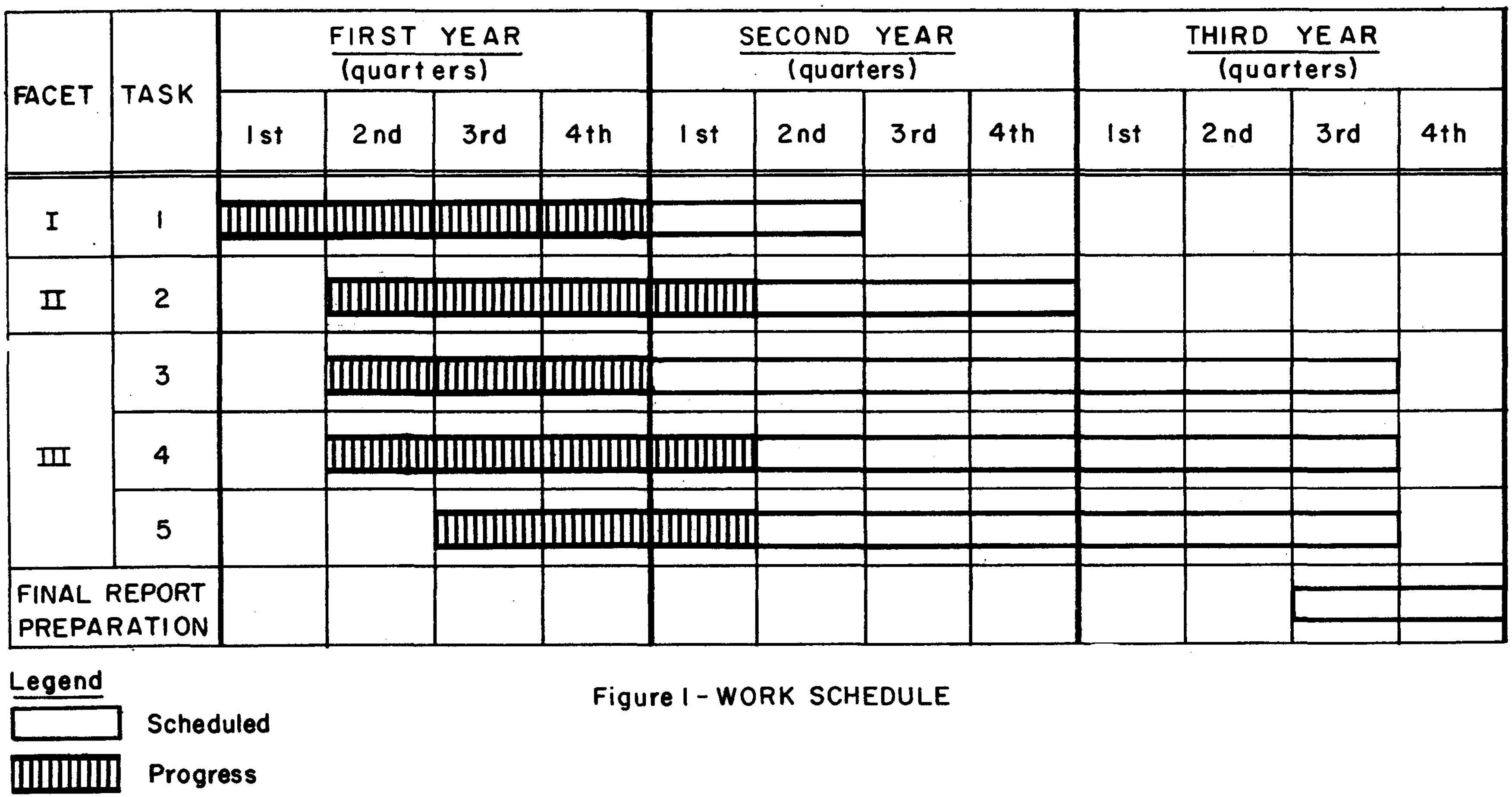


Figure I - WORK SCHEDULE

EFFECTS OF MINERALS ON COAL BENEFICIATION PROCESSES

FINANCIAL REPORT

5TH QUARTER, OCTOBER 1, 1978 - DECEMBER 31, 1978

Expenditures This Quarter	
Personal Services	$12,531.13
Equipment, R & A	201.61
Current Expense	
Overhead	7,064.60
Supplies	3,902.75
Travel	943.13
Printing	-----
Benefits	1,910.99
TOTAL EXPENDITURES 5TH QUARTER	$26,554.21
TOTAL EXPENDITURES TO DATE	152,671.34
TOTAL CONTRACT AWARD TO 9/30/79	280,000.00
CONTRACT BALANCE	$127,328.66

*U.S. GOVERNMENT PRINTING OFFICE : 1979 0-620-097/2298