TECHNICAL REPORT

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September 1, 1991 through November 30, 1991

Project Title: GRAPHIC VALUES FOR SOME ORGANIC CONSTITUENTS
OF BENEFICIATED COAL SAMPLES

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ABSTRACT

Graphic techniques exist which can accurately predict values for Btu, organic sulfur, and possibly other constituents of the organic portion of beneficiated coal fractions. These techniques also permit a more accurate determination of coal rank to be made without the use of approximations.

In beneficiation studies, plots of the calorific value of each fraction vs. its ash content invariably produce straight lines. The Y axis intercept is the mineral matter free Btu value used (on the moist basis) to determine coal rank and the X axis intercept is a value we will call "maximum ash". These two constants permit us to calculate all future Btu values of fractions of this coal from the ash, alone. Plots of organic sulfur vs. ash sometimes produce lines which are straight and at other times curved. In any event, they can be used to read off values of organic sulfur corresponding to a particular ash value in lieu of analyses. The X axis intercept must occur at the same maximum ash value obtained from the Btu/ash curve. When the line is straight, the Y axis intercept is the mineral matter free organic sulfur, and all future organic sulfur values may be calculated from the 2 constants and the ash.

Fractions of IBC-101 with varying ash contents were produced by froth flotation techniques. The data were evaluated as produced in order to assure that some ash values greater than 50% are represented. The Btu/ash, organic sulfur/ash, and possibly a few others will be plotted. A comparison of the graphic mineral matter value with the usual one calculated from the Parr approximation will be made and the impact of any differences on coal rank will be noted. All data will be available to researchers doing future studies of IBC-101. We feel these techniques, which we use routinely in our mass balance calculations will be very useful in on-line or remote analysis situations.

MASTER

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EXECUTIVE SUMMARY

We will attempt to show in this project that there are techniques available which can accurately predict values for calorific value, organic sulfur, and possibly other constituents of the organic portion of beneficiated coal fractions without the necessity of laboriously analyzing each and every fraction. We contend that the most used method of determining coal rank may be less accurate than the method we propose, which avoids the need to use approximations. The approach we intend to explore would save much time and analytical expense, involve fewer uncertainties, and result in far greater turn-around times for investigators involved in coal beneficiation studies.

In such studies, plots of the calorific value of each fraction vs. its ash content invariably produce straight lines. Once this relationship has been established, no further Btu analyses need ever be run for the particular coal being studied. So long as no chemical changes take place, if the ash is known, then so is the calorific value.

In plots of organic sulfur vs. ash, results are sometimes the same, but not in all cases. Some plots produce curved lines. These curves may still be used to read values of organic sulfur corresponding to the particular ash value for all future beneficiation fractions of the same coal. This can save an enormous amount of laboratory analyses and produce useable data quickly for accuracy checks and mass balance calculations.

Other information can be obtained from the plots, as well. In the calorific value/ash plot, the Y axis intercept is equivalent to the mineral-matter-free Btu value. When calculated to the moist basis, this becomes a most important bit of data for determining the rank of the coal according to A.S.T.M. D 388. The presently accepted method for determining this value is to calculate it from the Parr approximation. This has some disadvantages connected with it, including the fact that certain incorrect assumptions about the coal must be made. The X axis intercept produces a value we may call the maximum ash value of that coal. In other words, this is the ash value which would be produced if a fraction of the coal containing only its mineral matter were to be burned. The mineral matter/ash ratio of this coal will always be equal to 100/max. ash.

In cases where the organic sulfur/ash plot is a straight line, the Y axis intercept is equivalent to the mineral matter free organic sulfur value. Regardless of whether straight or curved, however, it must intercept the X axis at the same maximum ash value determined in the previous plot for calorific value, and this is a precisely known point.

We have separated a portion of IBC-101 into fractions of differing ash values using froth flotation. Plots of calorific value/ash and organic sulfur/ash will be made using Lotus and Freelance programs on the

computer. The types of data previously discussed will be taken from the plots and evaluated in a manner to show the comparison between the mineral-matter-free Btu value a la Parr and that of the graphically determined value. Other plots such as carbon, hydrogen, and nitrogen vs. ash will be made on an experimental basis to see if any other useful relationships might exist. All values obtained in this manner for IBC-101 will be available to other researchers doing beneficiation work on the same coal, and will no doubt be a valuable addition to the data list for this Coal Bank sample.

The techniques we are proposing are presently being used in routine fashion in our beneficiation work in effecting mass balance calculations with rapid turn-around times, since we are able to skip many time consuming lab. analyses. They would be ideal for use in on-line or remote analysis situations. In spite of the fact that organic sulfur/ash plots are composed of widely scattered points, we feel that the values obtained are equal to or better than the lab. analyzed equivalents for the very reason that the ASTM method used to obtain them is so fraught with error.

OBJECTIVES

The first objective of this one-year project is to obtain analytical data on a series of fractions of coal sample IBC-101 of widely varying ash content obtained via a froth flotation physical coal cleaning process. Froth flotation is the fractionation technique to be used rather than float/sink testing as in the Stansfield-Sutherland method* because 1) most of the data in our files which were used in the development of these techniques were from froth flotation tests and 2) as a way of showing that the fractionation is as effective by one technique as the other, so long as no chemical changes are effected. Analytical values will be obtained in the Coal Analysis Laboratory for moisture, ash, volatile matter, fixed carbon, total carbon, hydrogen, nitrogen, oxygen, total sulfur, sulfate sulfur, organic sulfur, and calorific value.

The next objective will be to plot the various values of each of the analyzed species versus its corresponding ash values to obtain x/y plots for each as a function of ash. From the resulting curves, it should be possible to calculate for coal sample IBC-101 a precise measure of its mineral matter content, its dry or moist mineral-matter-free calorific value as used in determining the rank of the coal sample, calculate organic sulfur values corresponding to each ash value in cases where the relationship is linear, and possibly find other analyzed values which have a direct correlation with the mineral matter content of the coal.

*Stansfield, E. and Sutherland, J. W., Trans. Am. Inst. Mining Met. Engrs., 88, 614-26 (1930).

EXPERIMENTAL

Most of the work covered in this first quarterly report is confined to the first objective or to tasks 1, 2, and 3 in the proposal.

Task 1. Sample Grinding

Eight 1 lb. bags of IBC-101 coal were taken from the storage barrel. Four separate 20 minute rod mill grinds were made, consisting of 2 bags of the coal plus an equal weight of water. The finely pulverized coal was recovered, using as little water as possible and stored as a slurry in covered plastic buckets. The slurry was divided into sixteen individual samples using the wet sample splitter to assure uniformity. Each of these was about the proper size to provide a pulp density of approximately 5.5% in the Denver cell used in Task 2.

Task 2. Flotation Tests

It was decided to use #2 fuel oil, exclusively, as collector in these tests. The frothers used were 2-ethylhexanol (2EH), methyl iso-butyl carbinol (MIBC), and a polyglycol-based reagent (M150). In each case, one of the slurry samples prepared in Task 1 was transferred to the Denver cell and appropriate quantities of frother and collector were added. The cell is now equipped with a froth crowder which directs the froth to the front of the cell preventing entrapment of coal-bearing froth behind the impeller where it might escape recovery. The liquid level in the cell is automatically maintained via a pump-fed syphoning system. As the coal-bearing froth layer is skimmed from the top of the cell into large collection pans, additional water flows into the cell from the bottom. The recovery of the calorific value (and likewise the rejection of pyrite and other ash forming minerals) can be controlled by such factors as reagent selection, quantities of reagent used, and sample collection times. One of the sixteen slurry fractions used in these tests contained too large a volume of water, resulting in the overflowing of the cell and contamination of the froth layer. This sample was discarded. Thus, there were a total of only fifteen flotation tests used in these experiments.

Task 3. Samples Preparation and Analyses

After each test was performed, the various pans containing the coal/froth mixtures were labeled and their contents suction filtered through large Buechner funnels. The filter cakes were returned to the appropriate pans and dried overnight in a forced air drying oven at slightly above ambient temperature. When thoroughly dry, the individual samples were weighed, removed from the pans, and an empty weight of the pan obtained. The weighed samples were broken up and passed through a Holmes mill fitted with a screen designed to pass everything smaller than 60 mesh. Samples too small to be ground in this manner were hand ground with a steel mortar and pestle. A portion of each sample was

split into a plastic laboratory sample bottle, labeled, and submitted to the Coal Analysis Laboratory to be analyzed for those items listed in the Objectives section. When the report of the Coal Laboratory was received, the data for each component fraction of the various flotation runs was fed into our materials balance program after which it became possible to evaluate the performances of each reagent combination and collection time with respect to recovery and rejection figures.

RESULTS AND DISCUSSION

Tables 1 through 3 show the results of the 15 flotation tests we have run so far. Choosing a similar dosage of each of the three frothers (such as the 1.8 #/ton MIBC, 1.7 #/ton 2EH, and 1.9 #/ton M150 all at 2' recovery times shows the relative efficiency of three reagents at separating the mineral matter from the organic portion of the coal. At this level of frother (plus approximately equal amounts of collector), the M150 is an easy winner with 95% Btu recovery compared to 84% for 2EH and 60% for MIBC. In fact, M150 appears to work much faster, recovering most of the fuel value in the first minute or less. By lengthening the recovery times of the middle fractions in those runs using the more efficient frothers, it was possible to pull over more mineral matter and thus produce higher ash fractions than one would ordinarily expect. It appears that we will have a few fractions with ash values greater than 50%, which we feel is necessary in order to get reliable plots.

Table 1. Tests with MIBC

MIBC (#/Ton)	#2 Fuel Oil (#/Ton)	Fraction Taken	Ash Reject (%)	Pyrite Reject (%)	Btu Recover (%)	Weight Recover (%)
1.82	1.82	1' Conc.	21.8	22.6	38.7	36.9
1.82	1.82	2' Conc.	34.9	37.4	60.4	57.7
1.82	1.82	4' Conc.	45.1	49.8	75.1	71.9
1.82	1.82	4' Tail	54.9	50.2	24.9	28.1
2.75	2.75	.5' Conc.	49.8	57.9	77.7	74.6
2.75	2.75	1' Conc.	62.4	72.5	93.3	89.8
2.75	2.75	2' Conc.	66.5	76.7	95.9	92.6
2.75	2.75	2' Tail	33.5	23.3	4.1	7.4
2.75	2.75	1' Conc.	55.6	66.7	84.9	81.8
2.75	2.75	2' Conc.	66.4	76.8	94.2	91.2
2.75	2.75	2' Tail	33.6	23.2	5.8	8.8

Table 2. Tests with 2EH

2EH (#/Ton)	#2 Fuel Oil (#/Ton)	Fraction Taken	Ash Reject (%)	Pyrite Reject (%)	Btu Recover (%)	Weight Recover (%)
.73	1.82	1' Conc.	83.2	84.5	71.8	73.1
.73	1.82	1' Tail	16.8	15.5	28.2	26.9
.73	2.95	1' Conc.	84.7	84.1	74.9	75.9
.73	2.95	1' Tail	15.3	15.9	25.1	24.1
1.77	1.77	1' Conc.	46.2	53.8	69.2	66.5
1.77	1.77	2' Conc.	58.1	66.9	84.1	81.1
1.77	1.77	4' Conc.	62.2	70.6	96.8	83.9
1.77	1.77	4' Tail	37.8	29.4	13.2	16.1
2.21	2.21	1' Conc.	54.2	63.3	81.4	78.5
2.21	2.21	2' Conc.	62.0	71.5	91.0	87.9
2.21	2.21	2' Tail	38.0	28.5	9.0	12.1
2.68	2.68	1' Conc.	36.1	44.2	61.2	58.5
2.68	2.68	2' Conc.	61.6	73.9	92.8	89.4
2.68	2.68	4' Conc.	69.7	81.2	96.8	93.5
2.68	2.68	4' Tail	30.3	18.8	3.2	6.5
3.17	3.17	1' Conc.	41.1	46.4	62.8	60.5
3.17	3.17	2' Conc.	52.4	59.9	78.1	55.4
3.17	3.17	4' Conc.	56.5	63.9	81.2	58.6
3.17	3.17	4' Tail	43.5	36.1	18.8	21.4
3.65	3.65	1' Conc.	45.0	47.2	71.2	68.2
3.65	3.65	2' Conc.	67.1	72.0	96.2	92.9
3.65	3.65	4' Conc.	76.3	81.4	99.0	96.4
3.65	3.65	4' Tail	23.7	18.6	1.0	3.6

Table 3. Tests with M150

M150 (#/ton)	#2 Fuel Oil (#/ton)	Fraction Taken	Ash Reject (%)	Pyrite Reject (%)	Btu Recover (%)	Weight Recover (%)
.98	1.96	.5' Conc.	40.5	48.6	64.2	61.5
.98	1.96	1' Conc.	45.1	54.0	71.3	68.4
.98	1.96	2' Conc.	51.2	59.8	77.8	74.8
.98	1.96	2' Tail	48.8	40.2	22.2	25.2
1.92	1.92	.5' Conc.	59.9	69.3	86.1	83.1
1.92	1.92	2' Conc.	71.2	80.3	95.3	92.5
1.92	1.92	2' Tail	28.8	19.7	4.7	7.5
2.58	2.58	1' Conc.	69.5	80.5	95.4	92.5
2.58	2.58	2' Conc.	74.4	85.4	98.3	95.6
2.58	2.58	2' Tail	25.6	14.6	1.7	4.4
2.90	1.93	.5' Conc.	61.4	70.8	84.9	82.2
2.90	1.93	1' Conc.	73.9	83.5	96.1	93.6
2.90	2.90	1' Tail	25.1	16.5	3.9	6.4
3.57	3.57	1' Conc.	76.5	86.2	95.6	93.3
3.57	3.57	2' Conc.	83.3	91.7	98.9	97.0
3.57	3.57	2' Tail	16.7	8.3	1.1	3.0

SUMMARY AND CONCLUSIONS

Now that all the samples have been prepared, flotation tests run, and sample analyses performed, we are ready to begin making x/y plots of the various parameters as functions of the mineral matter content. The next quarterly report will cover at least some of the early work in this area, which is listed as Task 4 in the proposal.

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COMMENTS

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