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**X-RAY DIFFRACTION AND SCATTERING STUDIES OF
COAL CONSTITUENTS**

Final Technical Report

By
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March 15, 1984

Work Performed Under Contract No. FG22-80PC30225

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Morgantown, West Virginia

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Final Technical Report

Department of Physics
West Virginia University
March 15, 1984

Title: X-Ray Diffraction and Scattering Studies of Coal Constituents

Grant No. DE-FG22-80PC30225

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Dr. John J. Renton

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I. Introduction

The general objective of this work has been to use x-ray diffraction and scattering to examine whole coals, coal macerals and minerals in order to perform the following studies:

- 1) to identify and explain differences in vitrinites and framboids from various coals.
- 2) to correlate differences with basic coal compositions and properties.
- 3) to determine the systematic variability in the micro compositional variation of macerals.

During the past year, extensive effort has been made to improve the correlation between Fourier transform x-ray characteristics and basic coal properties (proximate and ultimate analyses). These studies have been extended by obtaining more data, by enlarging the range of the Fourier transform and by the application of a multiple regression analysis. Considerable improvement has been made by this work on the samples available. Yet, there are still some problems in this technique. Also, it would be of value to be able to relate the Fourier transform techniques to more fundamental information than is given by proximate and ultimate analyses. Specifically, correlations with aromaticity and free radical content would be desirable. However, it has been impossible to obtain coal samples and coal macerals for which such information is available.

The survey of the types of x-ray diffraction patterns obtained from various maceral types in whole coals using the micro camera has continued. It should be recalled that these patterns are obtained from thin (1mm) slabs of whole coals. The variability of these patterns continues to be evident within a maceral group; however it is difficult to distinguish one

type of a maceral from another in the same group. Attempts have been made to identify the particular maceral subgroups and their structures but this has been difficult.

The small angle x-ray scattering (SASX) camera is finally taking shape. Dr. Richard Matyi of the Texas Instruments Company has provided us with some high quality silicon crystals. These have been channel cut and etched. They are functioning very close to what had been expected. In the mean time, the Apple Computer arrived and has been interfaced with the camera to facilitate the acquisition of data.

II. Fourier Transform X-Ray (FTXR) Diffraction Studies

The main effort there has been to improve the computer programs used to extract the initial data from the x-ray diffractometer traces, to increase the amount of data, to extend the range of the Fourier transform calculations and to obtain correlations between FTXR characteristics and coal properties using a multiple regression analysis. It should be recalled that in the last semi-annual report, some good correlations were obtained for unusual quantities like $\frac{P_2 \times A_2}{A_1}$ versus H/C, (H+O+S)/C % volatile matter and % carbon (P_i and A_i are the position of and area under the i th peak.) However, this was found by a purely empirical method which required some human intuitiveness. This is a highly undesirable method. For this reason the multiple regression approach was taken.

The previous approach to the problem was to obtain 70 data points from $2\theta = 4^\circ$ to $2\theta = 39^\circ$ (θ = the Bragg angle). This data was processed to obtain the intensity versus $S = 4\pi \sin \theta / \lambda$ (Polarization and background corrections as well as removal of the mineral peaks. From this data, the

Fourier Transform was calculated for a range of $r = 0$ to $r = 10\text{\AA}$ where r is the radial distance from a central atom. This gives a psuedo-radial function designated as $\text{Rho}(r)$. Such a $\text{Rho}(r)$ versus r yields a spectrum of up to 4 peaks which can be characterized by their positions, P_i , peak heights, H_i , and the area under the peak, A_i . A typical $\text{Rho}(r)$ versus r spectrum is presented in Fig. 7. Correlations between these FTXR parameters and coal properties (% Ash, % volatile matter, % fixed carbon, BTU/pound, Rank, % minerals, % vitrinite and % non-vitrinite) were attempted using a linear regression analysis. The good correlations were then used to determine coal properties of unknown coals.

This technique managed to give good results ($<\pm 5\%$ of the conventional results) for some coal properties: mainly % volatile matter, % fixed carbon, BTU per pound and Rank. But for other coal properties (% minerals, % ash, % vitrinites, and % non-vitrinites, poor results were obtained. In search for better results, two different approaches were taken:

- 1) The past method which used initial data from $2\theta = 4^\circ$ to 39° giving 70 data points was employed but the range of the psuedo-radial function, $\text{Rho}(r)$ was extended from $r = 10\text{\AA}$ to $r = 20\text{\AA}$. The FTXR parameters (P_i, H_i, A_i) of the $\text{Rho}(r)$ spectrum were used to make a linear and multiple regression analysis with coal properties. These correlations were then employed to determine the properties of unknown coals from their FTXR parameters.
- 2) The range of the initial data taken was increased from $2\theta = 4^\circ - 39^\circ$ to $2\theta = 4^\circ - 60^\circ$, giving a total of 112 data points. This increased data base is then used to calculate $\text{Rho}(r)$ versus r from $r = 0$ to $r = 20\text{\AA}$. The $\text{Rho}(r)$ versus r curves were calculated for a set of coals whose properties were determined by conventional methods. The FTXR parameters (P_i, A_i, H_i) were then used in a multi-regression analysis to obtain correlations between FTXR parameters and coal properties.

In each case, the criterion used to decide whether a correlation was good or not, was the reliability factor and the percent error in comparison with the conventional results.

Fig. 1. The pseudo-radial distribution function $\text{Rho}(r)$ versus r for 70 data points and $r = 0$ to $r = 10$ A.

In order to test the validity of these methods, two sets of samples were utilized:

- a) The first set employed to obtain the correlations was a suite of 28 samples from the Penn State Coal bank. Proximate and ultimate analyses were available for all samples.
- b) The second set, employed to test the correlations of the first set, consisted of 25 samples of coals: 14 samples of Chinese coals, 4 samples of Wyoming coals and 7 samples of Cannel Coals. The proximate analyses was known for all coals in this set but ultimate analyses was known for only 13 coals.

X-Ray diffraction traces were obtained in arbitrary intensity units from $2\theta = 40$ to 90° for both sets of samples. These data were corrected for polarization, background and for elimination of mineral peaks. The Fourier transform for each coal was calculated using 70 and 112 data points over a radial range $r = 0$ to $r = 20 \text{ \AA}$.

The 70 data points were used to calculate the Fourier transform or $\text{Rho}(r)$. A typical curve of $\text{Rho}(r)$ versus r is exhibited in fig. 2. The peaks in figure 2 are numbered 1, 2, 4, 5, 6, 7, and 8. Peak 3 has been omitted because some coals did not have a peak 3. The positions of these peaks did not vary much from one coal to another except for peak 2. However, the areas under the peaks and the peak heights did exhibit appreciable variation so they were considered in the analyses. Therefore in considering the 70 data point results, the Fourier Transform Parameters used in the analyses were $A_1, A_2, A_4, A_5, A_6, A_7, A_8, H_1, H_2, H_4, H_5, H_6, H_8$, and P_2 .

A typical Fourier transform versus r for the 112 data points is shown in fig. 3. In most of the coals twelve (12) peaks appeared. Peak 9 only appeared for some of the coals so it was ignored in the analysis.

Fig. 2. The pseudo-radial distribution function $Rho(r)$ versus r for 70 data points and $r = 0$ to 20 Å.

Fig. 3. The pseudo-radial distribution function $\text{Rho}(r)$ versus r for 112 data points and $r = 0$ to $r = 20$ Å.

Similarly peak 11 was ignored because for some coals, it had a negative height. Examination of the positions, heights and areas of the remaining peaks revealed that the peak positions and peak heights did not vary appreciably from coal to coal, whereas the peak areas did vary. As a result only the areas of peaks 1, 2, 3, 4, 5, 6, 7, 8, 20, and 12, were used for the analyses.

The method employed in this analysis is best summarized in the following steps:

- a) For each coal property, linear regression analysis was made with each of the FTXR characteristics. This resulted in reliability factors and the linear regression coefficients for both the 70 data point set and the 112 data point set.
- b) For each coal property, five (5) FTXR parameters with the highest reliability factors were selected for the multiple regression analysis. The reliability factors for each coal property are tabulated in tables 1 and 2. The five FTXR parameters selected are indicated by an asterisk. It must be pointed out that for some of the coal properties the reliability factors as a whole are quite low; however the highest of these are selected. A good example of a coal property with low reliability factors is wt% nitrogen.
- c) In executing the multiple regression analysis, every possible combination of 2, 3, 4 or 5 FTXR parameters were tried. For each combination of FTXR parameters, coal properties were computed for the second set of coal samples from the FTXR parameters obtained from the data for the second set of samples, along with the percent error and the average percent error as compared with the conventionally determined coal properties.
- d) All combinations which gave rise to negative coal properties calculated from the FTXR parameters were eliminated. For each coal property, only one combination of FTXR parameters was selected (both for the 70 and 112 data point sets); that one which possessed the highest reliability factor and the lowest average percent error. Those combinations which were selected are presented in tables 3 and 4. There is one exception to the above procedure. No combination would yield reasonable reliability factors and average percent errors for wt% ash for both sets of data. Other combinations were tried until a "best" combination was obtained.
- e) These best combinations were used to calculate the coal property values. Tables 5 to 8 have tabulations of these values for both sets of data. Finally, these coal property values were normalized to 100%.

Table 1. The Reliability Factors for Linear Regression Analysis of Coal Properties versus Fourier Transform Parameters using 112 Data Points

Fourier Transform Parameter	wt% Fixed Carbon	wt% Volatile Matter	wt% Ash	wt% Carbon	wt% Hydrogen	wt% Nitrogen	wt% Oxygen & Sulfur
A ₁	0.29	0.05	0.63*	0.51	0.00	0.06*	0.08
A ₂	0.16	0.01	0.45*	0.31	0.01	0.04*	0.03
A ₃	0.86*	0.70*	0.23	0.16	0.34*	0.11*	0.18*
A ₄	0.20	0.04	0.35*	0.33	0.00	0.07	0.07*
A ₅	0.06	0.07	0.00	0.03	0.00	0.00	0.07
A ₆	0.68*	0.42*	0.36	0.69	0.18	0.04	0.40*
A ₇	0.00	0.01	0.06	0.01	0.07	0.01	0.01
A ₈	0.66*	0.35*	0.46*	0.70*	0.12*	0.09*	0.33*
A ₁₀	0.76*	0.58*	0.24	0.64*	0.21*	0.02	0.51*
A ₁₂	0.58*	0.37*	0.25	0.51*	0.15*	0.03	0.31*

* The Fourier Transform parameters used in multiple regression analyses

Table 2. The Reliability Factors for Linear Regression Analyses of Coal Properties versus Fourier Transform Parameters using 70 data points.

Fourier Transform Parameter	wt% Fixed Carbon	wt% Volatile Matter	wt% Ash	wt% Carbon	wt% Hydrogen	wt% Nitrogen	wt% Oxygen & Sulfur
A ₁	0.38	0.10	0.64*	0.63*	0.00	0.09*	0.16
A ₂	0.66*	0.62*	0.10	0.41	0.45*	0.00	0.36
A ₄	0.80*	0.54*	0.36*	0.11	0.24	0.06*	0.14
A ₅	0.51	0.35	0.21	0.40	0.23	0.01	0.20
A ₆	0.57	0.72*	0.00	0.31	0.41*	0.01	0.64*
A ₇	0.72*	0.49	0.31	0.65*	0.18	0.03	0.41*
A ₈	0.47	0.36	0.15	0.34	0.16	0.00	0.22
H ₁	0.35	0.08	0.63*	0.58*	0.00	0.08*	0.13
H ₂	0.48	0.36	0.16	0.32	0.29*	0.01	0.14
H ₄	0.75*	0.48	0.39*	0.72	0.20	0.03*	0.39*
H ₅	0.41	0.24	0.25	0.35	0.15	0.01	0.13
H ₆	0.48	0.67*	0.00	0.22	0.40*	0.00	0.58*
H ₇	0.63	0.37	0.37*	0.09	0.12	0.07*	0.07
H ₈	0.42	0.28	0.20	0.35	0.12	0.00	0.16
P ₂	0.72*	0.75*	0.06*	0.53	0.34*	0.01	0.76*

* The Fourier Transform parameters used in multiple regression analyses

Table 3. The best combinations of Fourier Transform parameters, reliability factors, and average percent errors for the 112 data point set.

Coal Property	Fourier Transform Parameters	Reliability Factor	Average Percent Error	Number of Samples in Comparison
Wt% Fixed Carbon	A_3, A_6 A_8, A_{12}	0.88	8.4	25
Wt% Volatile Matter	A_1, A_6	0.62	25.4	25
Wt% Ash	A_1, A_5	0.65	26.2	25
Wt% Carbon	A_1, A_6 A_8, A_{12}	0.80	2.9	13
Wt% Hydrogen	A_6, A_8 A_{10}	0.22	10.8	13
Wt% Nitrogen	A_2, A_8	0.09	23.3	13
Wt% Oxygen & Sulfur	A_6, A_{10} A_{12}	0.52	34.3	13

Table 4. The best combinations of Fourier transform parameters, reliability factors and average percent errors for the 70 data point set.

Coal Property	Fourier Transform Parameters	Reliability Factor	Average Percent Error	Number of Samples in Comparison
Wt% Fixed Carbon	A_2, P_2	0.78	8.9	25
Wt% Volatile Matter	A_6, H_6 P_2	0.80	31.3	25
Wt% Ash	A_1, A_8	0.66	29.6	25
Wt% Carbon	A_7, H_1 H_4, P_2	0.82	2.66	13
Wt% Hydrogen	H_2, H_6	0.54	9.50	13
Wt% Nitrogen	A_1, H_1	0.17	26.8	13
Wt% Oxygen & Sulfur	A_7, H_6	0.69	17.9	13

Table 5. Wt% fixed-carbon, wt% volatile matter and wt% ash calculated without normalization for the 112 data point set.

Coal Sample Name	Wt% Fixed-Carbon	Wt% Volatile Matter	Wt% Ash
Chinese T1J	42.1	39.9	10.0
Coals T2J	50.7	38.6	4.1
T3J	78.4	40.6	0.94
T4J	61.2	33.0	5.1
T5J	57.4	31.3	8.1
T6J	70.3	20.6	6.7
T8J	73.0	13.4	9.4
T9J	98.5	18.3	6.4
T10J	87.4	9.7	1.5
T4Y	51.2	27.7	14.0
T5Y	47.4	23.7	16.7
T6Y	49.6	17.2	16.0
T8Y	59.0	10.8	17.4
T10Y	79.0	9.9	10.0
Cannel 9639	61.1	25.8	7.6
Coals 9640	43.8	28.1	12.2
9641	67.6	11.7	16.3
9642	38.3	35.5	13.5
9644	34.6	28.9	13.7
9645	48.0	41.4	5.6
9646	41.6	33.9	8.0
Wyoming 9654	49.2	47.1	3.6
Coals 9655	44.13	29.0	10.1
9656	44.4	36.2	10.8
9659	51.2	42.2	3.4

Table 6. Wt% Carbon, Wt% Hydrogen, Wt% Nitrogen, Wt% Oxygen and Sulfur and Wt% Ash calculated without normalization for the 112 data point set.

Coal Sample Name		Wt% Carbon	Wt% Hydrogen	Wt% Nitrogen	Wt% Oxygen & Sulfur	Wt% Ash
Chinese Coals	T1J	68.4	4.5	1.2	11.0	10.0
	T2J	73.7	4.0	1.2	7.2	4.1
	T3J	84.3	3.9	1	4.4	1.4
	T4J	76.8	4.2	1	8.0	1.3
	T5J	77.8	4.0	1.3	5.4	8.1
	T6J	86.0	4.1	1.4	5.5	6.7
	T8J	86.6	3.6	1.4	2.4	9.4
	T9J	86.4	3.7	1.5	2.6	6.4
	T10J	89.2	3.5	1.4	1.9	1.5
Wyoming Coals	9654	76.0	4.4	1.4	8.2	3.6
	9655	68.7	3.9	1.1	7.3	10.1
	9656	71.5	4.5	1.2	10.4	10.8
	9659	75.1	4.4	1.3	9.2	3.4

Table 7. Wt% fixed carbon, Wt% volatile matter and Wt% ash calculated without normalization for the 70 data point set.

Coal Sample Name		Wt% Fixed-Carbon	Wt% Volatile Matter	Wt% Ash
Chinese Coals	T1J	43.3	52.2	11.5
	T2J	46.5	47.0	4.0
	T3J	61.4	30.9	1.3
	T4J	57.5	37.2	4.1
	T5J	56.4	27.6	8.4
	T6J	64.6	20.4	6.2
	T8J	77.7	13.3	10.2
	T9J	85.8	12.0	4.3
	T10J	87.1	4.6	3.6
	T4Y	51.0	35.4	14.8
	T5Y	44.0	40.6	19.4
	T6Y	50.4	34.6	17.5
	T8Y	51.8	27.5	19.2
	T10Y	68.2	18.7	13.6
Cannel Coals	9639	62.9	21.8	7.1
	9640	44.1	41.9	13.1
	9641	68.6	17.4	17.0
	9642	42.1	39.7	12.0
	9644	38.5	46.4	14.6
	9645	50.2	47.7	7.2
	9646	48.2	32.0	6.8
Wyoming Coals	9654	47.3	44.4	4.4
	9655	51.4	36.4	11.3
	9656	42.7	48.6	12.2
	9659	47.8	45.5	3.6

Table 8. Wt% carbon, wt% hydrogen, wt% nitrogen, wt% oxygen and sulfur and wt% ash calculated without normalization for the 70 data point set.

Coal Sample Name		Wt% Carbon	Wt% Hydrogen	Wt% Oxygen & Sulfur	Wt% Ash
Chinese Coals	T1J	66.4	5.0	15.8	11.5
	T2J	74.8	5.2	14.5	4.0
	T3J	86.3	4.2	11.2	1.3
	T4J	79.0	5.1	12.9	4.1
	T5J	76.9	4.7	7.7	8.4
	T6J	81.6	4.2	6.4	6.2
	T8J	81.9	3.6	3.5	10.2
	T9J	88.1	3.4	4.7	4.3
	T10J	94.2	3.0	2.6	3.6
Wyoming Coals	9654	4.9	4.9	13.7	4.4
	9655	4.9	4.9	11.7	11.3
	9656	4.8	4.8	14.5	12.2
	9659	4.9	4.9	14.4	3.6

These results along with the conventionally determined coal properties are presented in tables 9 to 12.

Examination of the final results indicates consistency in the trends, since those coal properties that exhibited good correlations with the Fourier transform parameters showed results when compared with the conventionally determined coal properties. In particular, wt% fixed carbon, wt% carbon and wt% volatile matter showed good correlations with high reliability factors and low average percent errors. The poor results for wt% ash are understandable because ash is principally composed of minerals. The mineral contribution to the x-ray scattering is removed from the initial intensity data. Similar comments can be made for wt% hydrogen, wt% nitrogen and wt% oxygen and sulfur.

Examination of the results in the methods employed points out clearly that expanding the range of the Fourier transform or radial distribution function to $r = 20 \text{ \AA}$ was beneficial. The expanded range revealed more structure in the radial distribution function which improved the reliability factors. On the other hand, the normalized coal properties calculated by the above technique show good comparisons with the conventionally determined coal properties. Also, it is evident that the 70 data point results show remarkably less deviation from the conventional results than the 112 data point results.

The results discussed above are encouraging. There definitely is a correlation with the FTXR parameters and coal properties. Continued efforts and the refining of the data gathering and processing should lead to improvements. The most difficult aspect of data acquisition stems from

Table 9. The normalized values for Wt% fixed carbon, Wt% volatile matter and Wt% ash as calculated with the 112 data point set accompanied by the same coal property values determined by conventional methods.

Coal Sample		Wt% Fixed Carbon		Wt% Volatile Matter		Wt% Ash	
Name		Calculated	Convention	Calc.	Conv.	Calc.	Conv.
Chinese Coals	T1J	45.7	51.2	43.4	40.3	10.9	8.5
	T2J	54.3	56.0	41.3	39.2	4.4	4.8
	T3J	65.4	67.3	33.8	31.2	0.8	1.5
	T4J	61.6	64.5	33.2	28.7	5.2	6.8
	T5J	59.3	55.4	32.3	35.0	8.3	9.6
	T6J	72.0	65.4	21.1	27.0	6.9	7.6
	T8J	76.2	73.0	14.0	16.0	9.8	11.0
	T9J	80.0	82.0	14.9	11.6	5.2	6.4
	T10J	88.7	90.8	9.8	4.7	1.6	4.5
	T4Y	55.13	57.3	29.8	27.9	15.1	14.8
	T5Y	54.0	48.2	27.0	29.5	19.0	22.3
	T6Y	59.9	54.8	20.8	23.3	19.3	21.9
	T8Y	67.7	54.1	12.3	14.7	20.0	31.2
	T10Y	79.8	83.8	10.0	5.1	10.2	11.1
Cannel Coals	9639	64.7	59.8	27.3	32.2	8.1	8.0
	9640	52.0	42.7	33.5	41.9	14.5	15.4
	9641	70.7	65.5	12.2	12.3	17.1	22.2
	9642	43.9	28.9	40.7	61.5	15.4	9.66
	9644	44.8	32.6	37.4	46.8	17.8	20.6
	9645	50.5	49.4	43.6	47.8	5.9	2.8
	9646	49.8	46.7	40.6	44.7	9.5	8.6
Wyoming Coals	9654	49.3	48.5	47.2	48.27	3.5	2.7
	9655	53.0	45.4	34.9	38.9	12.1	15.7
	9656	48.6	49.5	39.6	43.5	11.8	7.0
	9659	52.9	47.16	43.6	49.65	3.5	3.19

Table 10. The normalized values for Wt% carbon, Wt% hydrogen, Wt% nitrogen, Wt% oxygen and sulfur and Wt% ash as calculated with the 112 data point set accompanied by the same coal property values determined by conventional methods.

Coal Sample Name		Wt% Carbon		Wt% Hydrogen		Wt% Nitrogen		Wt% Oxygen & Sulfur		Wt% Ash	
		Calc	Conv	Calc	Conv	Calc	Conv	Calc	Conv	Calc	Conv
Chinese Coals	T1J	72.0	65.8	4.7	4.4	1.2	0.9	11.5	20.5	10.6	8.5
	T2J	81.7	74.8	4.4	5.05	1.3	0.8	8.0	14.6	4.6	4.8
	T3J	88.8	82.3	4.1	4.7	1.4	0.8	4.7	10.7	1.0	1.5
	T4J	80.6	78.8	4.4	4.7	1.3	0.8	8.4	8.9	5.4	6.8
	T5J	80.5	78.6	4.1	5.2	1.4	1.45	5.6	4.8	8.4	9.6
	T6J	82.9	81.7	4.0	4.8	1.4	1.4	5.3	4.5	6.5	7.6
	T8J	83.7	80.4	3.5	4.2	1.3	1.3	2.4	3.1	9.1	11.0
	T9J	85.9	86.1	3.7	3.8	1.4	1.4	2.6	2.3	6.4	6.4
	T10J	91.4	89.0	3.6	3.0	1.5	1.1	1.9	2.5	1.6	4.5
Wyoming Coals	9654	81.3	74.2	4.7	4.9	1.4	1.6	8.8	16.4	3.8	2.9
	9655	75.4	66.2	4.3	3.8	1.2	1.1	8.0	13.4	11.1	15.5
	9656	72.7	67.58	4.6	4.5	1.2	1.5	10.6	19.3	11.0	7.2
	9659	80.4	74.6	4.7	4.7	1.4	1.3	9.8	16.1	3.6	3.3

Table 11. The normalized values for Wt% fixed carbon, Wt% volatile matter and Wt% ash as calculated with the 70 data point set accompanied by the same coal property values determined by conventional methods.

Coal Sample Name		Wt% Fixed Carbon		Wt% Volatile Matter		Wt% Ash	
		Calc.	Conv.	Calc.	Conv.	Calc.	Conv.
Chinese Coals	T1J	40.5	51.2	48.8	40.3	10.8	8.5
	T2J	47.7	56.0	48.2	39.2	4.1	4.8
	T3J	65.6	67.3	33.0	31.2	1.4	1.5
	T4J	58.2	64.5	37.7	28.7	4.1	6.8
	T5J	61.08	55.4	29.9	35.0	9.05	9.6
	T6J	70.8	65.4	22.4	27.0	6.8	7.6
	T8J	76.9	73.0	13.10	16.1	10.0	10.9
	T9J	84.1	82.0	11.7	11.6	4.2	6.4
	T10J	91.5	90.8	4.8	4.7	3.8	4.5
	T4Y	50.4	57.3	35.0	27.9	14.6	14.8
	T5Y	42.3	48.2	39.1	29.5	18.6	22.3
	T6Y	49.1	54.8	33.8	23.3	17.1	21.9
	T8Y	52.6	54.10	28.0	14.7	19.4	31.2
	T10Y	67.9	83.8	18.6	5.1	13.5	11.2
Cannel Coals	9639	68.5	59.8	23.8	32.2	7.7	8.0
	9640	44.5	42.7	42.3	41.9	13.2	15.4
	9641	66.6	65.5	16.9	12.3	16.5	22.2
	9642	44.9	28.9	42.3	61.5	12.8	9.7
	9644	38.7	32.6	46.7	46.8	14.6	20.6
	9645	47.7	49.4	45.4	47.8	6.9	2.8
	9646	55.4	46.7	36.8	44.7	7.8	8.6
Wyoming Coals	9654	49.3	48.5	46.2	48.3	4.5	2.7
	9655	51.8	45.44	36.7	38.9	11.4	15.6
	9656	41.3	49.5	46.9	43.5	11.8	7.1
	9659	49.3	47.2	47.0	49.7	3.7	3.2

Table 12. The normalized values for Wt% carbon, Wt% hydrogen, Wt% nitrogen, Wt% oxygen and sulfur and Wt% ash as calculated with the 70 data point set accompanied by the same coal property values determined by conventional methods.

Coal Sample Name		Wt% Carbon		Wt% Hydrogen		Wt% Nitrogen		Wt% Oxygen & Sulfur		Wt% Ash	
		Calc.	Conv.	Calc.	Conv.	Calc.	Conv.	Calc.	Conv.	Calc.	Conv.
Chinese Coals	T1J	66.7	65.8	5.0	4.4	0.9	0.9	15.9	20.5	11.6	8.5
	T2J	75.0	74.8	5.2	5.1	1.3	0.8	14.6	14.6	4.1	4.8
	T3J	82.7	82.3	4.0	4.7	1.3	0.8	10.7	10.7	1.2	1.5
	T4J	77.1	78.8	5.0	4.7	1.4	0.8	12.6	8.9	4.0	6.8
	T5J	77.6	78.6	4.8	5.2	1.4	1.5	7.7	4.8	8.4	9.6
	T6J	81.7	81.7	4.2	4.8	1.5	1.4	6.4	4.5	6.2	7.6
	T8J	81.5	80.4	3.6	4.2	1.3	1.3	3.4	3.1	10.1	11.0
	T9J	86.3	86.1	3.4	3.8	1.5	1.4	4.6	2.3	4.2	6.4
	T10J	89.8	89.0	2.8	2.2	1.4	1.1	2.5	2.5	3.4	4.5
Wyoming Coals	9654	75.8	74.2	4.9	4.9	1.2	1.6	13.7	16.4	4.4	2.9
	9655	71.3	66.2	4.9	3.8	1.1	1.1	11.5	13.4	11.2	15.5
	9656	67.1	67.6	4.9	4.5	1.0	1.5	14.7	19.3	12.4	7.2
	9659	76.1	74.6	4.86	4.7	1.2	1.3	14.3	16.1	3.6	3.3

the fact that the halo produced by the amorphous part of the coals is of low intensity in comparison to the diffraction from the minerals. This necessitates very careful calibration and alignment of the diffractometer. Changes in the electronics employed by the diffractometer does produce changes in the intensity data.

So far this technique has been employed to correlated FTXR parameters to proximate and ultimate analyses; however, it is firmly believed that these FTXR parameters are also connected to more subtle chemical and structural properties of coal. If such, a correlation could be made then a real advance in the study of coal structures could be made.

III. Microscopic Studies of Coal Macerals and Minerals

The examination of pyrite framboids and microscopic mineral crystals extracted from coals has continued without any new results. It was earlier believed that the chemical reactivity of pyrite framboids could be linked to defects in the microcrystallites in the framboids. However, none of the diffraction results lead to such a conclusion. To the contrary, all the diffraction patterns indicate a high degree of perfection. This is also true for other microscopic crystals of minerals like TiO_2 - Anatase and Brookite.

X-ray diffraction patterns of the amorphous maceral constituents has not revealed any new information. As reported previously, the diffraction patterns are not a standard pattern for all macerals but vary from one kind to another. The d-spacings change but it has not been up to now possible to connect a d-spacing with any particular maceral. Interpretation of these patterns has proven to be even hard. There is no doubt that coal structure

problem is a difficult one and will require continued effort over a long period of time.

IV. Small Angle X-Ray Scattering

The Bonse-Hart small angle x-ray scattering camera is finally at the stage where some work can be done with it.

Dr. Richard Matyi of the Texas Instrument research laboratory has provided several crystals that are of sufficient quality for the monochromators and detectors. These have been channel-cut and etched. Their performance has turned out to be excellent.

The Apple computer to be used for data acquisition and processing for the small angle x-ray camera has been delivered and interfaced with a d-c motorized translator to move the camera through the angular displacement needed to obtain the data. The resolution of the translator is about 1 micron which is more than enough for the needs of the camera. The detection system has been synchronized with the camera motion so that the data can be recorded or digitalized.

Unfortunately, the inability to obtain the crystals needed for the camera has delayed this part of the project. However, now future work can be done.

V. Summary

Now that the project has come to an end, it is evident that it was too ambitious. As things stand at this moment, the majority of the objectives can now be pursued. The accomplishments have been:

- a) The development of the Fourier transform technique to do proximate and ultimate analyses in a quick fashion.

- b) The investigate of the structure of pyrite framboids and preliminary studies of coal macerals.

Now that the equipment is available this work will continue in the future.

VI. Publications

1. A.S. Pavlovic, J.M. Cook, and J.J. Renton, (1980). "The use of X-Ray Diffraction to Characterize Coal," Proc. of the Fourth Symposium on Electron Microscopy and X-ray Applications to Environmental and Occupational Health Analysis, Penn State University, University Park, Pennsylvania, October 15-17, 1980.
2. A.S. Pavlovic, J.M. Cook and J.J. Renton, 1981. "Characterization of Coal Properties by X-ray Diffraction," Proceeding of the International Conference on Coal Science, Dusseldorf, 7-9 September.
3. A.H. Stiller, A.S. Pavlovic, and J.M. Cook, 1980. "X-ray Diffraction Studies of Pyrite Framboids," Journal of Sedimentary Petrology, to be published in 1984.

VII. Talks

1. A.H. Stiller, A.S. Pavlovic and J.M. Cook (1980). "X-Ray Diffraction Studies of Framboids," Conference on the Chemistry and Physics of Coal Utilization, West Virginia University, Morgantown, West Virginia.
2. A.S. Pavlovic, J.M. Cook and J.J. Renton (1980). "Coal Characterization by X-Ray Diffraction," Conference on the Chemistry and Physics of Coal Utilization, West Virginia University, Morgantown, West Virginia.

3. A.S. Pavlovic (1980). "X-Ray Diffraction Studies of Coals and Coal Constituents," given at
 - i. West Virginia Institute of Technology, Montgomery, West Virginia, February 22, 1980.
 - ii. James Madison University, Harrisonburg, Virginia, December 8, 1980.
4. A.S. Pavlovic, J.M. Cook and J.J. Renton (1980). "The Use of X-Ray Diffraction to Characterize Coal," Fourth Symposium on Electron Microscopy and X-Ray Application to Environmental and Occupational Health Analysis, Penn State University, University Park, Pennsylvania, October 15-17, 1980.
5. A.S. Pavlovic, J.M. Cook and J.J. Renton, 1981. "Characterization of Coal Properties by X-Ray Diffraction," Proceedings of the International Conference on Coal Science, Dusseldorf, 7-9 September.

VIII. Personnel

Four graduate students have received instructions in energy related, specialized fields. These are as follows:

Dr. Jason M. Cook; He received his Ph.D. in 1979 and remained working on the project until 1981. He is now employed by the Morgantown Energy and Technology Center where he is in charge of their ESCA program.

Howell Caton; he is completing his masters degree and, now, is employed by the Aberdeen Proving grounds in Maryland.

Gary Devine-King; he was associated with the project for one year and is now working in the field of surface physics pertaining to catalysis.

Paiboon Tangyonyong; he has been associated with the project for about two years. He is completing his master's degree and shall continue for a Ph.D.