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UTILIZATION OF COAL ASSOCIATED MINERALS

QUARTERLY REPORT No. 6

January 1 - March 31, 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

During the sixth quarter of work on the subject contract, laboratory analysis of physical and chemical properties has continued on representative samples of plant feed coal as well as washed coal and refuse from the Pittsburgh seam.

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 seam using IR, and a special die and pressing technique was developed for use with the Phillips^{*} APD-3501 X-ray diffractometer.

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

*

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

Characterization of Coal Samples

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 observed mineral matter was determined. Using IR, some mineral distribution trends were found for the head sample of the Pittsburgh coal. 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 3 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 1. 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 4 of Quarterly Report No. 5. Table 1, 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 1 if it exceeds the correlation coefficient of the member maceral to the group.

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 2), 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 1, shows a good agreement which is confirmed by the correlation coefficient (+0.99). Linear regression applied to the values (Table 3) plotted in Figure 1 resulted in the following equation for petrographic mineral matter (PMM):

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

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 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 ($4000 - 180 \text{ cm}^{-1}$) 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 the Pittsburgh seam 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 2). The refuse fraction (Figure 3) 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 2 through 4).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 adsorption 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 4). The sharp band at 1400 cm^{-1} in the spectrum of the Pittsburgh clean coal was the ammonium complex $(\text{NH}_4)^+$ (discussed in Quarterly Report No. 5, pp. 5-6).

Conclusions

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

All spectra of the Pittsburgh coals studied contained gypsum-hemihydrate. Carbonate concentration was highest in the Pittsburgh seam refuse fraction.

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 5). 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 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 6). Mounting in this manner allowed the pellet face to be flush with the sample holder surface.

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 Hidalgo and Renton, 1970)³ using LTA/methyl cellulose will be presented in future reports.

REFERENCES

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.

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 plus 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 Northern 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 17.38, 27.38, and 37.38 percent when using the Northern 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 lime 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 14-01-0001-488 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.

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

Table 1. 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)

	<i>Maceral or Maceral Group</i>	<i>Correlation Coefficient</i>
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 2. Correlations between specific gravity of the float-sink fraction and Maceral Groups, Macerals, and Mineral Matter. (n=29)

Table 3. 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
1X $\frac{1}{4}$ inch	1.30 float	7.2	6
1X $\frac{1}{4}$ inch	1.40 float	14.8	13
1X $\frac{1}{4}$ inch	1.60 float	28.7	26
1X $\frac{1}{4}$ inch	1.80 float	42.4	37
1X $\frac{1}{4}$ inch	1.80 sink	84.6	86
$\frac{1}{4}$ X8 mesh	1.30 float	7.1	4
$\frac{1}{4}$ X8 mesh	1.40 float	23.7	15
$\frac{1}{4}$ X8 mesh	1.60 float	27.7	21
$\frac{1}{4}$ X8 mesh	1.80 float	39.0	36
$\frac{1}{4}$ X8 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

Table 4

Chemical Analysis of Fluidized Bed Process
(Pope, Evans and Robbins) Waste Materials

	<u>Bed Ash</u>	<u>Flyash</u>
SiO ₂	22.40	31.20
Al ₂ O ₃	5.97	8.36
Fe ₂ O 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	0.63	0.64
S	8.63	3.87
LOI	4.4	36.0
C	2.99	32.18

Table 5
Composition and Properties of Northern West Virginia Flyash Brick
With Pope, Evans and Robbins Bed Ash

	1	2	3	4	5	6	7	8	9
Northern West Virginia Flyash	69.62	79.62	69.62	59.62	79.62	69.62	59.62	69.62	69.62
Bottom Slag - 20 Mesh	27.38	-	-	-	-	-	-	-	13.69
ER bed Ash - 20 Mesh	-	17.38	27.38	37.38	17.38	27.38	37.38	27.38	13.69
Illicate	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
Illicate Grade	47	47	47	47	47	47	47	47	47
Water	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0
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.	2190	2190	2190	2190	2190	2190	2190	2190	2190
None	6	6	6	6	6	6	6	6	6
Abs. 24 Hr. Soak	8.56	17.26	23.10	27.00	15.98	21.42	26.70	23.24	15.42
Abs. 5 Hr. Boil	12.41	21.99	28.22	32.58	21.29	26.76	31.76	28.76	20.53
Saturation Coefficient	0.68	0.78	0.81	0.82	0.74	0.79	0.83	0.80	0.74
Apparent Porosity, %	23.55	36.15	42.60	46.55	35.35	41.24	45.90	43.04	34.41
Bulk Density, Lbs./Cu. Ft.	118.3	102.6	94.2	89.2	103.6	96.1	90.1	93.4	104.5
Shrinkage	4.9	5.3	2.8	0.1	5.7	2.6	0.2	2.5	4.3
Unfired Compressive Strength, P.S.I.	984	82	25	63	60	49	99	89	49
Fired Compressive Strength, P.S.I.	7179	3526	1382	733	2801	1296	702	1204	2848
Conduction, gms. H ₂ O/30 Sq. In./Min.	104	172	257	334	156	242	303	248	164

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

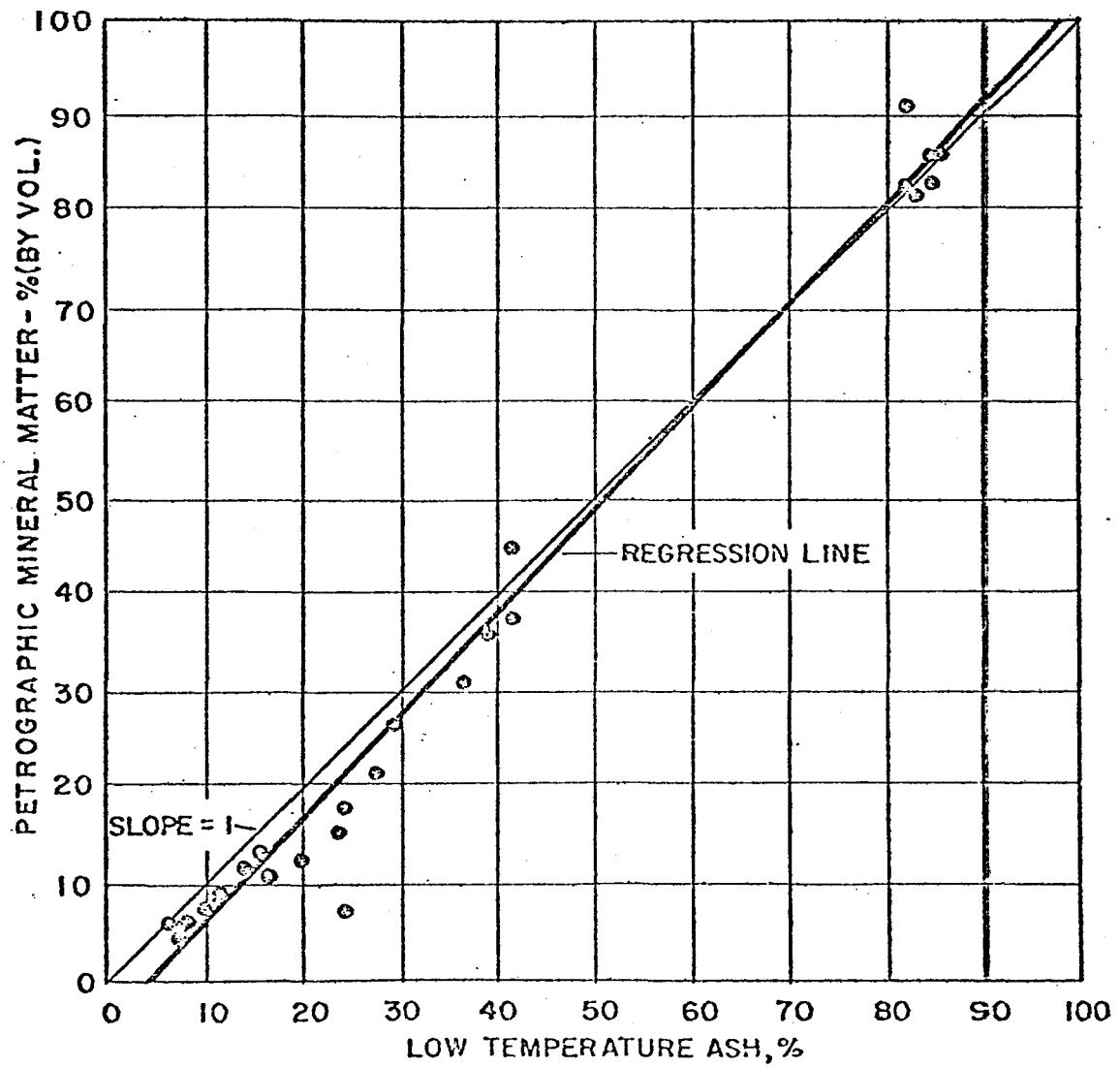


FIGURE 1

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

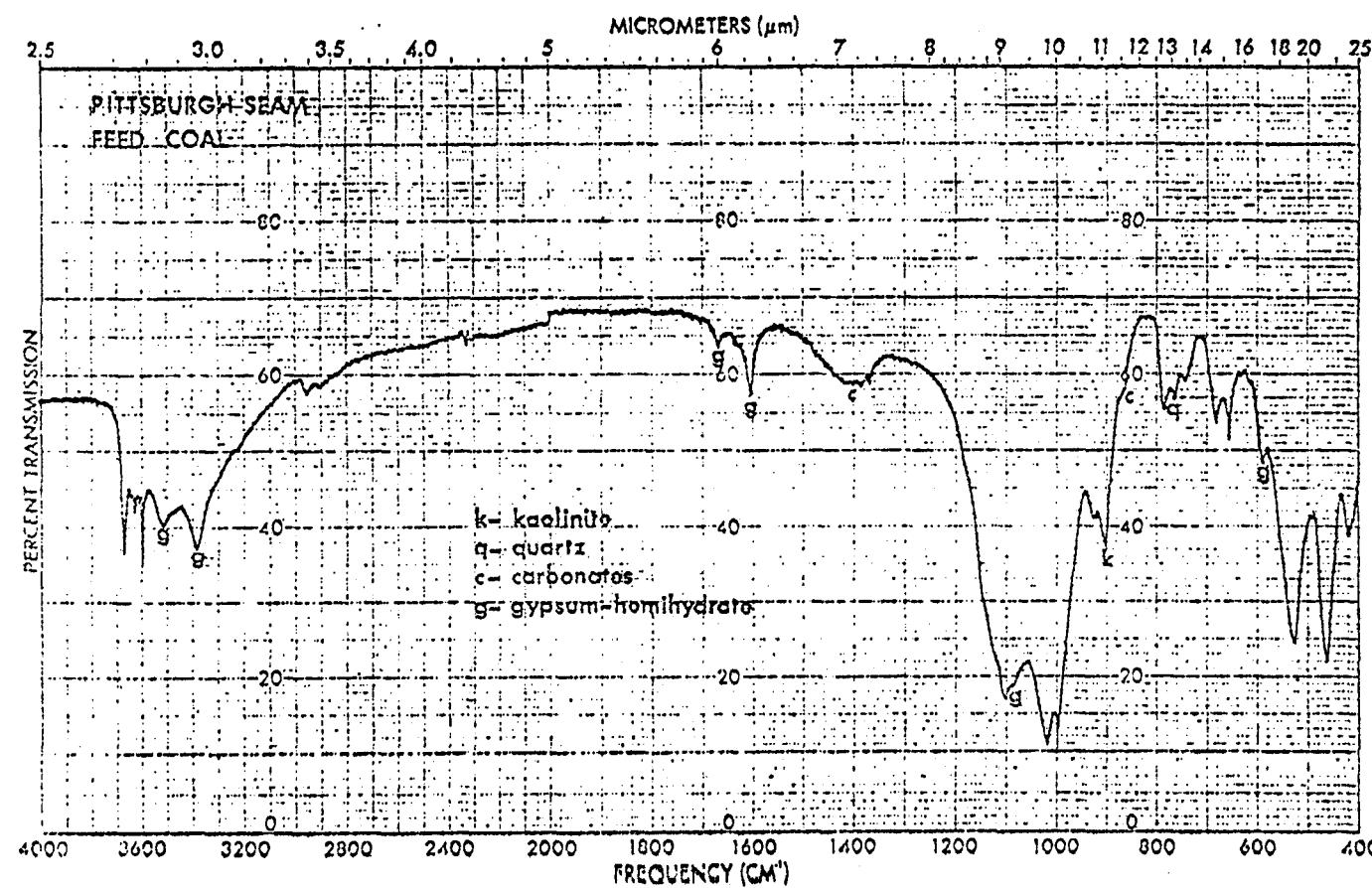


FIGURE 2.

INFRARED SCAN OF PITTSBURGH SEAM FEED COAL HEAD SAMPLE

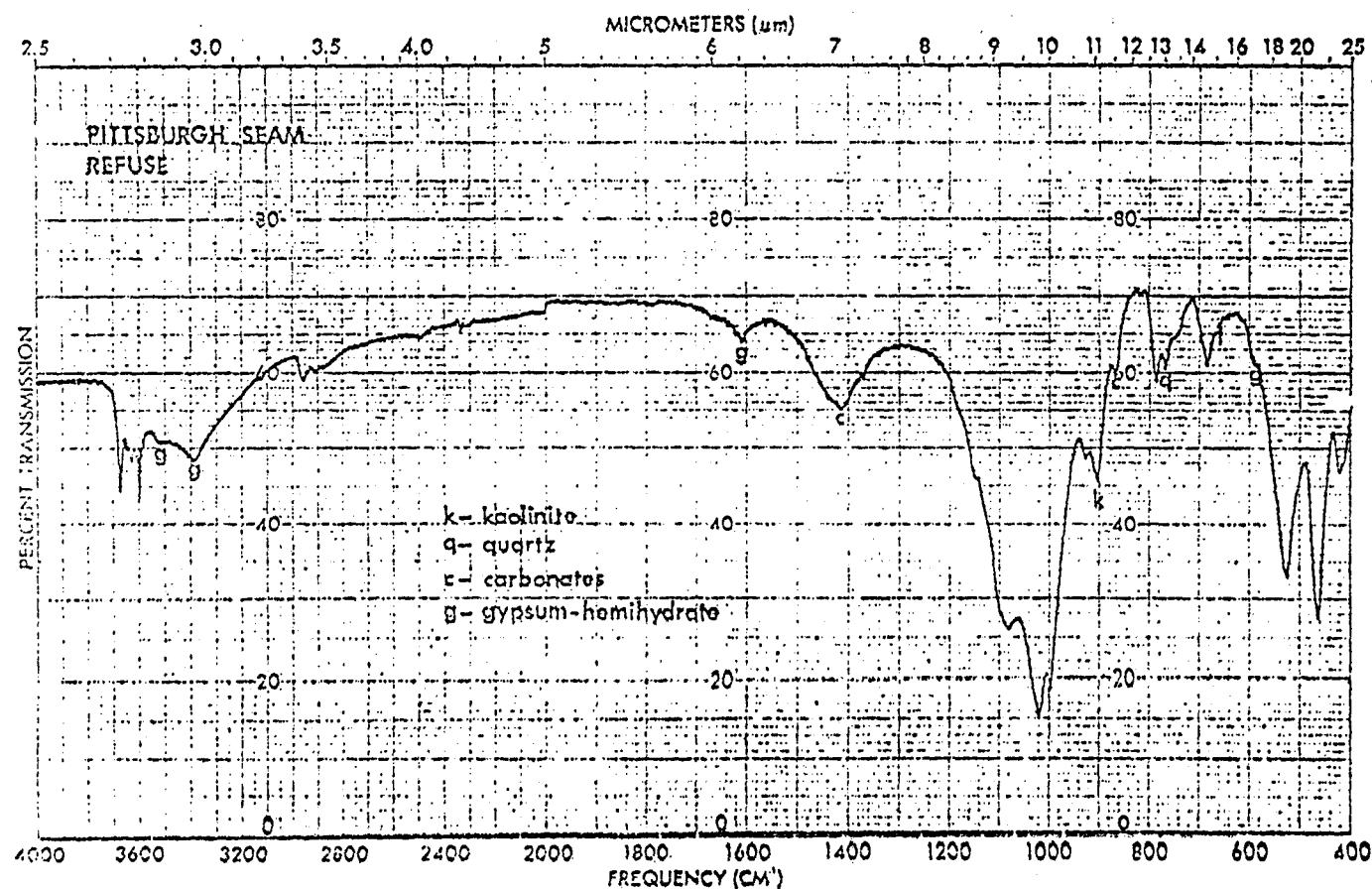


FIGURE 3

INFRARED SCAN OF PITTSBURGH SEAM REFUSE HEAD SAMPLE

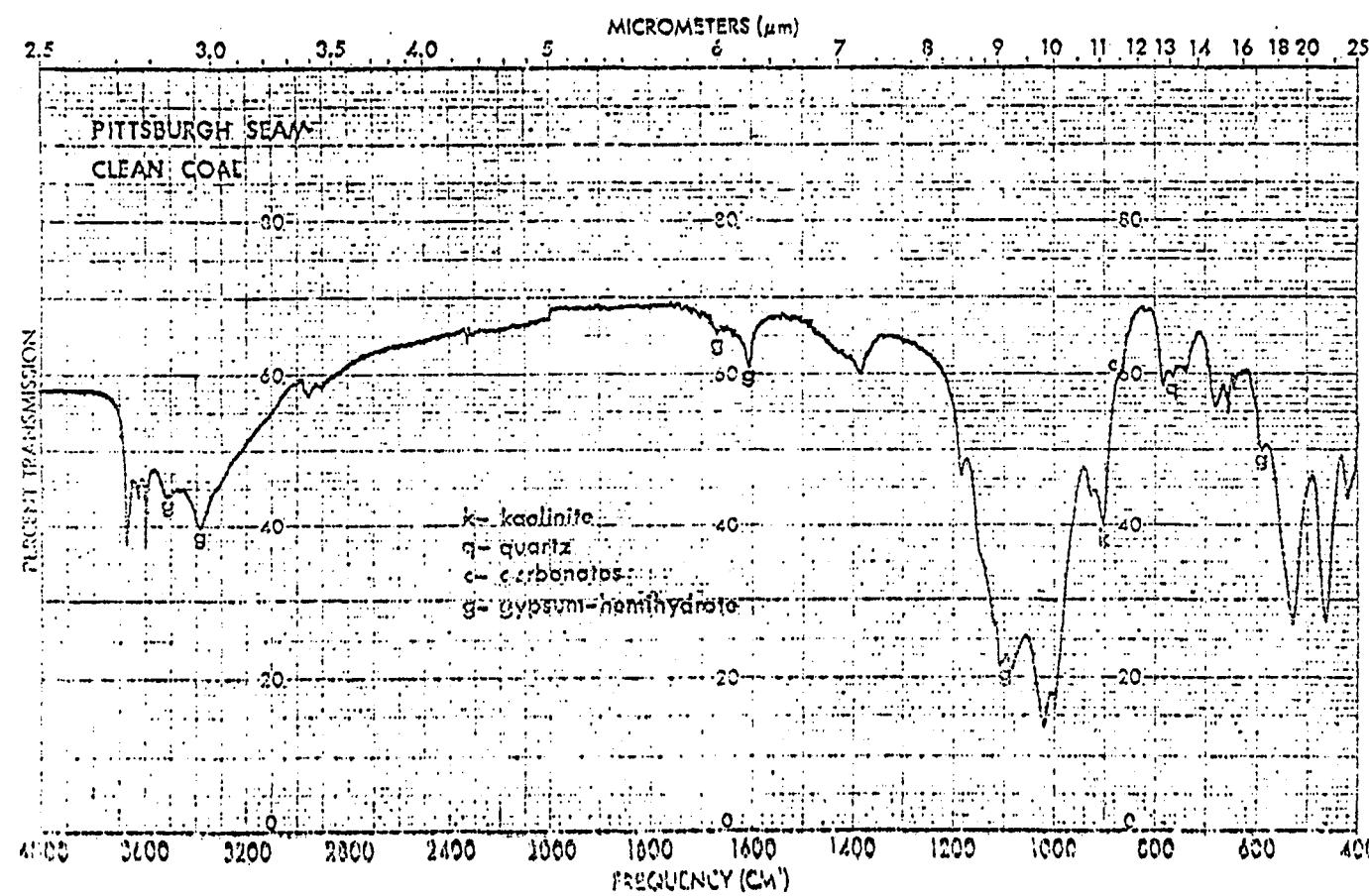


FIGURE 4

INFRARED SCAN OF PITTSBURGH SEAM CLEAN COAL HEAD SAMPLE

FIGURE 5

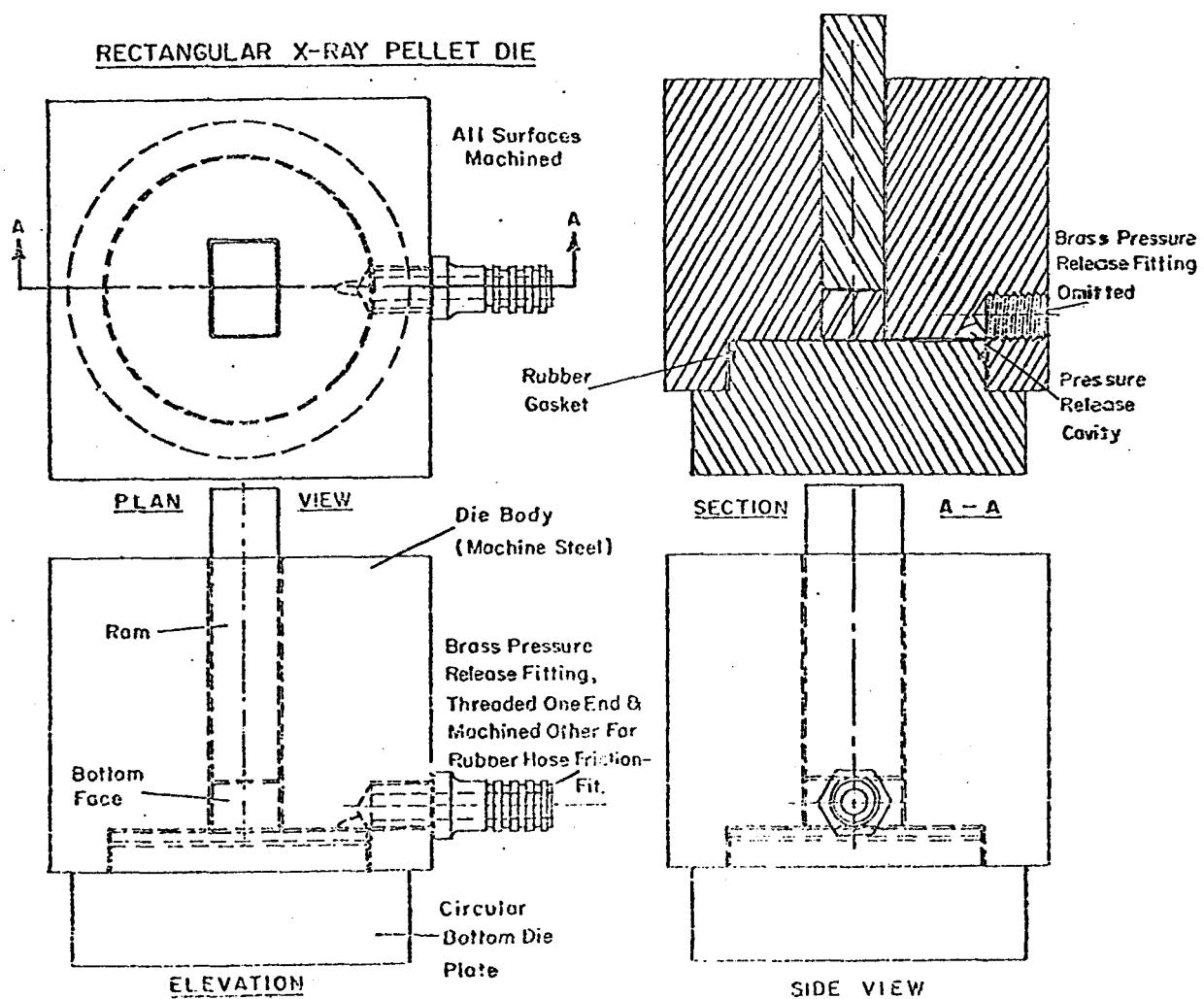
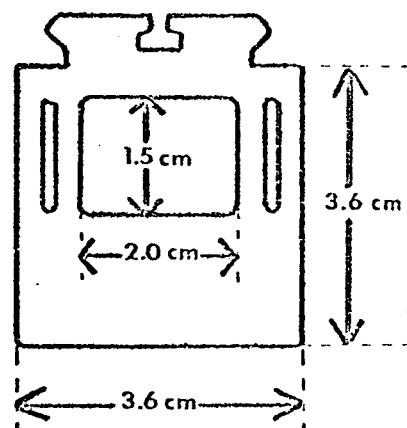


FIGURE 6



APD SAMPLE HOLDER

APPENDIX A

Financial Reports
and
Milestone Chart

UTILIZATION OF COAL ASSOCIATED MINERALS

West Virginia University

Facet	Task	FIRST YEAR (quarters)				SECOND YEAR (quarters)				THIRD YEAR (quarters)			
		1st	2nd	3rd	4th	1st	2nd	3rd	4th	1st	2nd	3rd	4th
I	1												
	2												
	3												
II	4												
	5												
	6												
	Final Report Preparation												

Work Schedule

UTILIZATION OF COAL ASSOCIATED MINERALS

FINANCIAL REPORT

6TH QUARTER, JANUARY 1, 1979 - MARCH 31, 1979

Expenditures This Quarter

Personal Services	\$15,967.26
Equipment, R & A	1,237.84
Current Expense	
Overhead	8,984.20
Supplies	10,682.50
Travel	653.97
Benefits	2,435.00
TOTAL EXPENDITURES 6TH QUARTER	39,960.77
TOTAL EXPENDITURES TO DATE	164,958.36
TOTAL CONTRACT AWARD TO 9/24/80	378,000.00
CONTRACT BALANCE	\$213,041.64