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THE DEVELOPMENT AND EVALUATION OF AN AUTOMATED
REFLECTANCE MICROSCOPE SYSTEM FOR THE PETROGRAPHIC
CHARACTERIZATION OF BITUMINOUS COALS

Technical Report 23

By
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NTIS-25
BinD-306
Special-164

October 1980
Date Published

Work Performed Under Contract No. AC01-76ET10615

The Pennsylvania State University
University Park, Pennsylvania

U. S. DEPARTMENT OF ENERGY

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Date Published - October 1980

PREPARED FOR
UNITED STATES DEPARTMENT OF ENERGY

Under Contract No. EX-76-C-01-2030

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ABSTRACT

Increased coal utilization has accelerated the demand for basic knowledge about coal properties and their relationships to coal processing. Petrographic analysis has been a useful method of coal evaluation, and its applications can be expected to expand. The development of automated coal petrographic techniques will lessen the demands on skilled personnel to do routine work. This project is concerned with the development and successful testing of an instrument which will meet these needs.

A survey of previous work indicated that the fundamental differences in reflectance of the three primary maceral groups should enable their differentiation in an automated-reflectance frequency histogram (reflectogram). Consequently, reflected light photometry was chosen as the method for automating coal petrographic analysis. Three generations of an automated system (called Rapid Scan Versions I, II, and III) were developed and evaluated for petrographic analysis. Their basic design was that of a reflected-light microscope photometer with an automatic stage, interfaced with a minicomputer. The minicomputer was programmed to provide the following functions: photometer control, stage control, data acquisition, data computation and output and data storage.

It was determined that the hardware elements used in the Rapid Scan Version I limited the system's flexibility and presented problems with signal digitization and measurement precision. Rapid Scan Version II was designed to incorporate a new microscope photometer and computer system. Its precision of reflectance measurement was found to be limited by photon flux and signal integration time. In order to increase the signal integration time (and improve the precision of the

measurement-) a digital stepping stage was incorporated into the Rapid Scan Version III system. The precision of reflectance determination of this system was found to be ± 0.02 percent reflectance.

The limiting factor in quantitative interpretation of Rapid Scan reflectograms is the resolution of reflectance populations of the individual maceral groups. Six factors which affected this resolution were delineated as follows: sample preparation, sampling technique, instrumental error, concentration of constituents present, overlap in the range of reflectance of macerals, and grain size. The effect in size (and hence, boundary readings) was shown to have, by far, the greatest influence.

Results generated from reflectance studies on the various coal macerals shed light on the geologic history of the test coals. The reflectance characteristics of fusinite suggested a multiple (or varied) origin, and that its optical properties change little in response to coalification. The mean reflectance of sporinite converged with that of vitrinite at a vitrinite reflectance of approximately 1.10% (mean average random reflectance). In higher rank coals the sporinite reflectance can exceed that of vitrinite. A comparison of mean average random reflectance to mean maximum vitrinite reflectance, for coals from western Pennsylvania, suggested that pressure-induced anisotropy was uniform across the basin. Uniform vitrinite anisotropy may indicate similar depth of burial (and tectonic history) for the different rank coals.

Statistical testing indicated that reflectograms were highly reproducible, and a new computer program, PETAN, was written to interpret the curves for vitrinite reflectance parameters and petrographic

composition. Vitrinite reflectance data derived from reflectograms by PETAN were shown statistically to be closely comparable to visually determined values. Maceral group determinations derived by PETAN showed deviations from visual results.

Petrographic information generated by Rapid Scan III was used to predict ASTM coke stability and to evaluate changes in coal composition resulting from fractionation procedures. Reflectograms were successfully used also for the detection and evaluation of coal blends. A method was suggested for developing coal performance prediction formulae which directly utilize reflectogram characteristics.

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	ii
LIST OF TABLES	vii
LIST OF FIGURES	ix
LIST OF SYMBOLS	xiv
I INTRODUCTION	1
A. The Role of Coal Petrography in Expanded Coal Utilization	1
B. Discussion of Coal Petrographic Characterization . .	5
II HISTORICAL REVIEW OF SYSTEMS EMPLOYED FOR THE AUTO- MATION OF COAL PETROGRAPHIC ANALYSIS	18
A. System Classification	18
B. Object-plane Scanners	19
C. Image Scanners	30
III SYSTEM STRUCTURE AND EVOLUTION	35
A. Selection and Configuration of Basic Equipment . . .	35
1. Basic Data Acquisition	39
2. Sample Preparation	47
B. Configuration of The Pennsylvania State Uni- versity Rapid Scan Version I System	54
C. Configuration of the Rapid Scan Version II System	60
1. Computer Configuration	60
2. Microscope Photometer System	65
3. Microscope-to-Computer Interface	68
4. Evaluation of Hardware Improvements	72
D. Configuration and Operation of the Rapid Scan Version III System	83
IV DISCUSSION OF RESULTS	95
A. Early Interpretations	95
B. Reflectogram Reproducibility	97
C. Resolution	108

TABLE OF CONTENTS (Continued)

	<u>Page</u>
V QUANTITATIVE INTERPRETATION OF RAPID SCAN REFLECTO-GRAMS	146
A. Determination of Vitrinite Reflectance Parameters . .	146
B. The Determination of Petrographic Compositional Data	167
VI APPLICATION OF RAPID SCAN PETROGRAPHIC COMPOSITIONAL DATA	178
A. Coke Strength Prediction	178
B. Evaluation of Coal Blends	185
C. Application to Washability Studies	194
VII SUMMARY, CONCLUSIONS, AND SUGGESTIONS FOR FUTURE RESEARCH	210
A. Summary and Conclusions	210
B. Future Work	214
REFERENCES CITED	219
APPENDIX I - Computer Program PETRO6 (Fortran IV)	229
APPENDIX II - Computer Program PLOTV1 (Fortran IV)	240
APPENDIX III - Computer Program KOLO3 (Fortran IV)	243
APPENDIX IV - Computer Program PLOTV2 (Fortran IV)	247
APPENDIX V - Computer Program CONAL3 (Fortran IV)	250
APPENDIX VI - Computer Program CBLEND (Fortran IV)	256
APPENDIX VII - Data for Coals Analyzed	258

LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	Modified Stopes-Heerlen Maceral Classification	7
2	ASTM Rank Classification	14
3	Comparison of a Simple Algorithm Programmed in Different Languages	40
4	Rapid Scan Programming Hierarchy	41
5	Comparison of Epoxy Contents of Twelve Pellets	50
6	Comparison of Calculated Epoxy Contents Versus Analyzed Epoxy Contents	53
7	Comparison of Photomultiplier Tube Sensitivity	71
8	The Effect of Photomultiplier High Voltage on Signal Stability	75
9	Effect of Luminous Flux on Signal Quality	78
10	Effect of Pre-Averaging on Signal Quality	79
11	Distance of Stage Movement During Data Acquisition at Various Stage-Drive Speeds	82
12	Example of Data Entered at the Beginning of PETR06	91
13	Results of Kolmogorov-Smirnov Test for Reflecto- gram Reproducibility	104
14	Comparison of Chemical and Petrographic Analyses for PSOC-1101 and PSOC-1102	109
15	Results of Kolmogorov-Smirnov Test for Sensitivity	111
16	Optical Properties of Bausch and Lomb Glass Reflectance Standards at 546 nm	114
17	The Number of Coal Particles Encountered During the Scanning of Coals of Different Ranks	118
18	Maceral Reflectances for Twenty United States Coals . . .	125
19	Comparison of Mean Average Random Vitrinite Re- flectance Determined Visually of Mean Average Random Vitrinite Reflectance Determined by VITFIT	165

LIST OF TABLES (Continued)

<u>Table</u>		<u>Page</u>
20	Comparison of Predicted Coke Stability (United States Steel Method) to Measured Coke Stability	182
21	Petrographic Composition of Coals Used for Blending	188
22	Density Fractionation Analysis of PSOC-134	200
23	Density Fractionation Analysis of PSOC-102	205

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	The hydrogen and carbon contents of different coal macerals at various levels of rank	8
2	An example of a typical reflectogram generated by the Leitz T.A.S.	33
3	The generalized configuration of the Rapid Scan systems	38
4	The sampling pattern for data acquisition	42
5	The plateau-slope-plateau signal response to scanning across reflectance boundaries	44
6	A comparison of signal response to scanning across various coal briquette components	46
7	The sample preparation flow chart	48
8	The hardware configuration of the Rapid Scan Version I system	55
9	The equipment used in the Rapid Scan Version I system	56
10	An example of an early Rapid Scan Version I reflectogram	58
11	The spread in reflectance readings from a glass reflectance standard (Rapid Scan Version I) . .	59
12	The hardware configuration of the Rapid Scan Version II system	61
13	The Digital Equipment Corporation PDP-8A computer system used in the Rapid Scan Versions II and III . .	62
14	The organization of the Digital Equipment Corporation PDP-8A computer system	64
15	The Leitz MPV-II photometer and AMEDA stage drive used in the Rapid Scan Version II	66
16	The modular design of the Leitz MPV-II microscope photometer system	67
17	The optical arrangement of the Leitz MPV-II microscope photometer system	69
18	The schematic for the filter-inverter-amplifier interface	70

LIST OF FIGURES (Continued)

<u>Figure</u>		<u>Page</u>
19	The variation in reflectance measurement due to improper focus	73
20	The reflectance distributions from a glass reflectance standard at two levels of photon flux . .	77
21	The relationships between the number of pre-averaged reflectance readings, the standard error of the mean, and time	80
22	The hardware configuration of the Rapid Scan Version III system	84
23	The Leitz MPV-II photometer and digital stepping stage used in the Rapid Scan Version III system . . .	85
24	The control logic for the activation of the digital stepping stage by the PDP-8A	86
25	The programming structure of PETRO6	89
26	The flow chart of the standardization program module	90
27	The flow chart of the data acquisition program module	92
28	Reflectograms of a series of coals ranging in rank from subbituminous B to semianthracite	96
29	The comparison of reflectograms from compositionally different lithotypes to visual petrographic results	98
30	The reflectograms of three subsamples of PSOC-1099 . .	99
31	The effect of increasing sample point density on the total proportional difference	101
32	A comparison of an artificial reflectogram to an actual coal reflectogram	112
33	The particle size decile statistics for six coals of varying rank and Hardgrove Grindability Index . . .	117
34	A reflectogram of a Lower Kittanning seam lithotype with a glass spike	121
35	The relationship between the standard error of the mean and increasing sample size for different macerals from three coals	123

LIST OF FIGURES (Continued)

<u>Figure</u>		<u>Page</u>
36	A comparison of the reflectances of different macerals to the associated sample vitrinite reflectance	128
37	The reflectance distributions of different macerals from PSOC-3	130
38	The reflectance distributions of different macerals from PSOC-274	131
39	The reflectance distributions of different macerals from PSOC-132	132
40	The overlap in reflectance distributions of different macerals for various rank coals	134
41	An artificial reflectogram produced from visually-derived data	135
42	A reflected light photomicrograph of a high volatile A bituminous coal containing several macerals	137
43	The relationship between perimeter and area for various shaped polygons	138
44	The relationship between particle size and the number of perimeter (boundary) Rapid Scan readings versus nonperimeter readings	140
45	The effect of decreasing particle size on a two-component system	142
46	The effect of boundary reflectance readings on two-component systems of increasing reflectance difference	144
47	The reflectance characteristics of different surfaces of a uniaxial negative vitrinite particle	148
48	A comparison of maximum and average reflectance	149
49	The correlation between mean maximum vitrinite reflectance and mean average random vitrinite reflectance for various coals	152
50	The correlation between mean maximum vitrinite reflectance and mean average random vitrinite reflectance for western Pennsylvania coals	154

LIST OF FIGURES (Continued)

<u>Figure</u>		<u>Page</u>
51	A flow chart of the logic used in VITFIT to calculate vitrinite reflectance parameters from reflectograms	158
52	An example of the program VITFIT's matching of a Gaussian distribution to the vitrinite reflectance peak	159
53	The correlation between mean maximum vitrinite reflectance and mean average random vitrinite reflectance	161
54	An example of the apportioning of vitrinite reflectance classes by program V-TYPE	163
55	An example of page one of the printout from PETAN	164
56	A comparison of mean maximum vitrinite reflectance (visual) to mean maximum vitrinite reflectance (calculated)	168
57	A flow chart of the logic used in PETAN to calculate petrographic composition from reflectograms	170
58	An example of page two of the printout from PETAN	173
59	A comparison of total reactive macerals (visual) to total reactive macerals (automated)	175
60	A comparison of predicted coke stability (automated data) to measured coke stability	183
61	A comparison of predicted coke stability (visual data) to measured coke stability	184
62	A reflectogram from a three-coal blend	187
63	The reflectograms of the three individual coals used for blending experiments	189
64	Actual and simulated reflectograms from blends of three coals in equal proportions	190
65	Actual and simulated reflectograms of similar blends	193
66	A flow chart of the coal fractionation procedure	197
67	Reflectograms from the density fractionation of PSOC-134	198
68	The change in sample composition as a result of density fractionation	202

LIST OF FIGURES (Continued)

<u>Figure</u>		<u>Page</u>
69	Reflectograms from the density fractionation of PSOC-102	203
70	The change in sample composition as a result of density fractionation	206
71	The washability curves for PSOC-134	208
72	The relationship between mean maximum vitrinite reflectance and ASTM coke stability for single coals	217

LIST OF SYMBOLS

$\bar{X}$	Sample mean
σ	Sample standard deviation
$\sigma_{\bar{X}}$	Standard error of the mean
R_0	Percent reflectance in oil
MARR	Mean Average Random Reflectance
R_w	Maximum reflectance
R_E	Minimum reflectance
R'_E	Apparent minimum reflectance
n	Refractive index
ARM	Automated Reflectance Microscope
DMMF	Dry Mineral Matter Free
MMF	Mineral Matter Free
DAF	Dry Ash Free
MMC	Mineral Matter Containing
Vit.	Vitrinite
Lip.	Liptinite
In.	Inertinite
Sin.	Semi-inertinite
Vis.	Visual
R.S.	Rapid Scan
μm	Micron
nm	Nanometer
K	Thousand
Ω	Ohm
mf	Microfarad
HGI	Hardgrove Grindability Index

I. INTRODUCTION

A. The Role of Coal Petrography in Increased Coal Utilization

The realization in recent years that future quantities of petroleum fuels are not limitless has led the United States to re-evaluate the role of coal as an energy resource. The Federal Government has supported increased coal utilization by formulating the National Energy Plan, which includes among its goals the substitution of the use of coal for oil and natural gas by utilities, and the development of a synthetic fuels industry (United States Department of Energy, 1979). Private industry, particularly the major oil companies, also has shown a vigorous interest in coal utilization. Eleven major oil companies have acquired control of 55.3 billion tons, or 25 percent, of the total United States recoverable coal reserves (Business Week, 1979). Coal production in the United States has been growing at a five to seven percent annual rate for the past two years which, although indicating increased coal utilization, is far below the 1.2 billion ton production level called for by President Carter for 1985 (Bagge, 1979).

Two thirds of the 1979 coal production (approximately 462 million tons) was consumed by the utility sector, predominantly for the generation of electricity (Bagge, 1979). By 1990, this requirement is expected to increase to approximately one billion tons of coal per year (Feibus et al., 1979). In addition to the utility sector, it has been predicted that the synthetic fuels industry will require 200 to 300 million tons of coal per year by the early 1990's (Coal Gasification and Liquefaction Newsletter, 1979).

This expected surge in coal utilization will require vigorous exploration programs to delineate the coal reserves which can contribute to expanding production. Coal consumers today are requiring long term supply contracts with guarantees on coal quantity and quality (Wilson, 1979). With increasing environmental restrictions on the quality of utility coals and more complex coal technology, such as synthetic fuels processes, coal evaluation will become of major importance in exploration programs.

Historically, the most comprehensive coal testing programs have been those applied to coal produced for the metallurgical market. Steel companies have employed petrographic, chemical and empirical-physical testing for two decades in their coal evaluation programs. Data from comprehensive testing programs have been used to gain a better understanding of the origin and geologic setting of coal seams, as well as to predict process-product quality. For the prediction of coal coking properties, the steel industries have in recent years relied heavily on coal petrographic analysis (Thompson, 1979). Their dependence on coal petrographic data developed from a need to better characterize the coals used for the production of blast furnace coke, in by-product ovens.

Microscopically, coal is a mixture of recognizable organic and inorganic components, several of which react uniquely during the coking process. Gross chemical analyses which treated coal as a homogeneous material often were insufficient to explain coking behavior. Research into the thermal behavior of the different coal components paved the way for the development of coke prediction techniques, applying coal petrographic compositional data.

A new period of basic research is developing in the coking industry (Robins, 1979). This research, in part, is directed toward understanding the coal to coke transformation, and how coal characteristics influence transformation mechanisms. Further coal and coke petrographic studies will be used to elucidate these mechanisms and how they may be modified by the addition of non-coal additives in the coking process. However, until new predictive techniques are developed and perfected, applied coal laboratories will no doubt continue to use coal petrographic analysis for routine coal evaluation and coal quality control.

Research already has indicated that coal characteristics may be important factors in developing techniques for predicting the yield and quality of liquid synthetic fuels from coal (Davis et al., 1976). Empirical prediction equations (developed from multivariate statistical testing) have shown that maceral composition, vitrinite reflectance and total sulfur content are among the most important compositional factors relating to process-product yield (Yarzab et al., 1980). Granoff (1979) has also shown that the amount and type of pyrite in coal may be related directly to process-product quality. Coal petrographic analysis has proved to be a useful technique to assess the above information during coal evaluation programs.

Coal petrographic research has also begun to utilize additional information which can be gained readily only by image examination techniques. In the past, coal petrographic analysis has been employed primarily to generate compositional data. However, to uniquely describe an aggregate material such as coal, additional information on the size, shape, association, and packing of components is necessary (Griffiths, 1961). Recent studies have begun to investigate the influence

of size of inorganic coal components on coal processing (Kuehn and Davis, 1979; Richardson, 1979). Petrographic studies of coal liquefaction residues and reactor solids, have shown the importance of species association in understanding reaction histories (Mitchell et al., 1977; Wakeley et al., 1979). Future studies may utilize additional information available only from petrographic analysis (such as particle description) to better characterize coals; and therefore, better understand their behavior in coal process technologies.

Coal petrographic analyses also have been used in various geologic investigations. Past researchers have used coal petrography to understand: environments of coal deposition (Hacquebard et al., 1968; Ting and Spackman, 1975; Reyes-Navarro and Davis, 1976; Austin, 1979; and others); correlation of coal seams (Hacquebard and Donaldson, 1974; Smyth, 1967; and others); coal metamorphic history (Teichmüller and Teichmüller, 1954; Hower, 1978); and structural fabric in coal fields (Stone and Cook, 1979; Levine and Davis, 1979).

The combination of expanded coal utilization (and the stimulus it affords for new research) with the proved utility of coal petrographic analysis, suggests that this technique should become a widespread tool for the coal scientist. Unfortunately, two factors may limit more routine use of petrographic analysis.

The first factor involves the distribution and utilization of trained petrographers in existing coal laboratories. A trained operator, employing accepted techniques, can complete an analysis (maceral composition and reflectance) on one sample in approximately three hours. Industrial acceptance of this technique has led to a situation where many available man-hours are spent doing routine analysis (Gray, 1979). Consequently, the demand for petrographic analysis has already

outstripped the capacity of existing laboratories, which are unlikely to hire and train additional personnel (Thompson and Benedict, 1976).

The second factor affecting increased use of coal petrographic data is the subjectivity of maceral analysis. During petrographic analysis the operator is required to make decisions regarding composition based on observation. The accuracy of the analysis is, therefore, highly dependent on the operator's training and experience (Chandra, 1963). Studies have shown that intralaboratory reproducibility of results for trained petrographers can be very good (Hoover, in preparation); however, interlaboratory compositional results can vary greatly (ICCP, 1978). Future acceptance of coal petrographic analysis may be affected by the ability of existing coal laboratories to narrow the differences obtained during interlaboratory analyses.

One development which could ease both of the aforementioned problems would be a system capable of rapid, objective petrographic analysis. Such a system should be capable of yielding data comparable to that of visual coal petrographic techniques, or data which provide the same basic information, perhaps in a different format. The scope of this dissertation is the development and evaluation of one such system (the Rapid Scan) at The Pennsylvania State University.

B. Discussion of Coal Petrographic Characterization

Coal can be described as a sedimentary rock composed of greater than 50 percent (by weight) combustible carbonaceous materials (Schopf, 1956). This description implies that coal is an aggregate of organic and inorganic phases. Stopes (1919, 1935) was one of the earliest workers to classify coal on its megascopic and microscopic appearances.

She observed that one could recognize a number of microscopically identifiable components comprising the organic phase of coal, and designated these components macerals (a classification term which was meant to be analogous to the term mineral, in inorganic petrology).

In the time since Stopes first defined the maceral concept, several methods have been used to classify these organic components. The system which is currently in use in most coal laboratories is the Stopes-Heerlen classification, endorsed by the International Committee of Coal Petrology (1963; 1971). This classification system (Table 1) establishes three primary maceral groups: vitrinite, liptinite (or exinite), and inertinite, and differentiates them based on origin and fundamental chemical and structural parameters. Each maceral group is subdivided into a series of individual macerals which are differentiated under the microscope by morphology and optical properties.

Vitrinite is perhaps the most important maceral group, as it is the dominant component in most United States coals ($\bar{X}$ = 78% by volume and σ = 9.0%) (Waddell et al., 1978). It is thought to be derived from the woody tissue, bark and leaves of trees, and occurs as bright, or vitreous bands and as attrital matrix. Its appearance is that of a medium gray material in reflected light, and various shades of red, orange, or brown in transmitted light. The chemistry of vitrinite is variable (depending on the level of rank or thermal metamorphism); however, its hydrogen content will be intermediate between those of the liptinite group and the inertinite group (Figure 1) and in bituminous rank coals, its oxygen content will be the highest of the three groups.

The vitrinite group macerals also are of great importance to understanding the technological behavior of coals in different processes.

Table 1

Modified Stopes-Heerlen Maceral Classification
(From Davis, 1976)

Maceral Group	Maceral	Origin		Appearance		Chemistry	Technological Behavior		
		Source	Process	Color in Thin Section	Reflected Light		Carbonization	Liquefaction	
VITRINITE	Telinite (cell walls) Collinite (cell fillings, structureless vitrinite)	Woody tissues, bark, leaves, etc.	Mummification	Red-brown	Intermediate grey	Intermediate hydrogen content and volatiles	Principal reactive constituent in coking coal	Susceptible to liquefaction	
LIPTINITE (OR EXINITE)	Pesinite	Resins	Resistant remains	Yellow	Dark grey	Higher hydrogen content and volatiles; more aliphatic	Reactive during carbonization	Susceptible to liquefaction	
	Sporinite	Spore exines							
	Cutinite	Cuticles							
	Alginite	Algae	Migration						
	Fluorinite	Plant Oils, Fats	Black						
	Bituminite	Bacterial lipoids, Fats, Proteins	Between Vitrinite & Sporinite						
Exudatinitite	Exudates from Liptinite & Vitrinites	Black							
INERTINITE	Micrinite (massive & granular)	Plant materials	Degradation products	Opaque	White, yellowish, light grey	Lower hydrogen content and volatiles; more aromatic	Generally inert during carbonization	Generally resistant to liquefaction	
	Fusinite	Woody tissues, etc.	Fire; biochem- ical oxidation						
	Semifusinite	Woody tissues, etc.	Intermediate between vitrinite and fusinite						
	Sclerotinitite	Fungal sclerotia, hyphae	Resistant remains

Figure 1. THE HYDROGEN AND CARBON CONTENTS OF DIFFERENT COAL MACERALS AT VARIOUS LEVELS OF RANK (after Dormans et al., 1957)

Various authors (Ammosov et al., 1957, Spackman et al., 1960; Schapiro et al., 1961; Thompson et al., 1966; Brown et al., 1964; and others) have shown that the proportion and characteristics of the vitrinite in a coal can be of major importance to the physical and chemical properties of its carbonized product. The importance of predicting and optimizing coke characteristics has prompted many major steel companies to establish coal petrographic laboratories for coal evaluation. Some of these industrial laboratories have found it useful to subdivide the vitrinite maceral group into two vitrinites, normal and "pseudovitrinite"; the latter is thought to have inferior coking properties (Benedict et al., 1968). The vitrinite group macerals also have been shown to be of importance during the liquefaction of coal into clean fuels. The proportion and characteristics of the vitrinite in a coal have been shown to control the yield and type of products obtained from the experimental liquefaction studies (Davis et al., 1976).

A second main group of macerals is the liptinite or exinite group. These components consist of the waxy coatings of plant parts (sporinite, cutinite), plant resins (resinite), algal remains (alginite) and various hydrocarbons of uncertain origin (fluorinite, bituminite, exudatinite). The liptinite group has the highest hydrogen content (Figure 1) and is believed to have the most aliphatic chemical structure. These components appear as dark grays or blacks in reflected light, and are yellow to red in transmitted light. In carbonization processes the liptinite group macerals are considered reactive components, and are known to become plastic and volatilize. Owing to their relatively high hydrogen content, it is thought that these components are beneficial in coal liquefaction, although their precise role is little understood.

In the prediction of coal behavior to technological processing, the liptinite components often are added to the vitrinite group, and the sum designated "total reactive macerals." Owing to their different chemical, physical and optical properties, this is undoubtedly an oversimplification; however, this use probably will be continued until more precise information is known about the different technological behaviors of the two groups.

The third, and final, group of macerals is the inertinite group. These components are thought to be of various origins, and all are altered highly from their plant precursors. In reflected light these components look bright yellow or white, and in transmitted light, are opaque or almost opaque. They have the lowest hydrogen content and are considered highly aromatic.

This particular maceral group derives its name, inertinite, from the general technological behavior of its components. It has been shown that some of these components exhibit little or no plastic behavior during carbonization (Berry, 1959). They are also relatively resistant to liquefaction processes, and are often seen virtually unaltered in the solid residues of these processes.

Some evidence exists which would indicate that semifusinite may be partially reactive in liquefaction processing. Davis (1976) reported on the liquefaction of inertinite-rich samples. He showed that in samples where the semifusinite plus fusinite reflectance was low, the conversion to liquids was higher than would be expected if these components were entirely inert. His data also showed that the semifusinite plus fusinite reflectance of the residues was significantly higher than that of feed coals, indicating that the lower reflectance species had been dissolved.

Micrinite also may prove to be partially reactive to coal processing. Stach et al. (1975) reported that the chemical properties of micrinite were not understood well, but that they differed from other inertinite macerals. They noted that micrinite yielded a large amount of volatile matter and was affected by oxidation. A survey of samples in The Pennsylvania State University, Coal Research Section, Coal Data Base showed that greater than half the samples which had high micrinite contents (micrinite greater than ten percent by volume), but otherwise normal petrographic compositions, had significantly higher than normal volatile yields. Micrinite generally cannot be seen during the petrographic examination of coke samples or liquefaction residues and may be present in some partially altered form.

Additional studies will be necessary to define the precise role of either semifusinite or micrinite in coal processing; however, it is apparent that coals selected for liquefaction ideally should have high contents of total reactive components. A certain amount of inert components, on the other hand, is beneficial for the production of strong coke for blast furnace operation.

Maceral analysis typically is carried out by petrographic examination of briquetted, polished coal samples in reflected light. The sample is moved beneath the microscope objective in a systematic grid pattern, and the operator records the maceral occurrence at each grid intersection. In accordance with the ASTM Standard procedure (1979), 500 points are determined on each of two pellets, and the data are combined and reported as volume percents.

The advantage of petrographic compositional analysis over chemical techniques is that it allows better discrimination between coal types.

Two coals can have similar ultimate analyses and yet have different maceral compositions. This occurs when the liptinite and inertinite group macerals are present in approximately equal proportions. The hydrogen-rich liptinite macerals balance the hydrogen-poor, carbon-rich inertinite macerals and the ultimate analysis can appear similar to one from a coal which predominantly is vitrinite. The two coals, however, can have significantly different technological properties. Until chemical, spectroscopic or other techniques are developed which can better characterize the organic fraction of coals, petrographic compositional analysis will continue to be of use.

A coal seam's maceral composition is established mostly at the time of its origin in the peat swamp. However, superimposed upon the initial seam composition are the changes which result from physical and thermal metamorphism acting upon the components over geologic time. This process of the change from peat to coal, is referred to as coalification, and the degree of coalification termed rank. Many authors have discussed the processes and results of coalification, including: White (1925), Schopf (1948), Dulhunty (1954), Francis (1954), van Krevelen (1961), Teichmüller and Teichmüller (1966; 1968; 1975), Stach et al. (1975), Waddell et al. (1978) and others. Past work has shown that coalification has a great influence on the composition and structure of coal and hence, affects its technological behavior. A precise measure of the degree of coalification is an important element in coal characterization.

Many different chemical parameters have been suggested as a measure of coal rank, including: carbon content, fixed carbon content, hydrogen content, volatile yield, moisture, oxygen, heating value, and various

combinations of these characteristics. The current ASTM classification system uses heating value for low-rank coals and volatile yield, or fixed carbon content, for high-rank coals (Table 2) (ASTM, 1979).

Chemical properties are influenced both by coalification and by the maceral composition of the coal. The use of systems employing bulk coal chemical analyses, such as the ASTM procedure, can result in the "misclassification" of some coals having unusual maceral compositions.

Two optical techniques have been developed which can be applied selectively to a single maceral and limit the influence of sample maceral composition on the determination of coal rank. The spectral fluorescence of liptinite (notably sporinite) has been used for low-rank coals and the reflectance of vitrinite for higher rank coals. Although spectral fluorescence has not yet been widely accepted, vitrinite reflectance measurements have been used for the prediction of the technological behavior of coal for approximately 25 years.

Reflectance is the amount of vertically incident light which is reflected from a polished, planar surface; it is generally expressed as a ratio or percentage of the amount of incident light. In the case of a highly absorbing medium, such as coal, the amount of light reflected under standard measurement conditions ranges from a few tenths of one percent to approximately seven percent.

Reflectance of an absorbing material can be calculated from Beer's equation:

Table 2
ASTM Rank Classification

Class	Group	Fixed Carbon Limits, percent (Dry, Mineral-Matter-Free Basis)		Volatile Matter Limits, percent (Dry, Mineral-Matter-Free Basis)		Calorific Value Limits, Btu per pound (Moist, ² Mineral-Matter-Free Basis)		Agglomerating Character
		Equal or Greater Than	Less Than	Greater Than	Equal or Less Than	Equal or Greater Than	Less Than	
I. Anthracitic	1. Meta-anthracite	98	--	--	2	--	--	} non-agglomerating
	2. Anthracite	92	98	2	8	--	--	
	3. Semianthracite ³	86	92	8	14	--	--	
II. Bituminous	1. Low volatile bituminous coal	78	86	14	22	--	--	} Commonly agglomerating ⁵
	2. Medium volatile bituminous coal	69	78	22	31	--	--	
	3. High volatile A bituminous coal	--	69	31	--	14,000 ⁴	--	
	4. High volatile B bituminous coal	--	--	--	--	13,000 ⁴	14,000	
	5. High volatile C bituminous coal	--	--	--	--	11,500	13,000	
						10,500	11,500	agglomerating
III. Subbituminous	1. Subbituminous A coal	--	--	--	--	10,500	11,500	} non-agglomerating
	2. Subbituminous B coal	--	--	--	--	9,500	10,500	
	3. Subbituminous C coal	--	--	--	--	8,300	9,500	
IV. Lignitic	1. Lignite A	--	--	--	--	6,300	8,300	} non-agglomerating
	2. Lignite B	--	--	--	--	--	6,300	

1. This classification does not include a few coals, principally nonbanded varieties, which have unusual physical and chemical properties and which come within the limits of fixed carbon or calorific value of the high-volatile bituminous and subbituminous ranks. All of these coals either contain less than 48 percent dry, mineral-matter-free fixed carbon or have more than 15,500 moist, mineral-matter-free British thermal units per pound.
2. Moist refers to coal containing its natural inherent moisture but not including visible water or the surface of the coal.
3. If agglomerating, classify in low-volatile group of the bituminous class.
4. Coals having 69 percent or more fixed carbon on the dry, mineral-matter-free basis shall be classified according to fixed carbon, regardless of calorific value.
5. It is recognized that there may be nonagglomerating varieties in these groups of the bituminous class, and there are notable exceptions in high volatile C bituminous group.

$$R_0\% = \frac{(n - n_0)^2 + n^2 \kappa^2}{(n + n_0)^2 + n^2 \kappa^2} \times 100 \quad (1)$$

where,

$R_0\%$ = the reflectance in a given measuring medium (o = oil), in percent,

n = the refractive index of the material,

n_0 = the refractive index of the measuring medium, and

κ = the absorption index of the material.

Calculation of reflectance using Beer's equation, requires the previous determination of the refractive and absorption indices. In practice, coal reflectances are generally determined directly, by calibrating the photometric system with a series of glass or mineral reflectance standards (see Section IV).

The vitrinite maceral group generally is selected for reflectance determinations, for the following reasons: A) it is abundant in most coals; B) it is present in large, homogeneous pieces; and C) its reflectance changes uniformly with increasing coalification. Vitrinite reflectance analysis is carried out under standard conditions using oil immersion microscope objectives and monochromatic light (ASTM, 1979). Huntjens and van Krevelen (1954) were able to show that the refractive and absorption indices of vitrinite changed in a uniform manner with increasing coalification. In lower rank coals the refractive index was the most important factor; whereas, with increasing rank the absorption index became the dominant factor. Various authors (van Krevelen, 1961; Davis, 1971; Benedict and Berry, 1966; Thompson and Benedict, 1974; McCartney and Teichmüller, 1972; and Waddell *et al.*, 1978) have shown the relationships between vitrinite

reflectance and the various chemical parameters used as rank indicators. Researchers also have shown how vitrinite reflectance is related to the carbonization properties of coals (Shapiro et al., 1961; Thompson et al., 1966; Brown et al., 1964; and Gray et al., 1978). Although there is some divergence of opinion as to which vitrinite type should be measured, and which vitrinite reflectance parameter (maximum or average random) should be reported (Davis, 1978), vitrinite reflectance analysis is considered by many as the best single indicator of coal rank and coal technological behavior (McCartney and Teichmüller, 1972; Gray, 1979).

Vitrinite reflectance data, along with coal maceral analysis, have been used in several bivariate, coal classification systems (e.g., Bennett and Taylor, 1970; Ting, 1969). Strauss et al. (1976) have related a two-component petrographic classification system directly to basic chemical and physical properties (such as calorific value, volatile matter, and swelling index). Davis (1978) related vitrinite reflectance directly to the ASTM coal rank classification system.

Although reflectance measurements are generally carried out on vitrinite, some data also exist for the reflectance of other macerals. Durmans et al. (1957) determined the reflectance of exinite, vitrinite, and micrinite from coals of different ranks. Their data, show that for coals from 80 to 88 percent carbon, the reflectance of the three groups is well separated, with exinite being the lowest reflecting, and micrinite having the highest reflectance. For coals with greater than 88 percent carbon, the reflectances of the three groups begin to converge. Davis (1978) reported the reflectances

of vitrinite, semifusinite and fusinite for several coals of different rank. He showed that in bituminous rank coals the reflectances of the three groups were well separated, but in a Pennsylvania anthracite the vitrinite and fusinite had approximately the same reflectance.

In summary, coal petrographic characterization commonly is concerned with determining the amounts of the various macerals present in coal and the reflectance of the vitrinite component. The fundamental differences in the reflectances of the different maceral groups have suggested for some time a method by which the petrographic analysis of bituminous rank coals could be automated. An instrument could be designed to scan the surface of the polished coal sample, and record reflectance readings from the components which fell beneath the sensing spot. A signal classifier then could discriminate if the reading was low (liptinite), intermediate (vitrinite) or high (inertinite). The relative number of readings in each class, at the completion of the scan, would be a measure of the volume of each component in the sample. Furthermore, if the discriminator was calibrated against reflectance standards, then the position of the vitrinite class could be used to calculate the actual vitrinite reflectance value. This, then, is the thesis upon which development of the Rapid Scan system, and prior systems, has been based.

II. HISTORICAL REVIEW OF SYSTEMS EMPLOYED FOR THE AUTOMATION OF COAL PETROGRAPHIC ANALYSIS

A. System Classification

For almost 50 years coal scientists have been attempting to automate coal petrographic analysis. During this period, electronics technology has progressed from simple vacuum-tube circuitry to large-scale, solid-state integration of microprocessors. Each new research effort has made use of the latest up-to-date electronic developments of its time period. However, despite changes in the equipment components, the approach to system design generally can be classified into one of two system types:

- A) Object- or Specimen-plane scanners and
- B) Image scanners.

Due to their optical and electronic simplicity, the majority of automated systems applied to coal analysis, fall into the object-plane scanner type. The sample in this system is moved mechanically beneath a stationary sensing device, generally consisting of a photorell or photomultiplier tube. The magnitude of the sensor output is classified in relation to the different components in the sample. Systems of this type are capable of determining modal composition by applying the Rosiwal method of lineal analysis, or point-count techniques. By applying a Rosiwal analysis, these systems also are capable of determining chord-size distributions of the different sample components.

Commercial image scanners were introduced for quantitative microscopic analysis in 1963 (Cole, 1971). These systems generally use a Vidicon or Plumbicon electron camera tube to detect and scan one

entire microscope image. The image is processed into picture elements or "pixels," which can, in turn, be classified into various gray levels by preset discriminators of densitometers. During automated scanning as many as 650,000 pixels are processed for each image, after which a new field is positioned beneath the camera by mechanical stage movement. Due to the high pixel density and on-line memory capability of image scanning, these systems can generate a variety of size and shape parameters, in addition to modal composition. (For a more rigorous discussion of automated microscope classification systems, see Kuehn and Davis, 1979.)

The subsequent historical review is divided into two sections discussing automated microscopy as applied to coal petrographic analysis. The first section will describe research efforts which employed object-plane scanners used for this purpose, beginning in 1934. The second section will deal with image scanners, which first were applied to coal petrographic analysis around 1975. Both types of systems still are being developed today.

B. Object-plane Scanners

The first effort to automate coal petrographic analysis occurred during the 1930's in Europe. Professor M. Vendl (1934) described an object-plane scanning system, which he proposed could be used for "a quick and effortless determination of the quantity of the mineral components of coals and those of mineral bearing rocks in volume-percent." The system consisted of a polarizing, reflected-light ore microscope, equipped with an automated "clockwork" stage and a photocell to detect the amount of light reflected by the specimen. The signal from the

photocell was coupled to a mirror galvanometer which recorded changes in output on a clockwork-driven metal cylinder covered with light-sensitive paper. At the completion of a scan, the paper was removed, developed, and the resulting trace was interpreted for volume percent and chord-size distributions of the various components, following the Rosiwal method of lineal analysis. Professor Vendl did not report on the application of his device; however, he did note that problems of interpretation arose when reflectance values were recorded on the intersection of two or more components.

Twenty-one years after Vendl's experiments, McCartney and Hofer (1955), working at the United States Bureau of Mines, published a paper on very similar research. Their equipment was almost identical to that of Vendl, with the exception that they utilized a photomultiplier detector for better sensitivity, and a pen-recording microammeter rather than a mirror galvanometer. Scans were made on briquetted coal samples and the output from the recording microammeter was interpreted on a binder-free basis, utilizing the Rosiwal method of lineal analysis. The derived data then were plotted as cumulative-frequency reflectance distribution curves. The authors showed that the resulting curves were very similar for duplicate runs, and could be used for detecting gross differences in petrographic composition between bright and splint coals. Their system was never used widely, owing to the time necessary to complete a single scan (3.5 hours), and the additional time necessary to translate the data from the recording microammeter into the cumulative-frequency, reflectance distribution curves. The authors also noted difficulties in data interpretation stemming from reflectance

readings on coal/binder areas and on areas consisting of more than one coal component.

The next period of research and development on object-plane scanners as applied to coal analysis occurred in the middle to late 1960's. These developments were catalyzed by the availability of high-speed, electronic counting circuitry. The application of electronic counters for data acquisition and storage promised to reduce drastically the time necessary to perform an analysis.

The first of these systems was that developed by Bomberger and Deul (1964) to study pyrite particle sizes in fine-coal cleaning processes. Their equipment consisted of an automated, scanning reflected-light microscope with a photomultiplier detector. The output from the photomultiplier was coupled to an electronics package which was able to discriminate eight chord-size classes using reflectance differences and record their frequency on electronic counters. There was no mention by the authors of using this system to determine coal petrographic composition or vitrinite reflectance data.

Bayer, Denton and Hassel (1968) capitalized on the research of Bomberger and Deul and developed the AMEDA (Automatic Microscope Electronic Data Accumulator), manufactured by Femco, Incorporated. Their equipment, similar to Bomberger and Deul's, utilized logic circuitry and electronic counters to discriminate and size microscopic constituents based on reflectance properties. The system, as designed, could discriminate between and count up to four different microscopic constituents, and size these entities into any of nine size classes. Data could be accumulated at rates of up to 1000 readings per second, and a complete analysis generally took less than 20 minutes.

The AMEDA was produced and sold commercially and found applications in several areas, including: the study of the amount and size distribution of pyrite in coal; the amount and size distribution of pores in metallurgical cokes; and the amount and size distribution of various metallographic features in steels and tin plates (Bayer et al., 1968). In addition to these applications, Berry et al. (1967) suggested that this system could be applied to coal petrographic-compositional analysis and vitrinite reflectance analysis.

Workers at the United States Bureau of Mines developed an automated microscope during the late 1960's and early 1970's (McCartney and Ergun, 1969; McCartney et al., 1971). The microscope component of their equipment was similar to that of the AMEDA with the exception that they employed a unique spiral scanning technique, thought to lessen sample vibration. The analog-signal output from the photomultiplier was fed to a digitizer and then to a printer, which printed the value on paper and also stored it on magnetic tape. The sample rate was approximately 80 readings per second, and the scan rate approximately 175 microns per second. At the completion of a sample scan, the reflectance data recorded on magnetic tape were processed by an off-line computer. The computer program could discriminate and count up to eight components and could generate size distribution data on the pyrite present in the samples.

The first applications of this equipment (McCartney and Ergun, 1969) were directed toward evaluating the influence of pyrite size distributions on coal cleaning methods. The authors showed that the volume percent of pyrite in the sample derived from an automated analysis compared favorably with visual and chemical analysis. However,

they also noted that pyrite-size parameters determined automatically were subject to problems with particle aggregation and insufficient sampling.

The second application of this equipment (McCartney et al., 1971) was the first systematic attempt to derive coal petrographic compositional data from automated microscope systems since McCartney and Hofer (1955). In this operational mode, the reflectance data were processed into eight reflectance classes. Individual class intervals were established for binder, exinite, vitrinite, inertinite and pyrite on the basis of a preliminary conventional reflectance analysis. The remaining three classes were used to explore reflectance boundary regions between binder and exinite, exinite and vitrinite, and vitrinite and inertinite.

Published data showed that the system was capable of differentiating between bright coals (vitrinite-rich) and dull coals (liptinite- and inertinite-rich). Attempts to duplicate visual petrographic compositional analyses were not as successful; the authors found that arbitrary adjustments to the class intervals were necessary to equate the automated result with visual analyses. They attributed these differences to the inability of automated scanning to resolve finely structured petrographic components, and difficulties in separating the vitrinite reflectance population from the inertinite reflectance population. They concluded with the suggestion that despite problems with data interpretation, automated petrographic analyses might be useful in predicting coal utilization behavior.

The next period of active research on the application of automated object-plane scanners to coal analysis began in the middle 1970's, and

still is continuing at present. This surge in research, like the previous, was the result of new technological developments. During the middle 1970's, developments on mini and microcomputer systems made computer technology economically attractive as on-line instrument components. The dedicated minicomputer could serve as the data acquisition device, the microscope controller and stage controller, and the data storage manipulation and output device. During this same period, improvements were made in microscope photometers, and digital stepping stages became available. These developments enabled coal researchers to approach the problem with a level of precision that was heretofore unavailable.

During the years 1973-1975 researchers at Nippon Steel Corporation and The Pennsylvania State University reported on simultaneous developments of computerized, object-plane scanners used for coal petrographic analysis. Both groups utilized automatic, scanning, reflected-light photometers interfaced to minicomputers for data handling.

Spackman et al. (1973) discussed preliminary results of interfacing an automated reflectance photometer with a PDP-9 computer. The computer registered four reflectance classes said to represent inertinite, semifusinite, vitrinite and exinite. The system was used for screening large numbers of samples to detect gross differences in maceral composition. An analysis required 15 minutes and reproducibility was reported as good.

Kojima et al. (1974), working at Nippon Steel Corporation, cited the following as the justification for automation: speeding up analyses, reducing the need for skilled microscopists and the reduction of operator-induced errors in analyses. The system configuration

consisted of a Leitz Orthoplan-pol reflected-light microscope photometer coupled with an IBM-1800 computer. The microscope was equipped with a digital stepping stage which, under computer control, moved in ten-micron increments. The output from the photomultiplier was interfaced to the computer analog-to-digital converter, which processed the signals into one of 512 voltage categories. The voltage categories were calibrated by an internal calibration curve, derived from reflectance standards into reflectance categories.

An analysis involved 10,000 individual reflectance readings, 50 microns apart, taken in about 12 minutes. The reflectance readings were compiled into a reflectance histogram, with frequency of reading on the ordinate and the 512 reflectance categories on the abscissa. Quantitative compositional and vitrinite reflectance data were derived from the reflectogram utilizing a curve-stripping technique. Gaussian curves were matched to the vitrinite peak, and to the region of the reflectogram they thought represented the semi-inertinite macerals (see Davis, 1978). The various areas of the profile were integrated to give approximate values for the volume composition of the different maceral groups. Corrections were then made for the epoxy binder, "noise," and inertinite reflectance readings which reported to the low reflecting region of the reflectogram. The average vitrinite reflectance also was determined from the mean of the vitrinite reflectance population.

The resulting automated compositional analyses were compared to conventional visual analyses for three coals. The reported agreement between the two techniques was within the error normally associated with visual analyses, and indicated that this tool held much promise for rapid coal compositional analysis. The authors revealed that data

derived from this equipment were used for coke quality predictions and for coal quality control.

Davis (1975) reported on further developments of the automated reflectance microscope (ARM) system at The Pennsylvania State University. This system was very similar to the Nippon Steel Corporation equipment, with the primary difference being the method of sample movement (continuous for the Rapid Scan, versus stepping for the Nippon Steel Corporation system). (See Section III for a description of the Rapid Scan equipment.) Davis' paper was concerned primarily with the measurement of pyrite chord-size parameters; however, he noted that the shape of the coal reflectogram was related to the samples' petrographic composition.

The progress of these two research groups sparked renewed interest in automated object-plane scanning techniques among various organizations, including United States Steel Corporation and West Virginia University. The next reported developments came in 1976 with the presentation of two papers by The Pennsylvania State University group. Davis and Vastola (1976) described the continued development of the Rapid Scan system at The Pennsylvania State University. They discussed the comparison of pyrite-size parameters derived from the automated system to those done visually, and concluded that the automated system could generate comparable results. They also demonstrated that different coals possessed quite different pyrite-size distributions.

In addition to studying pyrite, the authors reported that coal reflectograms could be used as a measure of the rank of a coal. They showed preliminary results indicating that mean maximum vitrinite reflectance values, calculated from the reflectogram, compared well with

those determined by the conventional technique. They also showed that the shape of the reflectogram responded predictably to changes in coal petrographic composition.

Hoover et al. (1976) also reported on the use of reflectograms to petrographically characterize coals. They showed that the mean average random vitrinite reflectance (see Section V) determined from the reflectogram could be converted directly to a mean maximum vitrinite reflectance through the use of a mathematical correction factor. They also suggested that statistical techniques could be used to calculate coal petrographic composition directly from the reflectogram.

In 1979, workers at the United States Steel Corporation reported on two generations of equipment developed during the preceding three years in conjunction with Scientek Instrumentation, Incorporated. Gray et al. (1979a) discussed the application of their Mark I and Mark II systems for determining vitrinite reflectances and summary petrographic compositional data. The Mark I system was very similar to The Pennsylvania State University's early Rapid Scan system (hereafter referred to as Rapid Scan Version I). The primary differences were that the United States Steel Corporation system used photon-counting techniques to record reflectances and a video graphics terminal to display reflectograms. The Mark II system included a more sophisticated microcomputer assembly and a ten-micron digital stepping stage. The computer programming for both systems was stored on Read-Only-Memory (ROM) integrated circuits, and various operational modes were activated by keyboard commands.

The authors revealed that a computer program was developed to process the Mark I data into conventional vitrinite reflectance

parameters. They indicated that for a set of 181 coals, the correlation coefficient for visual versus automated data was 0.99, and that the Mark I system was used routinely for the measurement of vitrinite reflectance.

Data from the Mark II system was used to generate summary petrographic compositional analyses, as well as vitrinite reflectance parameters. Comparisons of automated results to visual analyses showed that the system was not then capable of yielding comparable compositional data. The correlation coefficient for total reactive macerals (automated) versus total reactive macerals (visual) was only 0.60. The authors did suggest that a modified version of their Mark II system might be used for coke plant coal quality control.

The most recent review of world-wide developments occurred during a session of Automated Coal Petrology at the Ninth International Conference of Carboniferous Stratigraphy and Geology in May, 1979. Groups from the United States Steel Corporation, The Pennsylvania State University, Nippon Steel Corporation and West Virginia University all reported on recent research.

Gray et al. (1979b) discussed the evolution of a Mark III system jointly developed by the United States Steel Corporation and Scientek Instrumentation, Incorporated. This newest system incorporated a dual floppy disk for the storage of programs and reflectance data. The authors indicated that improved system flexibility enabled them to utilize more sophisticated data acquisition and processing programs. They felt that this would improve the system's ability to determine petrographic compositional data.

Hoover and Davis (1979) discussed the factors which control reflectogram shape, and how these must be included in any data reduction program. They discussed the procedure for deriving vitrinite reflectance and petrographic compositional parameters from Rapid Scan reflectograms. They also showed how these data were used for coke quality prediction and for coal blend detection.

Kuehn (1979) discussed the stereologic implications of using object plane scanners and image scanners for automated petrographic analysis. He noted that both systems were capable of performing accurate volumetric analyses if proper sampling techniques were observed. The apparent advantages of the two-dimensional data, available from image scanners, were offset by the simplistic models necessary for data interpretation. Kuehn also noted that non-random samples accounted for most of the analytical error in automated analysis of pyrite volumes and size distributions.

Kojima et al. (1979) provided an update on work in progress at the Nippon Steel Corporation. They presented results from a series of 22 coals, and indicated that their system could determine summary petrographic compositions to within ± 5 percent. Also, they stated that their device was in routine use for predicting coke quality and for coal blend control.

Ting and Klinkachorn (1979) presented some preliminary data from a system developed at West Virginia University. They used object-plane scanning to produce reflectograms, but employed a unique algorithm to reject reflectance readings on component boundaries. They also suggested that vitrinite mean maximum reflectance could be determined automatically, by using a rotating polarizer and a three-azimuth

calculation. The results presented were insufficient to indicate if any advantages were gained by utilizing their techniques.

In summary, object-plane scanners have been applied to coal analysis for approximately 50 years. During this time, three periods of accelerated research efforts have evolved as a result of technological advances, predominantly in the area of data acquisition and manipulation hardware. Recent developments have shown that minicomputer-based systems can generate useable vitrinite reflectance determinations, and probably these systems will find application in the coking industry. Scientek Instrumentation, Incorporated is currently offering a commercial version of the equipment developed in conjunction with the United States Steel Corporation for quality control applications. Future research is likely to be directed toward a better understanding of the response of the coal reflectogram to changing coal characteristics, and its relationship to coal utilization behavior.

C. Image Scanners

Image scanners have not been used widely in the petrographic characterization of coals for several reasons, including: Early versions lacked the sensitivity to detect small changes in gray levels. The systems had inherent problems with shading, response linearity and reliability. Technically, the equipment was complex and relatively expensive. Despite these drawbacks, several groups did experiment with systems during the mid-1970's, and recent technical advances have spurred new interest in their application to coal petrographic analysis.

Harris (1975) was the first to report the use of an image scanner for coal petrographic analysis. He employed a Quantimet 720 image

analysis instrument to examine pyrite size distributions from the Lower Kittanning seam of Pennsylvania. He claimed that estimates of the volume composition of pyrite in a sample, as well as particle size distributions, could be obtained by image scanning.

Harris (1976) later described experiments with the Quantimet 720 system to study coal petrography. In this project he correlated threshold (or gray level) settings on the instrument with reflectance standards. By scanning the sample, he was able to generate 30-channel reflectograms, similar to those obtained by object-plane scanners. He noted two peaks on the reflectograms, representing the epoxy binder and the vitrinite in the sample. The reflectance of the vitrinite peak was comparable to conventionally determined values and the peak increased in reflectance when he scanned coals of higher rank. Harris concluded that "the potential for doing automated coal petrography by means of optical image analysis is very promising."

Lebiedzki and Dutcher (1977) discussed the use of a different type of system to study pyrite occurrences in Illinois coals. Their equipment consisted of a high-resolution image dissector camera mounted on a reflected light microscope and interfaced to a control computer. Various pyrite characterization parameters, including mean size, volume percent, and a shape factor were determined using the instrument. Advantages listed for this system included high resolution and the ability to scan all pyrite occurrences in a single field of view without mechanical translation. However, no systematic studies using this equipment have been reported.

Zeiss (1979) reported on the development of an image scanning system at the Bethlehem Steel Corporation. His equipment was based

on a sophisticated Leitz T.A.S. image analyzer coupled with a DEC PDP-11/34 minicomputer for data reduction. The Leitz system was equipped with a special densitometer module which enabled it to discriminate 100 gray levels of resolution. With this equipment, the author was able to produce reflectograms with an individual channel resolution of 0.02 percent reflectance. Reproducible reflectograms could be produced by scanning 40 fields of view; the operation taking less than ten minutes. (See Figure 2.) Zeiss noted that the histograms could be used directly to detect gross changes in coal rank or type; however, he also indicated that to derive compositional data would require sophisticated statistical treatment.

Chao et al. (1979) detailed an image scanning system developed at the United States Geological Survey. Their particular equipment was software based and able to discriminate 64 gray levels. Unlike other researchers working to generate petrographic compositional analysis, these workers were studying coal seam structure as revealed by lithotype and microlithotype analysis. They were working with coal seam column samples consisting of blocks of coal. The blocks were scanned with only the aid of the television camera lens to characterize seam structural features and, later, complex regions were subjected to microscope analysis. Despite the elaborate nature of the study, it did present a novel and potentially fruitful application of image scanning applied to coal analysis.

The most recent group to disclose the use of image scanning for coal petrographic analysis, was the Broken Hill Proprietary Company Limited of Australia. England et al. (1979) reported using a Quantimet 720 to generate coal reflectograms. This instrument was an improved

Figure 2. AN EXAMPLE OF A TYPICAL REFLECTOGRAM GENERATED BY THE LEITZ T. A. S.
(after Zeiss, in preparation)

version of the type used by Harris (1975; 1976) and was capable of discriminating 100 gray levels over each scanned field. The output from the equipment was a limited-resolution reflectogram which could be used to "fingerprint" coals and could be processed to yield compositional and reflectance data. The authors also used the instrument's sizing algorithms to generate size parameters on inertinite macerals.

In summary, sophisticated image scanners are only now being systematically used for coal petrographic compositional analysis. Future developments may hinge on improved instrumentation sensitivity and the application of this technique's unique ability for pattern recognition (i.e. size and shape). Relative cost, reliability and hardware dependence are all factors which continue to be improved with each new generation of instrumentation.

III. SYSTEM STRUCTURE AND EVOLUTION

A. Selection and Configuration of Basic Equipment

During the course of this research program the fundamental question has been raised: why pursue automated reflectance object-plane scanning, rather than image scanning as the method for automating coal petrographic analysis? Essentially three arguments can be presented to indicate the rationale for choosing the former technique.

Sensitivity is the first major factor which must be evaluated in any instrument consideration. Image analysis employs digitization of a projected image into gray levels, in order to measure relative reflectance. The most sophisticated image analysis system employed for automated coal analysis (Leitz T.A.S. system) can discriminate only 100 gray levels. The systems available during the mid-1970's had even poorer resolutions, often 30 gray levels or fewer (Hougardy and Rose, 1971). Thus, in scanning a reflectance range of zero to two percent (the general range of coal reflectograms for bituminous rank coals) these systems had reflectance class intervals of approximately 0.10 percent, which was insufficient for detecting small reflectance differences between coal components. In addition, image analyzers of this period had severe problems with field shading and response linearity (Cole, 1971).

A photometer-based system has its precision limited only by the photon statistics of the object measured, and by the resolution of the signal digitizing device. The Leitz MPV-II photometer, as utilized in the Rapid Scan system, has a precision of reflectance reading of ± 0.02 percent reflectance ($\pm 2 \sigma$) (Table 10). Thus, this photometer system

has at least one order of magnitude better gray level resolution than image scanners; this is similar to differences in the resolving capability reported by Hougardy and Rose (1971).

The second area of consideration was the trade-off between speed and sampling. Image analysis systems generate excellent sampling statistics per field analyzed (several hundred-thousand picture points per field). However, the heterogeneity of a coal sample does not manifest itself in a single field; hence, the number of fields scanned, rather than the number of picture points per field, is a more important sampling criteria. Researchers employing these systems have yet to standardize the number of fields necessary for sufficient sampling.

The Rapid Scan system employs lineal analysis as its sampling method. This procedure yields an unbiased estimate of the volume contribution of different components in a short period of time. Experimental evidence shows that a scan length of 125-millimeters provides sufficient sampling for reproducible reflectance profiles (see Section IV). With current hardware and sample integration time constraints, a Rapid Scan analysis can be performed in less than 15 minutes, making it one of the faster automated reflectance microscope systems.

The final consideration was the reliability-cost relationship. The reflectance photometer is standard equipment in most coal petrographic laboratories; hence, its technology is understood well by laboratory personnel. On the other hand, image analysis systems are based on complex hardware-dependent technologies, new to coal scientists. It has been reported that analytical errors of up to 100 percent can be attributed to operator error (Hougardy and Rose, 1971). Current image analysis systems equipped for coal reflectance work, cost in

excess of \$100,000 with some systems costing in excess of \$225,000.

An automated reflectance microscope system can be assembled, including computer, for under \$50,000. If a coal laboratory already is equipped with a reflectance photometer, the investment necessary might be halved.

Hence, based on considerations of precision, sampling, cost, and reliability, automated reflectance object-plane scanning microscopy appeared to be the better technique for automating coal petrographic analysis. In some laboratories, however, image analysis systems have been purchased to provide services unrelated to coal analysis. In such cases, cost is no longer a factor, and advantage may be taken of the instrument's availability, to extend its use for coal petrographic investigations.

The general configuration of the three generations of automated reflectance microscope systems developed at The Pennsylvania State University is depicted in Figure 3. All three systems consisted of an automated object-scanning reflectance photometer, interfaced through a signal enhancement device to a minicomputer. The minicomputer provided a series of functions in these systems, namely; photometer control, stage control, data acquisition, data computation and output, and data storage. The computer also has been used in an off-line mode to provide statistical data treatment and a variety of coal-performance prediction calculations. These systems were structured as software-based devices, constructed predominantly of commercially available hardware. This approach was taken in order to achieve maximum system flexibility with minimum component dependence. For a similar reason, Fortran IV was chosen as the main programming language. In choosing a

Figure 3. THE GENERALIZED CONFIGURATION OF THE RAPID SCAN SYSTEMS

program language, ease of programming and flexibility must be weighed against execution speed and ability to address interface modules. Table 3 indicates the difficulties which can be experienced when programming in other than high-level languages, such as Fortran. Even a simple algorithm to add two numbers becomes many statements in assembly code, and an overwhelming array of octal numbers in machine code. However, interface modules, such as the analog-to-digital converter, can be addressed only by assembly or machine code programs. The most effective way to meet these programming needs was to adopt a system of modular programming, employing subroutines written in various level languages (Table 4). This approach enabled most of the software to be written in Fortran IV, with special subroutines tailored for specific operations. Also, with most of the software written in Fortran IV, changes to the program could be accomplished easily and rapidly, adding to system flexibility.

1. Basic Data Acquisition

The method of sampling and data acquisition varied very little between the different Rapid Scan systems. Lineal analysis was chosen as the sampling technique which could provide the most information in a given time. The automatic stage moves the sample in a continuous traverse (Rapid Scan I and II) or in a stepping mode (Rapid Scan III). The computer is programmed to read reflectance signals in such a manner that adjacent readings are recorded from the sample surface (Figure 4). After each reflectance spot is sampled, its corresponding element in a 1000-element array is increased by one increment. Thus, the data is stored as a numeric histogram with frequency of occurrence recorded in

Table 3

Comparison of a Simple Algorithm Programmed
in Different Languages (From Galbraith et al., 1975)

(1) *English*

Add twelve and nine.

(2) *Fortran*

```
JVAL = 12 + 9
STOP
END
```

This program is the simplest possible, a *reductio ad absurdum*; it does not even print the result.

(3) *Assembly code*

<pre>.TITLE MAIN .CSECT .GLOBL MAIN MAIN.: JSR %4,\$POLSH .GLOBL \$POLSH,\$NAM \$NAM,0,0,050561,055740 .GLOBL \$SEQ \$SEQ,000001 \$P0001 \$P0002 .GLOBL \$ADI \$ADI .GLOBL \$POP2 \$POP2,JVAL .EVEN</pre>	<pre>↓ .CSECT \$SEQ,000002 .GLOBL \$STOP \$STOP .BYTE 0 .EVEN \$P0001: MOV #000014,-(%6) JMP @(%4)+ \$I0000: 000014 \$P0002: MOV #000011,-(%6) JMP @(%4)+ \$I0001: 000011 JVAL: 0 .GLOBL \$OTS .END MAIN</pre>
--	--

This program is incomplete, as it contains calls to library subroutines which have not yet been linked to the main program.

(4) *Machine code expressed as 6-digit octal numbers*

```
004467 000206 074044
000000 000000 050561 055740 074130 000001 073756 073766
074004 074000 073776 074130 000002 074020 000000 012746
000014 000134 000014 012746 000011 000134 000011 000000
012634 000134 062616 102002 004567 000122 000134 006403
010446 012746 000004 104042 016746 000006 000004 004567
001750 000350 016700 001572 010467 001566 005714 001016
010024 016724 001556 022424 000134 016704 001544 011467
```

↓
(27 more sections of 32 numbers each are omitted here)

```
000000 000000 000000 000000 000000 000000 000000 000000
000000 000000 000000 000000 000000 000000 000000 000000
000000 000000 000000 000000 000000 000000 000000 000000
005724
```

The total length of the program is 948 octal numbers (or words).

(5) *Machine code expressed as 16-bit binary words*

```
0000100100110111 0000000010000110 0111100000100100
0000000000000000 0000000000000000 0101000101110001 0101101111100000
0111100001011000 0000000000000001 0111011111101110 0111011111110110
0111100000000100 0111100000000000 0111011111111110 0111100001011000
0000000000000010 011110000010000 0000000000000000 0001010111100110
0000000000001100 0000000010111100 0000000000001100 0001010111100110
0000000000001001 0000000010111100 0000000000001001 0000000000000000
```

Only the first section of 27 words is shown, equivalent to the first section of octal numbers.

Table 4

Rapid Scan Programming Hierarchy*

-
- Level 1: Fortran IV Main Programs. These programs are written in Fortran IV and represent the bulk of the software developed for the Rapid Scan system. They contain most of the data acquisition algorithms and all data reduction algorithms.
- Level 2: Fortran IV Subroutines. These subroutines are called by the main Fortran programs and provide the ability to do repetitious operations without lengthy main programs.
- Level 3: Library Programs. These are system programs provided by the computer manufacturer in Fortran, assembly, and machine code. They control system peripherals such as printers and video terminals, and provide support for the X-Y plotter programs.
- Level 4: Assembly Code Programs. These are subroutines written in assembly language by programmers of The Pennsylvania State University, Coal Research Section to provide specific functions such as rapid address to the analog-to-digital converter. They are called by both level 1 and level 2 programs.
-

*Listed in order of increasing complexity.

PATTERN OF TRAVERSES ON BRIQUETTE SURFACE

SAMPLING PATTERN ON COAL PARTICLES

Figure 4. THE SAMPLING PATTERN FOR DATA ACQUISITION

the array element and reflectance represented by the position of the element in the array. The data array is written on magnetic media at the conclusion of the analysis and stored for later manipulation.

Several experiments were performed to evaluate the potential to process the data sequence as it was accumulated, or prior to its being stored in the data array. The conceptual design of this type of data processing is illustrated in Figure 5. As the image of the microscope object moves across the sensing spot, interfaces between high, intermediate, and low reflecting material are encountered. The reflectance readings recorded while the boundary passes under the sensing spot are not representative of either material; hence, they are recorded as "misplaced readings" in the data array. In order to reject these readings, an algorithm was developed which would scan a data sequence of several thousand readings and eliminate boundary areas using a so-called plateau-slope-plateau logic. Reflectance readings on a single homogeneous phase form a plateau in the data sequence (area A, Figure 5). As the boundary between two phases moves under the sensing spot, the data assumes a slope (positive or negative, depending on the direction of reflectance change) where magnitude is proportional to the difference in reflectance between the two phases (area B, Figure 5). The same kind of sequence is repeated each time a boundary is encountered. The processing algorithm was designed to search a string of data and locate all areas where the slope exceeded a preset threshold level. Where this threshold was exceeded, these readings were removed and stored in a different data set.

The successful application of this type of data processing is limited severely by several factors. The first of these is the

Figure 5. THE PLATEAU-SLOPE-PLATEAU SIGNAL RESPONSE TO SCANNING ACROSS REFLECTANCE BOUNDARIES.

complexity of the data sequence, owing to the presence of many fine coal particles (less than 50 microns) in the briquetted pellet.

Figure 6 shows a data sequence and its relationship to a photomosaic of the typical briquetted coal surface from which it was drawn. Area A represents a portion of the scan, encountering a large number of fine coal particles suspended in the epoxy matrix. Using a plateau-slope-plateau type of algorithm, all of these readings would be eliminated. Removal of the less than 50-micron material, during sample preparation, would alleviate this problem. However, size analyses of four coals prepared following the standard method (See Figure 7), showed that up to six percent (by weight) of the sample reported to the minus 50-micron fraction. Furthermore, microscopic analysis of this material indicated that it was enriched in inertinite group macerals, particularly fusinite. Thus, removal of the fine-size fraction through sample preparation or data processing algorithms could significantly bias the results of the analysis.

The second factor limiting the effectiveness of this type of data processing is the presence of cracks in coal particles. An algorithm of this type needs to be able to distinguish between a liptinite maceral and a crack in a coal particle. The presence of the fine-grained liptinite macerals (most spore exines are less than four microns in thickness) in coal particles produces a data sequence pattern very similar to the data sequence produced by the crack shown in area B of Figure 6. It has been nearly impossible to separate epoxy filled cracks from liptinite in coals of high volatile bituminous rank, where the liptinite reflectance is quite close to that of the epoxy.

Figure 6. A COMPARISON OF SIGNAL RESPONSE TO SCANNING ACROSS VARIOUS COAL BRIQUETTE COMPONENTS (after Davis and Vastola, 1976)

Higher rank coals, in which the liptinite reflectance is separated significantly from the epoxy reflectance, are simpler to process.

Finally, the presence of many fine-sized components in coals (such as micrinite, fusinite with bogen structure, and clays) tend to complicate any type of data sequence processing. Their fine size greatly increases the probability of recording interface reflectance readings (see Section IV). Thus, data processing which removes these interface readings could bias any results toward the large particle-sized components.

Owing to the problems noted above, the Rapid Scan system does no processing of the data sequence while it is being acquired. Rather, all the readings are stored in array form, and unwanted information is removed during later data analysis. If one could produce briquetted pellets with reduced amounts of fine particles, few particle cracks, and low coal densities it might be possible to employ a type of data processing algorithm (Liscinsky and Vastola, 1980); however, with current sample preparation techniques this appears unlikely.

2. Sample Preparation

Samples scanned on the Rapid Scan systems generally are briquetted coal pellets, 2.50 centimeters in diameter, prepared according to ASTM-Standard D2797-72 (ASTM, 1979). In addition to adhering to the requirements of the standard, three additional areas of sample preparation quality control are implimented (Figure 7). The first of these involves standardization of the method used in reducing the sample particle size to minus 840-microns (20 mesh, U.S. sieve size). As will be discussed in Section IV, particle size controls the number of

coal-epoxy reflectance boundaries in a scan of a given length. The Rapid Scan system of reflectogram data interpretation employs an algorithm to remove these interface readings. In order for the algorithm to work consistently, particle size for different samples of a given coal rank must be relatively constant. Careful stage crushing with a minimum of five steps has been found to produce relatively constant particle size, even with coals of different friability.

The second control involved the ratio of coal to epoxy in the briquetted sample. In order to remove reflectance readings on epoxy from the reflectogram, it is necessary to estimate the volume percent of epoxy on the sample surface. A combined approach of standardizing the epoxy-coal ratio during sample preparation and, when critical, measuring the actual ratio in the pellet, yields acceptable results.

Standardization is achieved by the preparation technician using constant volumes of coal and epoxy, regardless of coal rank. In order to test the consistency of the sample preparation techniques currently used at The Pennsylvania State University, Coal Research Section, 12 pellets from four different coals were selected and analyzed for their epoxy content by point counting. A minimum of 500 points were counted for each pellet and the resulting data are shown in Table 5. As can be seen from these results, the variation in epoxy content for different pellets from the same sample is relatively small (generally, ± 1 percent). The mean epoxy content for three of the four samples also is relatively constant, around 23 percent. However, PSOC-1101, which has a significantly higher epoxy content (27 percent), would not be processed accurately by the reflectogram data-reduction algorithms. Thus, standardization of epoxy contents in briquetted pellets seems possible if reasonable care

Table 5

Comparison of Epoxy Contents of Twelve Pellets

Sample Number	Number of Counts on Coal	Number of Counts on Epoxy	Volume Percent Epoxy
PSOC-1137-1	390	123	24%
PSOC-1137-5	428	114	21%
PSOC-1137-3	398	119	<u>23%</u>
			$\bar{X} = 23\%$
PSOC-1101-2	378	133	26%
PSOC-1101-1	410	159	28%
PSOC-1101-6	415	153	<u>27%</u>
			$\bar{X} = 27\%$
PSOC-1099-1	447	134	23%
PSOC-1099-5	442	147	25%
PSOC-1099-2	403	120	<u>23%</u>
			$\bar{X} = 24\%$
PSOC 1082-2	438	132	23%
PSOC-1082-3	450	125	22%
PSOC-1082-5	431	129	<u>23%</u>
			$\bar{X} = 23\%$

is exercised during sample preparation. However, if coals are to be prepared routinely for Rapid Scan analysis, more care will be necessary to eliminate samples such as PSQC-1101.

A second method of determining the epoxy content in the coal briquette is to weigh the coal used in the briquette, and weigh the final product. The weight of the epoxy is calculated by difference, and density values are assigned to the coal and the epoxy for volume calculations as follows:

$$W_E = W_B - W_C \quad (2)$$

where,

W_E = the weight of the epoxy,

W_B = the weight of the finished briquette, and

W_C = the weight of the coal used in the briquette. Also

$$V_E = W_E / D_E \quad (3)$$

$$V_C = W_C / D_C \quad (4)$$

where

V_E = the volume of epoxy in cc,

V_C = the volume of coal in cc,

D_E = the density of the epoxy (1.20 g/cc*), and

D_C = the density of the coal (1.35 g/cc**). Also

$$P_E = [V_E / (V_E + V_C)] \times 100 \quad (5)$$

$$P_C = [V_C / (V_C + V_E)] \times 100 \quad (6)$$

where,

P_E = the percentage of epoxy in the sample, and

P_C = the percentage of coal in the sample.

*provided in product specifications for APCO R313 Epoxy.

**An experiment was performed to test the sensitivity of the calculation of P_E to ranges in D_C . A one percent change in P_E was noted when D_C was allowed to vary from 1.25 to 1.45; thus, an average value of 1.35 was chosen to simplify the calculation.

This procedure was checked against volumetric data derived by point-count techniques. Five briquetted samples were prepared using a constant weight of coal and varying the weight of the epoxy. The epoxy content (by volume) was then calculated using equations 2-6, and these data were compared to the point-count analyses of the five samples (Table 6). The results indicate that this procedure is valid for accurate estimates of epoxy in coal briquettes. This estimate then can be used as an input parameter to further refine the reflectogram data reduction algorithm.

The final consideration necessary for the preparation of briquetted samples for automated reflectance analysis is the addition of an opaquing agent to the epoxy. The epoxy used at The Pennsylvania State University, Coal Research Section, is APCO R313 with hardener B, produced by Applied Plastics Company, Incorporated. When hard, this epoxy is translucent, which leads to problems with subsurface particle flare. High reflecting coal or mineral particles, which are present just below the polished epoxy surface, will reflect back some of the light penetrating the translucent epoxy. This causes the photometer to record reflectance readings considerably higher than the reflectance of the epoxy surface alone. In order to limit this problem, an opaquing agent, PP-100 Black Pigment Paste (also produced by Applied Plastics

Table 6
Comparison of Calculated Epoxy Contents Versus Analyzed Epoxy Contents

Sample Number	Wt. of Coal, g	Wt. of Pellet, g	Wt. of Epoxy, g(1)	Volume of Coal, cc(2)	Volume of Epoxy, cc(3)	Volume % Epoxy (4)	Volume % Epoxy (5)
VOL-1	5.0	9.3	4.3	3.70	3.55	49%	48%
VOL-2	5.0	7.5	2.5	3.70	2.08	36%	37%
VOL-3	5.0	8.2	3.2	3.70	2.68	42%	42%
VOL-4	5.0	6.5	5.1	3.70	1.25	25%	25%
VOL-5	5.0	6.1	1.2	3.70	0.93	20%	21%

(1) Calculated from equation (2).

(2) Calculated from equation (4).

(3) Calculated from equation (3).

(4) Calculated from equation (5)

(5) Determined by point count.

Company, Incorporated), is added to the epoxy. This agent considerably reduces the subsurface flare and leads to more uniform readings for the epoxy, without greatly increasing the magnitude of the epoxy reflectance.

B. Configuration of The Pennsylvania State University
Rapid Scan Version I System

The first Rapid Scan system was set up at The Pennsylvania State University, Coal Research Section, in the Fall of 1972. It was assembled from existing hardware, most of which had been purchased over a number of years for a variety of purposes. The overall configuration of this first system (Figure 8) was not unlike the following versions except that the later possessed improved hardware and software elements. The microscope portion of the equipment consisted of a Leitz MPV-I reflectance photometer, equipped with a modified Femco AMEDA stage-drive unit (Figure 9a). The Femco AMEDA stage drive was re-gearred to enable a top scanning speed of 1600 microns per second. It also was equipped with microswitches which signalled the computer to stop taking data when the stage was moving between traverses.

The computer was a Digital Equipment Corporation PDP-9 discrete-transistor computer with 4000 bytes of core memory (Figure 9b). The interface between these two elements was an electrometer, low-pass filter designed in house (Hartmann, 1976). This device provided a voltage signal to the analog-to-digital converter on the PDP-9, and also filtered all noise above the sampling frequency to improve the signal-to-noise ratio.

Figure 8. THE HARDWARE CONFIGURATION OF THE RAPID SCAN VERSION I SYSTEM

a. Microscope-Photometer System

b. DEC PDP-9 Computer

Figure 9. THE EQUIPMENT USED IN THE RAPID SCAN VERSION I SYSTEM

Although the system mainly was employed to characterize pyrite in coal, early experiments (Davis, 1975) indicated that the coal portion of the reflectogram might contain useful information about gross changes in coal petrography. During 1974-1975, this portion of the reflectogram was expanded and studied in detail. The shape of the reflectograms generated at this time partially was obscured by the presence of many finely spaced spikes on the data array (Figure 10a). As can be seen in Figure 10b, an expanded section of Figure 10a, the spikes appear to have a regular periodicity. When the spacing of the individual array elements containing the spikes was examined it was found that the periodicity was a direct binary multiple, suggesting that the spikes were artifacts of the digitizing process. To test this hypothesis, a smooth, controlled voltage-ramp signal was fed into the analog-to-digital converter and sampled by the computer. The resulting output contained the same periodic data spikes confirming the artifact hypothesis. With this confirmation several digital filtering algorithms were developed to smooth the data and make it more interpretable. These algorithms have the effect of taking any major deviation in the data-slope (i.e. a spike) and proportioning that spike between adjacent data elements. The result is a curve whose shape has not been altered, but which is easier to interpret because it has a smoother profile.

In addition to the spikes on the data, another major shortcoming of the Version I system involved the precision of the reflectance determinations. Figure 11 shows a reflectance distribution (which has a spread of ± 0.12 percent R_0 ($\pm 2\sigma$)) from a 1.006 percent R_0 glass standard. A reflectogram generated from a photometer system with this order of precision would have poor resolution, owing to the smearing

Figure 10. AN EXAMPLE OF AN EARLY RAPID SCAN VERSION I REFLECTOGRAM

Figure 11. THE SPREAD IN REFLECTANCE READINGS FROM A GLASS REFLECTANCE STANDARD (RAPID SCAN VERSION I)

together of closely spaced maceral reflectance populations. The lack of precision of reflectance measurements was attributed to two primary factors: fluctuation of electronics and power supplies, and insufficient photon statistics (Hartmann, 1976). Both areas have been improved in later versions by using better system hardware and software controlled signal integration.

C. Configuration of the Rapid Scan Version II System

The Rapid Scan Version II system incorporated several major hardware improvements over the Version I system. As seen in Figure 12, the DEC PDP-9 computer was superseded by a new DEC PDP-8A computer. Also, the Leitz MPV-I photometer was replaced with a Leitz MPV-II photometer system. The filter, as used in the Version I equipment, was redesigned to incorporate an amplifier inverter, in order to interface with the new computer. The only hardware element remaining from the Version I system was the FEMCO stage drive.

1. Computer Configuration

The new PDP-8A computer (Figure 13), is a much more versatile machine than the old PDP-9. It incorporates a PDP-8A central processor with 32K (thousand) bytes of random access core memory. A FPP8/A floating-point processor is included to enable faster arithmetic manipulations to be made than is possible with complex software routines. Voltage signals from the filter are processed by an AD8-A analog-to-digital converter interfaced with 16 multiplexed input channels. This module has a ten-bit resolution (one part in 1024) and a sample acquisition time of approximately three microseconds. Information input-output is controlled by a KL8-A interface which has a

Figure 12. THE HARDWARE CONFIGURATION OF THE RAPID SCAN VERSION II SYSTEM

**Figure 13. THE DIGITAL EQUIPMENT CORPORATION PDP-8A COMPUTER SYSTEM
USED IN THE RAPID SCAN VERSIONS II AND III**

RS 232 output, a 1200-baud output linked to a high-speed modem and a parallel port interfaced to a microprocessor-based KIM controller. The RS 232 output is controlled by switch to two VT-52 video terminals operating at 3600-baud. The video terminals are the primary operator-input devices to the computer and are used for program development, program control and, to a limited extent, data output. The modem enables a high-speed link to The Pennsylvania State University's IBM 370/3033 computing facility. A number of statistical program packages are available on the IBM 370, and have been used to process data generated by the Rapid Scan.

Three devices are used for magnetic storage of data and programs on the PDP-8A. The first is a RX8 floppy disk system, incorporating two disk-drives, each capable of storing 128K, 12-bit words. The floppy disks routinely are used for system start-up and for back-up data storage. The second magnetic storage system is a RK05 J removable disk pack drive, capable of storing 1.6 million 12-bit words. In addition to having approximately 12 times the storage capacity of a floppy disk, the later system can access the information in about one-tenth the time necessary with a floppy disk. Hence, the RK05 J system is used for all program storage and development, and also as the primary data storage device. The third magnetic storage system is a RK05 F fixed disk-drive with a 3.2 million 12-bit word capacity. This device is used for other computer functions, and is not part of the Rapid Scan configuration. (See Figure 14 for schematic of PDP-8A configuration.)

The KIM controller is a stand-alone microcomputer system based on a 6502 microprocessor manufactured by Commodore Calculator Company. Its function here is to provide information input-output to a number of

Figure 14. THE ORGANIZATION OF THE DIGITAL EQUIPMENT CORPORATION PDP-8A COMPUTER SYSTEM

independent devices. The KIM controller has four 20-milliampere current loop "ports" which can be used for printer or terminal communication, as well as four TTL-(transistor-transistor-logic) level ports which can be used to communicate with other microprocessor systems. In this configuration, one 20-milliampere port is linked to a LA-36 Decwriter II printer, which is used for routine large format printouts and for program source listings. A second 20-milliampere port is dedicated to a Xerox 1700 printer, used for high-quality printouts in an 8½ by 11 inch format.

The final module in the PDP-8A system is a XY8-E plotter control interface. This interface connects to a Houston Instruments Complot DP-1 digital plotter. The plotter, under software control, draws and labels diagrams as well as plotting data arrays. In the Rapid Scan system, its primary use is to plot reflectograms.

2. Microscope Photometer System

In the Version II equipment, the Leitz MPV-I (used in the Version I equipment) was superseded by a Leitz MPV-II photometer system (Figure 15). The MPV-II system is of modular design for maximum flexibility (Figure 16), and utilizes solid-state electronics for optimum signal stability.

The microscope is a Leitz Orthoplan-pol, equipped for reflected light microscopy. Illumination for observation and for measurement is provided by a 100-watt quartz halogen lamp, powered by a stabilized voltage supply. The microscope vertical illuminator utilizes a Berek compensating prism, for minimum polarization distortion. The microscope objective is a 50X Achromatic oil immersion lens, giving an observation

Figure 15. THE LEITZ MPV-II PHOTOMETER AND AMEDA STAGE DRIVE USED IN THE RAPID SCAN VERSION II

Figure 16. THE MODULAR DESIGN OF THE LEITZ MPV-II MICROSCOPE PHOTOMETER SYSTEM

magnification of 625X. Under standard operating procedures, a 546-nanometer filter is inserted below the photomultiplier, in order that reflectance determinations can be carried out in monochromatic light. A variable diaphragm, also located below the photomultiplier is adjusted to an effective measurement spotsize of two microns square. (See Figure 17 for a diagram of the optical arrangement.)

The photomultiplier used in this system is a specially selected EMI 9659B with extended sensitivity range, manufactured by Gemcom Division/Emitronics, Incorporated. During this research, it was discovered that the sensitivity of this type of photomultiplier tube varies substantially. Table 7 shows the supply voltage necessary to reach an overall sensitivity of 200 amperes per lumen for four EMI 9659B photomultiplier tubes. The grid-potential voltage varies from 900 volts to 1320 volts, a variation of approximately 32 percent. The higher voltages necessary to compensate for the poor sensitivity of some of the tubes, resulted in unacceptable signal-to-noise ratios and stabilities. It was found that for automated microscopy, the photomultiplier supply voltage at an acceptable level of sensitivity should be below 1000 volts; hence, tubes C and D listed in Table 7 were unsuitable.

3. Microscope-to-Computer Interface

The interface connection between the microscope photometer and the analog-to-digital converter on the PDP-8A consists of a filter-inverter-amplifier circuit, designed and built in house (Figure 18). The filter is a low-pass circuit similar to the one designed by Hartmann (1976) for the Version I equipment. The purpose of this device

1. Quartz Halogen Lamp
2. Diffusion Filter
3. Berek Illuminator
4. Oil Immersion Objective
5. Sample Briquette
6. Photometer Tube
7. Observation Tube
8. Film Transport
9. Adjustable Diaphragm
10. 546nm. Filter
11. EMI Photomultiplier Tube

Figure 17. THE OPTICAL ARRANGEMENT OF THE LEITZ MPV-II MICROSCOPE PHOTOMETER SYSTEM

Figure 18. THE SCHEMATIC FOR THE FILTER-INVERTER-AMPLIFIER INTERFACE

Table 7

Comparison of Photomultiplier Tube Sensitivity

Tube Number	Overall Sensitivity (1)	Cathode Voltage
A	200	900
B	200	1000
C	200	1140
D	200	1320

(1) Amperes-per-lumen.

is to eliminate detrimental high-frequency noise. The system's electronics have the potential to respond to signal frequencies in excess of 10,000 hertz. The sampling frequency, limited by the speed of the computer, is several orders of magnitude less than the system capability; hence, the filter is designed to pass, unchanged, only signals whose frequency is below 1000 hertz and attenuate all signals above this frequency. The result is a more stable signal, devoid of unused and unwanted high-frequency noise.

The inverter-amplifier circuit provides two functions in this interface. The first is to change the signal polarity of the MPV-II from a positive potential to the negative potential required by the AD8-A analog-to-digital converter. The second is to provide a variable gain control so that small adjustments in system gain can be made, without changing the photomultiplier high-voltage supply. This gain control is useful in standardizing the equipment for reflectance measurement.

4. Evaluation of Hardware Improvements

Owing to the hardware component changes of the Version II system, it was necessary to evaluate the various factors controlling unit operations. This evaluation enabled all systems to be operated at maximum capability for maximum resolution. It also indicated areas where additional modifications in operation could yield further improvements in data acquisition.

The first area investigated was the effect of loss of focus on reflectance determinations. During the scanning of briquetted coal samples, minor changes of focus occur due to the surface relief and non-level surface alignment. During operation of the system, the operator adjusts the focus by observing the image of the field diaphragm during scanning. This procedure is effective for correcting focusing errors of five microns and greater.

In this experiment reflectance determinations were made on two glass reflectance standards and on a particle of vitrinite. Reflectance readings were taken at five microns, ten microns, 15 microns, and 20 microns above and below the correct focus plane. The results, shown graphically in Figure 19, have been corrected to indicate relative measuring differences by the following equation:

$$X = \frac{V^1 - V}{V} \quad (7)$$

where,

X = the proportional difference between the measured values,

V^1 = the value measured with the sample out of focus, and

V = the measured value with the sample in focus,

so that direct comparisons can be made on materials with differing reflectances. Figure 19 shows that when the plane-of-focus moves above

Figure 19. THE VARIATION IN REFLECTANCE MEASUREMENT DUE TO IMPROPER FOCUS

the object plane, the measured reflectance for all three materials increases. This is due to light from areas surrounding the effective measuring spot passing through the photometer diaphragm and registering on the photomultiplier tube (Piller, 1977, p. 163). When the plane of focus is below the stage plane, the measured reflectance value decreases, because less light reaches the photomultiplier tube. In the case of vitrinite, the decrease is relatively greater, due to its opaqueness. However, in all three cases, a focusing error of five microns results in a measuring error of less than one percent. Thus, errors in reflectance determination due to focusing problems probably are not significant, providing an operator visually maintains focusing during the scan.

The second system variable investigated was the effect of photomultiplier gain on the stability of the system. The gain of any photomultiplier tube is a linear function of the high voltage supply, approximately proportional to the number of dynodes (Piller, 1977, p. 105). In this experiment, three gain settings of 650 volts, 750 volts, and 830 volts were used to test the photomultiplier tube output. The signal transmitted to the computer was adjusted by varying the gain of the interface amplifier so that a constant signal level was maintained, irrespective of photomultiplier gain. The results shown in Table 8 are the summary statistics for distributions of 7500 reflectance measurements taken by the computer from one spot on a glass reflectance standard. These data indicate that no significant degradation in measurement precision (as indicated by $\pm 2\sigma$) can be noted with increasing gain. However, at 830 volts the system is more susceptible to drift, as can be seen in the change of the mean value. As a consequence of

Table 8

The Effect of Photomultiplier High Voltage
on Signal Stability

Photomultiplier High-Voltage	Interface Amplifier Gain	Mean % R_0	2 Times Standard Deviation R_0	Population
650 volts	Maximum	1.006	.02	7,500
750 volts	Mid Range	1.007	.02	7,500
830 volts*	Low	1.010	.02	7,500

*Under these test conditions, this voltage was just below the level of photomultiplier tube saturation.

these experiments, a photomultiplier supply voltage of 725 volts was adopted as the standard operating parameter. Changes in the system gain, for the purpose of calibrating the equipment, are carried out by adjusting the interface amplifier.

The third operating variable investigated was the effect of luminous flux on signal quality. Luminous flux is a measure of the amount of energy per unit time, emitted or received as light. In this experiment the amperage delivered to the quartz halogen lamp was used as a relative measure of luminous flux. Four amperage levels ranging from 5.5 to 8.5 were used to give a range of luminous flux. After setting the lamp amperage, the system was allowed to stabilize for 30 minutes. Following the stabilization period, 7500 reflectance readings were taken by the computer from one spot on a glass reflectance standard, and the summary statistics calculated for the resulting distribution. The lamp amperage then was increased to the next test

level and the procedure repeated. As in the photomultiplier high-voltage experiment, the interface amplifier was used to compensate the photometer output to a constant voltage. The data in Table 9 shows that, with increasing luminous flux, the population mean stabilizes (indicating less drift) and the spread of the reflectance distributions decreases significantly at higher light levels. The overall effect of these changes (as seen in Figure 20) is an improvement in the precision of the reflectance determination. Therefore, as a standard operating procedure the lamp power supply is set at 8.5 amperes. This is slightly above the rating for a 100-watt quartz halogen lamp, and leads to a shorter life span; however, the gain in precision of measurement justifies the more frequent lamp replacement.

The final operating variable investigated was the effect of signal averaging on the precision of reflectance measurement. The precision of a population mean generally can be improved by increasing the size of the sample population. Griffiths (1967, p. 22) suggested one way of evaluating the effect of sample size on the precision of the population mean is to compare the standard error of the mean at larger and larger sample sizes. Thus, in this experiment all the operating variables established on the foregoing experiments were held constant, and the computer was programmed to sample the signal from one to 512 times, and record the average reflectance value. The program accumulated 7500 of the pre-averaged values and calculated the summary statistics (Table 10), including the standard error of the mean as follows:

$$\sigma_{\bar{X}} = \sigma/\sqrt{N} \quad (8)$$

Figure 20. THE REFLECTANCE DISTRIBUTIONS FROM A GLASS REFLECTANCE STANDARD AT TWO LEVELS OF PHOTON FLUX

Table 9
Effect of Luminous Flux on Signal Quality

Lamp Amperage	Mean % R_0	Standard Deviation, % R_0	Population
5.5	1.013	.032	7,500
6.5	1.009	.020	7,500
7.5	1.006	.014	7,500
8.5	1.006	.010	7,500

Where,

$\sigma_{\bar{X}}$ = the standard error of the mean,

σ = the standard deviation of the distribution, and

N = the number of readings in the sample.

Figure 21 shows a plot of the standard error of the mean versus the number of pre-averages in each reading. The standard error of the mean decreases an entire order of magnitude from one to 128 pre-averages, and then decreases only slightly from 128 to 512 pre-averages. These results indicate that increasing the number of pre-averages above 128 would result in little gain in measurement precision.

The preceding four experiments (focus, photomultiplier tube gain, luminous flux and signal averaging) show that for this photometer system, precision of measurement is affected mainly by luminous flux and signal pre-averaging. Luminous flux is optimized in the system by operating the lamp at its maximum output and adjusting the microscope optical system for the optimum light passage through the effective measuring spot. Further increases in precision of the version II system

Table 10
The Effect of Pre-Averaging on Signal Quality

Number of Averages	Time for Acquisition of One Value (1)	Mean % R_0	Standard Deviation % R_0	Population	Standard Error of The Mean. (2)
1	1.3	1.006	.026	7,500	3.00
2	1.4	1.006	.027	7,500	1.80
4	1.5	1.006	.027	7,500	1.30
8	1.6	1.006	.022	7,500	0.90
16	1.9	1.006	.021	7,500	0.60
32	2.4	1.006	.018	7,500	0.40
64	3.4	1.006	.014	7,500	0.20
128	5.3	1.006	.011	7,500	0.10
256	9.0	1.006	.009	7,500	0.06
512	16.0	1.006	.005	7,500	0.03

(1) Milliseconds.

(2) Calculated from equation (8), $\times 10^{-4}$.

Figure 21. THE RELATIONSHIPS BETWEEN THE NUMBER OF PRE-AVERAGED REFLECTANCE READINGS, THE STANDARD ERROR OF THE MEAN, AND TIME

by increasing the luminous flux would have entailed using a higher wattage lamp source, which was not then available commercially. Therefore, at that time, the only way to increase further the precision of the system would have been to increase the number of signal pre-averages. However, as shown in Figure 21, the time necessary for pre-averaging increases markedly when greater numbers of pre-averages are accumulated. One must bear in mind that the Version I and II systems used a modified Femco AMEDA stage-drive unit. This unit moved the stage continuously using standard synchronous motors. Thus, stage speed had to be matched with the signal acquisition time so that the sample would not have moved more than a fraction of one micron during the time necessary for the reading. Table 11 shows the distance the stage object moved during the time necessary to perform each of the pre-averaging operations, for various stage-drive speeds. The dotted line shows the relationship between the number of pre-averages and the stage-drive speed for a sample movement of 0.25 microns. If, as indicated previously, 128 pre-averages were selected as the minimum number desirable, then for a stage movement of 0.25 microns, the stage-drive speed would need to be set at 50 microns per second. At this scan speed, it would take approximately 35 minutes to complete a 100-millimeter scan. Owing to factors such as drift of the photomultiplier output and the need for a truly rapid analysis, it was deemed desirable to complete the scan within a time closer to 15 minutes. Thus, in the Version II equipment, a compromise in both the number of pre-averages and the amount of stage movement was accepted at 32 pre-averages and 0.48 microns of movement, respectively. These values certainly were not optimal operating parameters, and the need to improve upon them led to the development of the Version III system.

Table 11

Distance of Stage Movement During Data Acquisition at Various Stage-Drive Speeds*

Number of Pre-Averages	Stage-Drive Speed (microns per second)					
	25	50	100	200	400	800
1	0.03	0.07	0.13	0.26	0.52	1.04
2	0.04	0.07	0.14	0.28	0.56	1.12
4	0.04	0.08	0.15	0.30	0.60	1.20
8	0.04	0.08	0.16	0.32	0.64	1.28
16	0.05	0.10	0.19	0.38	0.76	1.52
32	0.06	0.12	0.24	0.48	0.96	1.92
64	0.08	0.17	0.34	0.68	1.36	2.72
128	0.13	0.26	0.53	1.06	2.12	4.24
256	0.22	0.45	0.90	1.80	3.60	7.02
512	0.44	0.80	1.60	3.20	6.40	12.80

*All values in microns.

D. Configuration and Operation of the

Rapid Scan Version III System

The Rapid Scan Version III system used all the same hardware components of the Version II system, with the exception of the Femco stage drive (Figure 22). As was discussed in the previous section, the precision of the reflectance values recorded while scanning were limited by the continual movement of the Femco stage drive. Thus, the Version III system utilizes a Leitz digital stepping stage (Figure 23).

The digital stepping stage moves in discrete increments of 2.5 microns and can be programmed to move to a sample location, stop while the signal is integrated as long as is deemed desirable, and then move again. In order to control the stage, the computer must be capable of providing TTL-(transistor-transistor-logic-) level signal pulses under program control. The PDP-8A, as it originally was configured, did not contain the proper interface module to provide this type of signal. Thus, a communications interface system was developed utilizing the PDP-8A's microprocessor-based KIM controller and a second KIM microprocessor. Under this system a signal is transmitted under a level-four program from the PDP-8A to the KIM controller. The KIM controller interprets the signal, and reroutes it out of one of its four TTL-level ports to a second KIM microprocessor. The second KIM microprocessor is programmed, by cassette tape, to interpret the signal and send the stage-drive controller the proper combination of four TTL-level signals to move the stage one step in any of eight directions (Figure 24). When the stage-movement is complete, the KIM microprocessor signals the PDP-8A, through the KIM controller, that it is finished and ready for another instruction. This type of control is complex and time consuming.

Figure 22. THE HARDWARE CONFIGURATION OF THE RAPID SCAN VERSION III SYSTEM

Figure 23. THE LEITZ MPV-II PHOTOMETER AND DIGITAL STEPPING STAGE USED IN THE RAPID SCAN VERSION III SYSTEM

Figure 24. THE CONTROL LOGIC FOR THE ACTIVATION OF THE DIGITAL STEPPING STAGE BY THE PDP-8A

A much more efficient operation could be performed with the appropriate interface module plugged directly into the PDP-8A to eliminate the two microprocessors.

By moving the stage in discrete increments, it is possible to increase the signal pre-averaging time. In the Version II equipment, utilizing the Femco stage drive, the maximum number of averages was 32 with 0.48 microns of stage movement. With the stepping stage, the number of pre-averages is increased to 128 (approximately five milliseconds of integration time) with almost no stage movement. Total time for one reading is approximately 15 milliseconds, allowing ten milliseconds for the stage to come to a stop after moving the 2.5-micron step. Thus, a standard scan of 125 millimeters takes approximately 12 minutes, compared to approximately nine minutes for the Version II system. This time penalty is offset in the improved precision of each reflectance measurement. (See Figure 21.)

Operation of the Version III equipment begins by placing a RK05 J disk pack and a Fortran system floppy disk into the appropriate drive mechanisms. The floppy disk allows initial startup control of the PDP-8A. After startup, system control is transferred to the RK05 J, which contains all the necessary system operation programs and all the Rapid Scan operating programs.

To begin a Rapid Scan analysis, the basic data acquisition program, PETRO6, is loaded into the computer, along with the name of the future data output file. PETRO6 (Appendix I) is a level-one program, written in Fortran IV, containing nine basic program modules. It calls two level-two subroutines (Table 4) (MOVE2 and MOVE3) and two level-four subroutines (SETADC and FASTAD). Both level-two subroutines call

level-four subroutines (PUTB and GETB). (See Figure 25, for the programming structure.) Fortunately, for the operator, all the program co-ordination is handled by the PDP-8A operating system.

Once the computer finishes loading the programs, a series of questions appear to the operator on the VT52 video terminal (Table 12). These questions allow the operator to put information into the computer about the sample and the analysis operating parameters. At the completion of the scan, this information is stored on magnetic disk with the data accumulated during the analysis as an output file. Thus, in later data processing programs, the analysis conditions can be displayed along with the analytical results.

After the operating information is entered, the computer proceeds to the third program module, which is used for standardizing the microscope. The primary glass reflectance standard is placed on the microscope and the computer reads the photomultiplier output, calculates the reflectogram full-scale reflectance based on the reflectance value the operator entered for the primary standard, and displays this value on the VT52 video screen. (See Figure 26 for a flow chart.) The operator can adjust the gain on the interface amplifier and vary the full scale reflectogram reflectance value. After every 20 reflectance readings, the computer asks the operator whether or not he is satisfied with the value. If the operator answers "no," the computer takes 20 more readings and repeats the question. If the operator answers "yes," the computer proceeds to the next program module. Once the system is standardized, all reflectance readings from the photometer are assigned properly to the first 1000 array elements in the data array. Any reflectance reading which exceeds the full scale standardized value is accumulated in the 1000 to 1024 array elements.

Figure 25. THE PROGRAMMING STRUCTURE OF PETRO6

Figure 26. THE FLOW CHART OF THE STANDARDIZATION PROGRAM MODULE

Table 12

Example of Data Entered at the Beginning of PETR06

Computer Question	Operator Response
ENTER SAMPLE ID (10 CHAR. MAX.)	PSOC-1099
ENTER FULL-SCALE REFLECTANCE (REAL)	2.0
ENTER PRIMARY STANDARD VALUE IN %	1.006
ENTER SECONDARY STANDARD VALUE IN %	0.533
ENTER SPOTSIZE IN MICRONS (REAL)	2.5
ENTER NUMBER OF READINGS	50000
ENTER ANY COMMENTS-18 CHAR. MAX.-	PITTSBURGH SEAM

The next module allows the operator to enter the scanning information into the program. The operator responds to two questions, the first asking the number of readings to be taken per traverse, and the second asking the number of steps between traverses. This information ensures that the stage does not move beyond the edge of the sample briquette during scanning.

The fifth and sixth program modules control the scanning of the pellet and the data acquisition. They consist of two level-two subroutines, MOVE2 and MOVE3, which are called alternately by the main program. (See Figure 27, for a flow chart.) MOVE2 controls moving the stage in the X direction, reading the analog-to-digital converter, and incrementing the appropriate data array element. MOVE3 controls the stage motion between X traverses in the Y direction. Between each X traverse the VT52 video screen displays the number of

Figure 27. THE FLOW CHART OF THE DATA ACQUISITION PROGRAM MODULE

traverses completed and the number of the traverse currently being scanned. When the number of data points specified by the operator have been sampled, the program proceeds to the next module.

The seventh module in this program contains the algorithms for digital filtering of the data array. The first algorithm scans the data array and locates adjacent elements which have an absolute numerical difference in excess of eight. When such a difference is found, it is proportioned equally between the two adjacent data elements. The second algorithm in this module also smooths the data by applying a three element weighted-running-average. The effect of passing the data array through these algorithms is to smooth out any noise spikes, without distorting the shape of the reflectogram.

The final module in the program controls the output of data to a storage file. The initial sample information and analysis parameters are stored in a 20 real-element array, set in an 5F10.3 format. The reflectance data gathered during the scan is stored in a 1024 integer-element array, set in a 10I8 format. Both arrays are written sequentially on a floppy disk under one output file name and require 23 blocks of storage space.

Once the data have been stored on floppy disks, they are available for several processing operations. The simplest is the plotting of a reflectogram of the data array. This procedure is accomplished by using the program PLOTV1 (Appendix II). PLOTV1 is a level-one Fortran IV plotting program, written specifically for plotting Rapid Scan reflectograms. It utilizes a series of level-three subroutines provided by Digital Equipment Corporation. The program first draws, scales, and labels the plot axes. It then reads the data from the

storage disk into a 1024 integer-element data array. After checking the array for scale, it plots the first 1000 data elements across the diagram's horizontal axis in a point-to-point fashion. At the completion of plotting the data, the plotter labels the diagram with the sample number and proceeds to the origin of the next plot.

From the reflectogram, the operator can deduce qualitative and some quantitative data. Qualitatively, the reflectogram immediately shows the operator whether the sample was a single coal or a blend of several coals. It also allows the operator to speculate on the gross petrographic composition of the sample. (See Section V, for the reflectogram interpretations made early in the development of the system.) The only quantitative information which can be determined directly from the reflectogram plot is the mean average random vitrinite reflectance (MARR). It can be determined by locating visually the mode of the vitrinite peak and comparing it to the reflectogram scale. Other quantitative information about the distribution of vitrinite reflectance values and petrographic composition are determined by the PETAN program and are discussed in Section V.

IV. DISCUSSION OF RESULTS

A. Early Interpretations

Researchers began investigating the potential for gathering petrographic information from the Rapid Scan in 1973 (Spackman et al., 1973). Davis (1975) reported that Rapid Scan "reflectance spectra" indicated gross changes in sample petrographic composition; however, it was not until 1976 that the reflectogram was interpreted in more than a semiquantitative manner.

Davis and Vastola (1976) showed that a Rapid Scan Version I reflectogram contained quantifiable information about coal rank, as well as being an indicator of petrographic composition. They showed that with increasing coal rank the vitrinite peak on the reflectogram increases in reflectance (Figure 28). The mean average random vitrinite reflectance was determined for ten coals by visually locating the mode of the vitrinite peak, and reading the corresponding reflectance from the ordinate. The reflectance values then were calculated to maximum reflectance by the addition of a correction factor. Comparison of Rapid Scan determined mean maximum reflectance values to those determined conventionally in polarized light proved encouraging for these provisional experiments.

Davis and Vastola also graphically showed how a Rapid Scan reflectogram indicates a coal's petrographic composition. They scanned three lithotype samples from a single coal seam, the first of which (PSOC-4) was a sample rich in vitrinite (vitrinite greater than 70 percent). The second lithotype (PSOC-2C) was a clarain; and the third (PSOC-2D) was a durain. The reflectograms of these samples,

Figure 28. REFLECTOGRAMS OF A SERIES OF COALS RANGING IN RANK FROM SUBBITUMINOUS B TO SEMIANTHRACITE (from Davis and Vastola, 1976)

along with bar graphs showing their petrographic compositions are shown in Figure 29. Davis and Vastola reported that as the vitrinite content in the sample decreases, the height of the vitrinite peak decreases. Conversely, as the inertinite content of the samples increases, the area of the reflectogram higher in reflectance than the vitrinite peak increases. The encouraging results shown in these experiments stimulated further research into the quantification of vitrinite reflectance parameters and reflectogram response to changing sample petrographic composition.

B. Reflectogram Reproducibility

If one visually compares the reflectograms from three different briquetted pellets of the same coal sample (Figure 30), it is apparent that Rapid Scan reflectograms appear reproducible. Early recognition of this characteristic was a primary factor prompting a continuation of the research work.

Rapid Scan analyses on the Version I equipment generally utilized scan lengths of 80 centimeters or 400,000 data points (Davis and Vastola, 1976). However, using the sample integration times employed on the Version II and Version III systems, such a scan would have taken a prohibitive amount of time. (Approximately 1.6 hours for the Version III system.) Therefore, an experiment was designed to determine the optimal number of data points which would ensure sufficient sampling of the briquetted pellet in a reasonable time period. Three coal samples (PSOC-127, PSOC-104 and PSOC-1099) were analyzed on the Version III system at sample point densities of 1000, 10,000, 25,000, 50,000, and 100,000 points per sample surface.

Ex., Exinite; Vit., Vitrinite; In., Inertinite

Figure 29. THE COMPARISON OF REFLECTOGRAMS FROM COMPOSITIONALLY DIFFERENT LITHOTYPES TO VISUAL PETROGRAPHIC RESULTS (from Davis and Vastola, 1976)

Figure 30. THE REFLECTOGRAMS OF THREE SUBSAMPLES
OF PSOC-1099

Replicate analyses were performed at each sample point density. The replicate reflectograms were compared to each other and the total proportional difference was calculated by the following equation:

$$C_D = \frac{1000}{\sum_{i=1}^{} (|x^1 - x|)} / NR \quad (9)$$

where,

C_D = the total proportional difference,

x^1 = the array element (i) of scan 1,

x = the array element (i) of scan 2, and

NR = the total number of data points.

The total proportional difference is a numerical value which indicates the relative variation in the entire distribution between replicate analyses. As seen from Figure 31, this value decreases significantly (approximately five-fold) from point densities of 1000 to 50,000. However, from point densities of 50,000 to 100,000, the decrease is much less pronounced. These results indicate that a sample point density of 50,000 points on the briquette surface is sufficient to detect the important variations in the distribution of the sample reflectance; accordingly, for the Version III system a point density of 50,000 points (or a scan length of 125 millimeters) was selected as the standard operating procedure. In samples where the reflectogram shape is particularly critical, duplicate analyses are made using two briquettes and checked for reproducibility.

The reproducibility obtained from multiple briquettes of the same coal sample was checked once the number of points necessary for replication had been standardized. As was noted visually from Figure 30,

Figure 31. THE EFFECT OF INCREASING SAMPLE POINT DENSITY ON THE TOTAL PROPORTIONAL DIFFERENCE

reflectograms from the same sample appeared reproducible; however, in order to test this impression objectively, it was necessary to explore various types of inferential statistical techniques. Owing to the bi-modal nature of reflectograms and a desire to test overall curve shape, standard parametric statistical tests were considered inappropriate. From the various nonparametric techniques which can be used to test the shape of histograms, the Kolmogorov-Smirnov two-sample test was chosen for testing reflectograms. This test is used to determine if two samples have been drawn from the same parent population. It is sensitive to any difference between the distributions, such as location, dispersion, and skewness (Siegel, 1956, p. 127). The test makes no statement about the nature of the difference between the distributions, and hence, is useful only as a check on agreement and not for quantifying the differences.

The Kolmogorov-Smirnov test operates on cumulative frequency distributions of the same size interval for both samples. It compares the distributions, class by class, and focuses on the class which shows the greatest deviation as follows:

$$\text{Cum 1 } (x) = \text{Tot 1}/\text{Num 1} \quad (10)$$

$$\text{Cum 2 } (x) = \text{Tot 2}/\text{Num 2} \quad (11)$$

where,

(x) = the individual class interval,

Cum 1 and Cum 2 = the observed cumulative step value for the
first and second samples,

Tot 1 and Tot 2 = the total number of readings in this class,
plus all the readings in the previous classes
for sample 1 and sample 2, and

Num 1 and Num 2 = the total number of readings in sample 1
and sample 2;

and

$$D = \text{Maximum (Cum 1 (x) - Cum 2 (x))} \quad (12)$$

where,

D = the maximum difference between the cumulative steps.

The observed maximum deviation (D) value is tested for significance against D values calculated from the sampling distribution of D, at various probability levels. A D which exceeds the critical value at a given probability level will lead to the rejection of the null hypothesis, that the two distributions were drawn from the same population.

To check reflectograms with the Kolmogorov-Smirnov test a computer program (KOL03) (APPENDIX III) incorporating equations 10, 11 and 12 was written. Because of the decreasing power-efficiency of this test with increasing sample size (Siegel, 1956, p. 136), the program collapses the class interval on the reflectograms to 20 reflectance classes.

Sample group A in Table 13 shows the testing of replicate analyses on three different briquettes from coal samples PSOC-1099 and PSOC-1137. None of the six tests show significance at a probability level of 0.05, and therefore, the null hypothesis that the replicate runs were drawn from the same population cannot be rejected. These results tend to confirm that 50,000 data points are sufficient to sample the briquette surface.

Sample group B in Table 13 shows the results of testing the 12 possible interactions between the six scans for each of the two samples in sample group A, as well as six interactions for three

Table 13

Results of Kolmogorov-Smirnov Test for Reflectogram Reproducibility

Sample Group	Samples Tested Sample 1 X Sample 2*	Class Where Max. D Observed	Max. D Observed	Critical D Alpha = .05	Not Significant at .05	Test		
						.05	.01	.001
A	1099 (2A) X 1099 (2B)	6	.01	.06	X			
A	1099 (3A) X 1099 (3B)	6	.01	.06	X			
A	1099 (5A) X 1099 (5B)	6	.02	.06	X			
A	1137 (1A) X 1137 (1B)	11	.04	.06	X			
A	1137 (3A) X 1137 (3B)	11	.04	.06	X			
A	1137 (5A) X 1137 (5B)	11	.03	.06	X			
B	1099 (2A) X 1099 (3A)	8	.05	.06	X			
B	1099 (2A) X 1099 (3B)	8	.05	.06	X			
B	1099 (2A) X 1099 (5A)	1	.05	.06	X			
B	1099 (2A) X 1099 (5B)	1	.04	.06	X			
B	1099 (2B) X 1099 (3A)	8	.05	.06	X			
B	1099 (2B) X 1099 (3B)	8	.05	.06	X			
B	1099 (2B) X 1099 (5A)	1	.04	.06	X			

Table 13 (Continued)

B	1099 (2B) X 1099 (5B)	1	.05	.06	X	
B	1099 (3A) X 1099 (5A)	1	.06	.06		X
B	1099 (3A) X 1099 (5B)	1	.06	.06		X
B	1099 (3B) X 1099 (5A)	1	.06	.06		X
B	1099 (3B) X 1099 (5B)	1	.06	.06		X
B	1137 (1A) X 1137 (3A)	11	.05	.06	X	
B	1137 (1A) X 1137 (3B)	11	.02	.06	X	
B	1137 (1A) X 1137 (5A)	1	.03	.06	X	
B	1137 (1A) X 1137 (5B)	1	.04	.06	X	
B	1137 (1B) X 1137 (3A)	11	.04	.06	X	
B	1137 (1B) X 1137 (3B)	11	.03	.06	X	
B	1137 (1B) X 1137 (5A)	1	.03	.06	X	
B	1137 (3A) X 1137 (5A)	11	.06	.06	X	
B	1137 (3A) X 1137 (5B)	11	.04	.06	X	
B	1137 (3B) X 1137 (5A)	11	.03	.06	X	
B	1137 (3B) X 1137 (5B)	11	.04	.06	X	

Table 13 (Continued)

B	1101 (1A) X 1101 (2A)	8	.03	.06	X
B	1101 (1A) X 1101 (6A)	8	.05	.06	X
B	1101 (2A) X 1101 (6A)	8	.01	.06	X
C	1099 (2A) X 1137 (1A)	9	.51	.06	X
C	1099 (2A) X 1101 (1A)	8	.09	.06	X
C	1137 (1A) X 1101 (1A)	10	.47	.06	X

*The first four digits of the sample number refer to the PSOC sample number and the first character in the brackets refers to the briquette number while the second refers to the scan designation on the briquette.

briquettes of PSOC-1101. The objective of this experiment was to determine if reproducible results could be gained from separate subsamples (the individual briquettes) of the same head sample (i.e. PSOC-1099). As can be seen from the table, 23 of the 27 tests were not significant at a probability level of 0.05, suggesting that reflectograms from subsamples are reproducible. The four interactions that were significant at the 0.05 probability level, gave the maximum D in the first class interval. This class interval represents most of the reflectance readings on the epoxy matrix of the briquetted sample, suggesting that differences in the proportion of epoxy may have led to rejection in these cases.

Sample group C in Table 13 is the test results comparing the interactions of example reflectograms from each coal. In this case the test statistics are all significant at the 0.001 level, indicating that the three reflectograms are significantly different for each of the three coals.

The results from these experiments show that the Kolmogorov-Smirnov two-sample test statistic generally cannot reject reflectograms that have been drawn from the same parent sample. However, it also is important to know if the test can reject reflectograms which are drawn from very similar but different sample populations.

PSOC-1101 and PSOC-1102 are both samples of the Pittsburgh seam from Washington County, Pennsylvania. They were collected from the same mine, however PSOC-1101 is the middle portion of the seam, whereas PSOC-1102 is the lower portion of the seam. The proximate, petrographic, and vitrinite reflectance analyses for the two samples

are fairly similar (Table 14). Thus, a comparison of the reflectograms of these samples by means of the Kolmogorov-Smirnov test should indicate the sensitivity of the reflectogram for discriminating between very similar samples.

Table 15 shows the test results for nine interactions between six briquettes of the two samples. In all cases, the D value was found to be significant at probability levels of 0.01 or 0.001. Therefore, the null hypothesis clearly can be rejected, and one must conclude that the reflectograms were drawn from different populations. This shows that even when scanning two samples as closely related as PSOC-1101 and PSOC-1102, the Rapid Scan is capable of determining that the two samples are, indeed, different.

C. Resolution

The experiments in the preceding sections demonstrated that the Rapid Scan is capable of generating reproducible coal reflectograms. Also, it has been suggested that these reflectograms contained information about coal rank and petrographic composition. However, difficulties in quantifying this information arise from the lack of resolution on the reflectogram of the maceral reflectance populations other than vitrinite.

As discussed in Section I, the different macerals in coal have the inherent characteristics of reflecting differing amounts of vertically incident light. Therefore, if reflectance was the only characteristic of coal macerals, one might expect a coal reflectogram to appear like Figure 32a which shows six discrete reflectance populations. Unfortunately, this figure is a complete fabrication; actual

Table 14
Comparison of Chemical and Petrographic
Analyses for PSOC-1101 and PSOC-1102

Analysis Parameter	Sample Number			
	PSOC-1101		PSOC-1102	
Proximate Analysis (Wt. %)	Dry	DMMF (1)	Dry	DMMF
% Ash	5.43	--	7.72	--
% Volatile Yield	36.13	37.75	35.19	37.32
% Fixed Carbon	58.44	62.25	57.09	62.68
% Total Sulfur	0.83	--	1.89	--
Btu/Lb.	14487	15420	13950	15289
Petrographic Analysis (Vol %, MMF (2))				
% Vitrinite	77.5		74.9	
% "Pseudovitrinite"	4.3		6.1	
% Fusinite	4.7		2.1	
% Semifusinite	5.8		7.9	
% Micrinite	4.1		6.3	
% Macrinite	0.4		0.4	
% Sporinite	2.6		1.5	
% Resinite	0.6		0.8	
Vitrinite Reflectance				
Mean Maximum R ₀	0.90		0.89	

Table 14 (Continued)

$\frac{1}{2}$ VType Table	% of Vitrinite	% of Vitrinite
$\frac{1}{2}$ VType 7.75	--	1%
$\frac{1}{2}$ VType 8.25	8%	9%
$\frac{1}{2}$ VType 8.75	29%	53%
$\frac{1}{2}$ VType 9.25	51%	30%
$\frac{1}{2}$ VType 9.75	12%	7%

(1) Dry Mineral Matter Free.

(2) Mineral Matter Free.

Table 15

Results of Kolmogorov-Smirnov Test for Sensitivity

Sample Group	Samples Tested Sample 1 X Sample 2*	Class Where Max. D Observed	Max. D Observed	Critical D Alpha = .05	Test		
					Not Significant at .05	Significant at .05 .01 .001	
D	1101 (1A) X 1102 (1A)	12	.09	.06			X
D	1101 (2A) X 1102 (2A)	12	.09	.06			X
D	1101 (6A) X 1102 (4A)	13	.14	.06			X
D	1101 (2A) X 1102 (1A)	12	.13	.06			X
D	1101 (6A) X 1102 (1A)	12	.14	.06			X
D	1101 (1A) X 1102 (2A)	12	.08	.06		X	
C	1101 (6A) X 1102 (2A)	12	.08	.06		X	
D	1101 (1A) X 1102 (4A)	13	.14	.06			X
D	1101 (2A) X 1102 (4A)	13	.14	.06			X

*The first four digits of the sample number refer to the PSOC sample number and the first character in the brackets refers to the briquette number while the second refers to the scan designation on the briquette.

Figure 32. A COMPARISON OF AN ARTIFICIAL REFLECTOGRAM
TO AN ACTUAL COAL REFLECTOGRAM

coal reflectograms look like Figure 32b. In Figure 32b there are only two recognizable reflectance populations, those of the epoxy matrix and the vitrinite. Therefore, the question arises of why the five other macerals known to be present in this coal are not manifested in the reflectogram. Before any quantitative reflectogram interpretation could be attempted it was necessary to answer this question and understand the factors which control reflectogram shape and resolution.

A study of coal reflectograms demonstrated that six primary factors control resolution. Of these factors, three (instrumental error, sample preparation, and sampling technique) are dependent factors over which the operator has some control. Three other factors (concentrations of constituents present, reflectance overlap of macerals, and grain size) are independent factors, representative of the complex nature of coal.

The effects on reflectogram resolution due to instrumental errors can be minimized by good operating techniques. The main influence on reflectogram reproducibility is system calibration. Proper calibration of the Rapid Scan is accomplished by using a minimum of two Bausch and Lomb glass reflectance standards as reference points (Table 16). The reflectance of these glasses has been calculated using the Fresnel equation:

$$R\% = \frac{(n_2 - n_1)^2}{(n_2 + n_1)^2} \times 100 \quad (13)$$

where,

R = the reflectance of the standard, expressed as a percentage,

n_2 = the refractive index of the glass at 546 nanometers, and

n_1 = the refractive index of the immersion medium at 546 nanometers.

Table 16
Optical Properties of Bausch and Lomb Glass
Reflectance Standards at 546 nm

Glass (1)	n_e	R_0 ($n = 1.515$) (3)
Bausch & Lomb 689309	1.6945	0.302
Bausch & Lomb 751278	1.7572	0.533
Bausch & Lomb 850324	1.8564	1.006
Bausch & Lomb 915213 (2)	1.9255	1.400
Bausch & Lomb 980222	1.9908	1.815

- (1) These five standards are embedded in a single mount.
- (2) This standard has been found to give a lower reflectance than the calculated value and is not generally used for calibration.
- (3) The refractive index of the Cargille type A immersion oil used at The Pennsylvania State University, Coal Research Section.

One glass (usually the 1.006 percent R_0) is used as the primary standard, and one, or possibly two glasses (higher or lower in reflectance, depending on the sample rank), are used as checks against the primary standard. During standardization the operator must be sure the glasses are leveled properly and have a scratch-free and tarnish-free surface. The immersion oil on the glass standards must be free from dust and in equilibrium with the temperature of the sample.

Focus is another potential instrumental error which affects both standardization and resolution during scanning. The effects of loss of focus have been discussed in Section III, and it is sufficient to state here that correct focus must be maintained by the operator at all times.

Proper system adjustment and alignment are critical if instrumental error is to be avoided. The optic system and light source should be adjusted to eliminate image flare while maximizing the luminous flux. (See Section III for a discussion of the instrument adjustment necessary for maximum precision.) The settings of all diaphragms and shutters should be checked routinely.

Finally, any reflectance analysis should be carried out in a room with subdued lighting. This minimizes the possibility of stray light entering the optic system and leading to anomalously high readings from the photomultiplier tube.

A second dependent factor controlling reflectogram resolution, sample preparation, has been discussed partially in Section III. Standardization of the proportion of epoxy in the briquette is the first step in ensuring reproducible reflectograms. However, one of the major effects on reflectogram resolution results from the production of large amounts of fine particles during stage crushing of the sample. As was discussed in Section III, nearly every time the Rapid Scan moves from a component of one reflectance to a component of another reflectance, an interface reflectance reading is recorded. The boundary reading is not representative of either component, and its presence on the reflectogram obscures unique reflectance populations. The fewer coal particles encountered during the 125-millimeter scan, the fewer boundary readings between coal and epoxy will be recorded. Hence, sample preparation must be directed toward the production of a minimum number of fine particles while maintaining sample representativeness.

In order to acquire some quantitative information about coal particle size in briquetted samples, two samples each of high volatile A,

medium volatile and low volatile bituminous coals were analyzed using Rapid Scan programs developed for pyrite particle-size scanning (Kuehn and Davis, 1979). The only modification to the program was a change in the sensing algorithm, enabling the Rapid Scan to size all coal particles, and not just pyrite. The samples were selected to cover a range in Hardgrove Grindability Index (HGI) (a measure of the friable characteristic of coal) for each coal rank. The decile statistics from the scans of the six samples are shown graphically in Figure 33. The graphs indicate that, with increasing coal rank, of the range studied, the number of fine-coal particles (less than 50 microns) increases. This trend also is supported by an increase in the Hardgrove Grindability Index from an HGI of 48 for PSOC-1104 to an HGI of 122 for PSOC-618. Thus, even with careful stage crushing of the sample, the more friable, low volatile bituminous coals produce a greater number of fine-coal particles. In any sample having more fine particles, the total number of particles encountered during a scan increases (Table 17), leading to a greater number of coal/epoxy boundary reflectance readings being recorded. These interface readings will, of course, affect reflectogram resolution and must be removed in any quantitative reflectogram interpretation.

A final dependent factor affecting resolution is the choice of a sampling plan. The Rapid Scan system utilizes a systematic lineal or Rosiwal analysis (Figure 4). The lineal analysis method provides an unbiased estimate of the volume fraction of randomly dispersed phases on a two-dimensional surface (Rosiwal, 1898, pp. 143-175). It has a sampling efficiency equal to areal analysis and better than random

hvAb

mvb

lvb

Figure 33. THE PARTICLE SIZE DECILE STATISTICS FOR SIX COALS OF VARYING RANK AND HARDGROVE GRINDABILITY INDEX

Table 17

The Number of Coal Particles Encountered During the Scanning of Coals of Different Ranks

Rank	Sample	Number of Particles Encountered	Number of Total Interface Readings (1)	Percent of Total Coal Readings that were Interface Readings (2)
High Volatile A Bituminous	PSOC-1104	751	1502	3.9%
High Volatile A Bituminous	PSOC-1099	723	1446	3.7%
Medium Volatile Bituminous	PSOC-320	934	1868	4.9%
Medium Volatile Bituminous	PSOC-336	884	1768	4.6%
Low Volatile Bituminous	PSOC-620	1063	2126	5.5%
Low Volatile Bituminous	PSOC-618	1140	2280	5.9%

(1) Calculated as twice the total number of particles encountered.

(2) Calculated from the number of readings on the coal. (50,000 readings were taken. 38,500 readings (or 77 percent) were on coal and 11,500 readings (or 23 percent) were on epoxy.)

point-count techniques (Hilliard and Cahn, 1961). Thus, for the Rapid Scan, it is the preferred sampling technique which can provide the greatest amount of unbiased information in a given time.

Other automated reflectance microscope systems utilize variously modified fine-mesh lattice point-count techniques (Kojima et al., 1979). With this approach, one must beware of interactions between the sampling grid and the size consist of the components sampled. Such interactions may produce a bias in the sampling and distort the resolution of the reflectogram.

The first independent factor affecting resolution is the concentration of the various coal macerals in the sample. Generally, six to nine different macerals are identified and counted during a visual petrographic analysis of bituminous coals. In a study of United States coals, Waddell et al. (1978) showed that vitrinite was by far the predominant maceral ($\bar{X}$ = 78.0 percent; σ = 9.0). They also noted that for the 119 coals studied all inertinite group macerals combined represented 16.2 percent (σ = 6.8) and all liptinite macerals combined averaged 5.8 percent (σ = 5.7) by volume of United States coals; hence, it is probable that inertinite or liptinite macerals could exist in individual coals in concentrations of five percent or less.

In order to test if the Rapid Scan was sensitive to a concentration of five percent of a material, an artificial sample was prepared with a five percent (by volume) spike. The study coal was a lithotype of the Lower Kittanning seam, in which the vitrinite, inertinite and liptinite contents were 43.1 percent, 40.6 percent, and 16.3 percent, respectively. The spike was prepared from an optical glass with a size range of minus 500 microns (35 mesh, U.S. sieve size) by plus

350 microns (45 mesh, U.S. sieve size). As can be seen from the reflectogram of the mixture (Figure 34), the five percent concentration spike is quite apparent despite the lack of resolution of the more abundant coal macerals. Thus, the concentration of a component in a sample is not, in itself, a factor limiting resolution. However, as will be described later, where maceral concentration of five percent or less is one of several limiting conditions, concentration does become an important factor.

The second independent factor affecting reflectogram resolution is the overlap and spread in the reflectance distributions of individual macerals. Since reflectance is the sole property which is being utilized to characterize coal by all automated reflectance microscope systems, one would expect that a detailed knowledge of the reflectance of coal macerals would exist already. However, with the exception of the aforementioned results of Dormans et al. (1957), little comprehensive data have been published on the reflectance of macerals other than vitrinite. The reflectance of inertinite and semi-inertinite macerals has been reported by Austen et al. (1966), Alpern and de Sousa (1970) and Davis (1978). Reflectance ranges for some other macerals also have been reported by ICCP (1961) and Stach et al. (1975, pp. 74-97). In one example of the importance of this data, Kojima et al. (1974) attempted to deconvolute and quantitatively analyze reflectograms based on individual maceral reflectance distributions. Unfortunately, none of these published data provide the comprehensive information about maceral reflectance necessary for interpretation of their effects on Rapid Scan reflectograms.

Figure 34. A REFLECTOGRAM OF A LOWER KITTANNING SEAM
LITHOTYPE WITH A GLASS SPIKE

In order to obtain a better base of data on maceral reflectances, measurements were taken on up to five individual macerals in 20 United States coals, varying in rank from high volatile C bituminous to semianthracite. Because of the difficulties in measuring the reflectance of some macerals, such as small size, low concentration, and poor polish, a control experiment was carried out prior to doing the analyses on the sample suite. The object of the control experiment was to determine the minimum number of reflectance readings necessary to calculate a reasonably precise mean reflectance for each maceral. As discussed in Section III, one method of comparing precision to sample size is by calculating the standard error of the mean. In this experiment three coals, PSOC-3, PSOC-274 and PSOC-132, were analyzed and the standard error of the mean was calculated at various sample sizes following equation 8. The results from the analyses are shown graphically in Figure 35.

The general trend in these analyses is for the standard error of the mean to decrease (indicating increasing precision) as the number of samples increases. Most of the improvement in precision (up to three-fold) is associated with an increase in sample size from ten to 50 readings. Sample sizes greater than 50, generally show only limited improvement in precision. Thus, because of the difficulties referred to above, a sample size of 50 was set as the target number for all macerals other than vitrinite. The target number for vitrinite, being more abundant, more homogeneous, and present in larger sizes, was set at 100.

It is interesting to note the ranking of the different macerals in Figure 35. Generally micrinite and semifusinite have the highest

Figure 35. THE RELATIONSHIP BETWEEN THE STANDARD ERROR OF THE MEAN AND INCREASING SAMPLE SIZE FOR DIFFERENT MACERALS FROM THREE COALS

standard error of the mean, and vitrinite and sporinite have lower values. This would indicate that micrinite and semifusinite inherently have higher variations in their reflectance properties than vitrinite and sporinite. In PSOC-132, however, micrinite has the lowest standard error of the mean. In this coal the micrinite generally was agglomerated into masses whose area was well in excess of the four-square micron sensing spot. This probably led to reflectance readings which were representative of the true reflectance of micrinite (unaffected by surrounding maceral material) and, hence, the variation was smaller.

The reflectance analyses for the 20 coals were carried out on the same microscope-computer system used in the Rapid Scan Versions II and III equipment. The reflectance values were measured using a four-square micron sensing spot. They were average random reflectance readings, determined in non-polarized light without stage rotation in order to simulate conditions during automated scanning. The mean reflectance and standard deviation were calculated for each maceral which was sufficiently abundant to yield 50 readings, and are reported in Table 18.

One point which is apparent in Table 18 is that no reflectance data were collected for sporinite or semifusinite in the low volatile and higher rank coals. In the case of sporinite, its reflectance equals (and in some cases exceeds) that of the associated vitrinite in the medium volatile rank range. In low volatile coals the similarity in reflectance between sporinite and vitrinite makes its (sporinite's) recognition almost impossible. The lack of reflectance data for semifusinite in these samples is the result of a definitional boundary.

Table 18

Maceral Reflectances for Twenty United States Coals

Sample (Rank)		Vitrinite R_0 (%)	Sporinite R_0 (%)	Semifusinite R_0 (%)	Fusinite R_0 (%)	Micrinite R_0 (%)
PSOC-309 (hvCb)	$\bar{X}$	0.50	0.12	1.34	2.96	--
	σ	0.04	0.03	0.36	0.70	--
PSOC-773 (hvBb)	$\bar{X}$	0.54	0.14	1.08	2.73	1.10
	σ	0.03	0.02	0.38	0.94	0.36
PSOC-462 (hvAb)	$\bar{X}$	0.66	0.16	1.38	3.51	--
	σ	0.05	0.02	0.26	0.40	--
PSOC-261 (hvBb)	$\bar{X}$	0.67	--	1.75	4.56	--
	σ	0.05	--	0.22	0.66	--
PSOC-375 (hvAb)	$\bar{X}$	0.75	0.18	1.34	3.37	1.11
	σ	0.07	0.02	0.29	0.90	0.25
PSOC-369 (hvAb)	$\bar{X}$	0.79	0.20	1.30	3.31	1.42
	σ	0.05	0.03	0.26	0.90	0.36
PSOC-12 (hvAb)	$\bar{X}$	0.81	0.21	1.45	3.27	1.36
	σ	0.06	0.03	0.25	0.80	0.30
PSOC-3 (hvAb)	$\bar{X}$	0.84	0.22	1.56	2.80	1.60
	σ	0.07	0.02	0.23	0.76	--
PSOC-266 (hvBb)	$\bar{X}$	0.92	0.43	1.43	3.01	1.60
	σ	0.05	0.06	0.25	0.85	--
PSOC-359 (hvAb)	$\bar{X}$	1.02	0.34	1.37	3.63	--
	σ	0.05	0.04	0.19	0.98	--
PSOC-274 (mvb)	$\bar{X}$	1.05	0.58	1.48	3.74	1.74
	σ	0.07	0.08	0.26	1.04	--
PSOC-255 (mvb)	$\bar{X}$	1.16	1.13	1.63	3.62	--
	σ	0.05	0.09	0.19	1.09	--
PSOC-256 (mvb)	$\bar{X}$	1.21	1.05	1.63	4.28	--
	σ	0.04	0.05	0.27	1.12	--
PSOC-336 (mvb)	$\bar{X}$	1.25	1.30	1.72	2.91	1.92
	σ	0.05	0.06	0.15	0.94	0.30
PSOC-132 (mvb)	$\bar{X}$	1.41	1.50	1.88	4.04	2.14
	σ	0.04	0.09	0.23	0.98	0.28

Table 18 (Continued)

PSOC-142	$\bar{X}$	1.52	--	--	3.84	2.57
(mvb)	σ	0.06	--	--	0.96	0.40
PSOC-319	$\bar{X}$	1.60	--	--	3.74	2.16
(lvb)	σ	0.08	--	--	1.22	0.22
PSOC-405	$\bar{X}$	1.67	--	--	4.33	2.38
(lvb)	σ	0.07	--	--	1.02	0.30
PSOC-646	$\bar{X}$	1.71	--	--	3.34	--
(lvb)	σ	0.08	--	--	0.77	--
PSOC-84	$\bar{X}$	2.49	--	--	3.75	--
(sa)	σ	0.14	--	--	0.71	--

Although several attempts have been made at setting a reflectance boundary between fusinite and semifusinite (Bell et al., 1969), no international standard currently exists. In this experiment the lower reflectance boundary for semifusinite was set at the reflectance of the associated vitrinite, and the upper boundary was set arbitrarily at two percent R_0 . This level was chosen because it represents the reflectance at which vitrinite begins to lose its thermoplastic properties upon carbonization. Therefore, in the higher rank coals, very few particles of structured material which could be called semifusinite fell in this narrow definitional range.

The reflectance data for the various macerals has been plotted in Figure 36 against the vitrinite reflectance of the individual sample. From this figure, it is apparent that the mean reflectances of sporinite, vitrinite and fusinite are separated sufficiently that their resolution in a reflectogram should be expected. The mean reflectances of semifusinite and micrinite however, are intimately intermixed. This may be due to the measured reflectance values for the micrinite being lower than their true values. Micrinite is present in coals as granular micron to submicron particles. The reflectance measurements were made on aggregates of the material which were greater than four-square microns and which appeared homogeneous. There is the distinct possibility that some unnoticed vitrinite material was included in these aggregates, lowering the measured reflectance value. This is the same situation which exists when measurements are made with the automated microscope and, hence, the intermixing of micrinite and semifusinite reflectance readings would occur in reflectograms.

Figure 36. A COMPARISON OF THE REFLECTANCES OF DIFFERENT MACERALS TO THE ASSOCIATED SAMPLE VITRINITE REFLECTANCE

Macrinite was not abundant in most of these coals, but some reflectance data were collected. These readings indicate that its mean reflectance would fall in the semifusinite-micrinite range. However, its spread in reflectance is large and it could contribute some reflectance readings as high as the fusinite range.

Although the mean reflectance values of sporinite, vitrinite and semifusinite-micrinite complex and fusinite are separated well, the spread of the reflectance distributions and the amount of overlap also are important. Figures 37, 38, and 39 show the individual maceral reflectance distribution for three coals plotted by program PLOTV2 (Appendix IV), which combines data into half steps (class interval 0.05 percent R_0). As seen in these figures, vitrinite and sporinite have the tightest reflectance distributions, fusinite has the greatest reflectance spread, and micrinite and semifusinite have spreads intermediate between vitrinite and fusinite. The very wide spread in the reflectance values of fusinite, and the lack of strong central tendency, indicates that it is unlikely that fusinite could ever form a distinct peak on a reflectogram. The other four components display sufficiently tight reflectance distributions that one would expect distinct peaks (or at least low rises on the reflectogram) if reflectance is the only factor controlling resolution.

The overlap of these reflectance distributions also is an important control on reflectogram resolution. During a visual petrographic analysis, the operator takes into account both reflectance and morphology in identifying maceral types. Because the automated system uses only reflectance as the determining criterion, overlapping reflectance distributions can lead to misplaced information in the reflectogram.

Figure 37. THE REFLECTANCE DISTRIBUTIONS OF DIFFERENT MACERALS FROM PSOC-3

Figure 38. THE REFLECTANCE DISTRIBUTIONS OF DIFFERENT MACERALS FROM PSOC-274

Figure 39. THE REFLECTANCE DISTRIBUTIONS OF DIFFERENT MACERALS FROM PSOC-132

Figure 40 shows the same data plotted in Figure 36 except that the approximate range in reflectance (given by $\pm 2\sigma$) is shown for the different macerals by stippled patterns. The range in reflectance for sporinite is well separated from the vitrinite reflectance range, until medium volatile bituminous rank coals are encountered. In a visual maceral analysis, when sporinite reflects similarly to the associated vitrinite, it is counted as vitrinite; therefore the overlap which develops at higher ranks poses no problem with misplaced readings in the reflectogram. Both the semifusinite and micrinite distributions overlap with that of the vitrinite, but not so significantly as to limit resolution. As was expected from the comparison of mean reflectances, the distributions for micrinite and semifusinite overlap totally, ruling out any possibility of resolving these components one from the other by automated reflectance microscopy. The fusinite reflectance distribution has considerable overlap with that of micrinite, and lends doubts about the ability to resolve these components.

In summary, the mean reflectance, the spread of reflectance distributions, and the overlap of these distributions indicate that some components (e.g. micrinite and semifusinite) will never be separated from one another in a reflectogram; however, there is promise that other macerals could be resolved. To demonstrate these relationships an artificial reflectogram has been produced in Figure 41. The percentage, the mean reflectance, and the spread in reflectance of the different macerals have been calculated into this reflectogram. As has been suggested, the epoxy, the sporinite, and the vitrinite peaks all are resolved well in the reflectogram. The micrinite-semifusinite complex is evident as one wide skewed peak. Fusinite,

Figure 40. THE OVERLAP IN REFLECTANCE DISTRIBUTIONS OF DIFFERENT MACERALS FOR VARIOUS RANK COALS

PEAKS	% OF TOTAL READINGS
A. Epoxy	23
B. Sporinite	4
C. Vitrinite	59
D. Micrinite + Semifusinite	11
E. Fusinite	3

Figure 41. AN ARTIFICIAL REFLECTOGRAM PRODUCED FROM VISUALLY-DERIVED DATA

due to its low concentration in this sample (approximately three percent) and its wide spread in reflectance, is apparent only as a broad spread in reflectance readings. If one compares this artificial reflectogram to the actual reflectogram of this coal (PSOC-176) shown in Figure 32b, it is evident that at least one other major control is affecting reflectogram resolution.

The final control affecting reflectogram resolution is the relationship between the size and shape of the different macerals and the size of the sensing spot of the photometer. In a single sample briquette coal macerals may vary in size from 850 microns for occurrences of vitrinite down to less than one micron grains of micrinite. With the exception of vitrinite, which ranges in size from the upper limit of sample particle size (850 microns) to less than five-micron particles, most macerals are present in coal in occurrences of 20 microns or less (Figure 42).

Figure 42 also illustrates that most macerals, especially sporinite, occur in non-equidimensional shapes. Granting that macerals occur in various sizes and shapes, one would expect that a relationship would exist between the finite size and shape of the measuring spot, and the likelihood of that spot intersecting the perimeter of a given maceral (Frompovicz, 1978).

Figure 43 draws the relationship of perimeter to area for three regular polygons. In the case of a square (an equidimensional form) the ratio of perimeter to area decays rapidly with increasing size (area). This decay is a result of the relationship being:

E-Epoxy, V-Vitrinite, S-Sporinite, M-Micrinite, F-Fusinite

■ 2.5 μ m X 2.5 μ m Sensing Spot

Figure 42. A REFLECTED LIGHT PHOTOMICROGRAPH OF A HIGH VOLATILE A BITUMINOUS COAL CONTAINING SEVERAL MACERALS

Figure 43. THE RELATIONSHIP BETWEEN PERIMETER AND AREA FOR VARIOUS SHAPED POLYGONS

$$A = K(P)^2 \quad (14)$$

where,

A = the area,

K = a constant, and

P = the perimeter.

As the form becomes more and more elongated, the decay ratio decreases as the constant becomes smaller and smaller. Thus it is evident that as a particle becomes smaller, or more elongated, the length of the perimeter increases in relationship to form area. Hence, with smaller, or more elongated particles, the number of potential reflectance readings occurring on the perimeter (interface with other macerals) increases.

The Rapid Scan, utilizing lineal analysis, will, in nearly all cases record two perimeter (interface) reflectance readings for each particle traversed. Figure 44 illustrates the concept that given a Rapid Scan sensing spot of 2.5 microns square, the ratio of perimeter (interface) reflectance readings, to nonperimeter readings increases exponentially as the random chord width of the particle decreases. Because the random chord lengths of most macerals other than vitrinite will be less than 20 microns, one would expect a high number of interface readings for these macerals to be recorded in a reflectogram.

It was shown in Section III that boundary reflectance readings have values intermediate between those of the two phases involved. Obviously a large number of these readings are generated during a Rapid Scan analysis, so it is important to understand how they contribute to reflectogram shape. In order to investigate this effect, a series of scans were made on a two-component system in which various

Figure 44. THE RELATIONSHIP BETWEEN PARTICLE SIZE AND THE NUMBER OF PERIMETER (BOUNDARY) RAPID SCAN READINGS VERSUS NONPERIMETER READINGS

particle sizes were simulated using glass reflectance standards. Figure 45 shows the results of three scans in which the percentage of the two components and their respective reflectances were held constant and only particle size was varied. In the case of the 800-micron particle, the few interface readings that were recorded are scattered more or less evenly between the two dominant populations. Note also that even with large particle sizes, the two populations are skewed toward one another with somewhat more skewness displayed by the lower reflecting material. This phenomenon becomes more prominent as particle size decreases, being particularly evident in the scan of 50-micron particles which, as anticipated, display many more boundary readings.

The cause of this disproportionate skewing is related to the relative reflectances of the two components involved. An infinite number of combinations exist in which the two components can be present in different proportions under the sensing spot, leading to an interface reflectance reading. Consider firstly the case where precisely 50 percent (by area) of each component is present. In order that the resulting reading be located correctly in relation to these proportions, it should lie one-half way between the two populations. However, because one phase has a distinctly higher reflectance, it will contribute more in terms of reflected photons than its 50 percent proportion warrants. Hence, the reading will be displaced from the center toward the higher reflecting population. By extrapolating this relationship to all possible combinations, it is evident that each reading is skewed incrementally toward the higher reflecting component, leading to the skewness displayed by the low reflecting components in

**Figure 45. THE EFFECT OF DECREASING PARTICLE SIZE
ON A TWO-COMPONENT SYSTEM**

Figure 45. Despite the skewness developed in these scans, the modal value of the populations remains unaffected. This fact becomes important in quantitative interpretation of the vitrinite peak, as is described in Section V.

Figure 46 shows how interface reflectance readings affect a two-component system in which particle size and percent constituent are constant, and reflectance difference varies. Where components A and B are close in reflectance, both populations similarly are skewed and the interface readings are stacked up between the two populations. In the case of components A and D, for which a large difference in reflectance exists, both populations are skewed, with a greater number of readings skewed from the lower reflecting component A population. Thus, the more reflectance difference which exists between the two components forming the interface reading, the more the readings are displaced from the parent populations, and the more the lower population, in particular, is skewed.

These studies on the effect of size (and hence, boundary readings) on the shape of reflectograms, show that resolution is severely reduced. Four types of interface readings are encountered during the course of a scan: epoxy/maceral, epoxy/mineral, maceral/maceral, and maceral/mineral. The number of epoxy/other interfaces has been considered earlier in this section. Their location in the reflectogram mainly will be between the vitrinite and epoxy peaks for most United States coals, thus, the sporinite peak will be partially obscured. Owing to the low reflectance of most clays and quartz, the majority of the maceral/mineral interactions also report in this region. The sporinite peak is obscured further by its inherent large number of

Figure 46. THE EFFECT OF BOUNDARY REFLECTANCE READINGS ON TWO-COMPONENT SYSTEMS OF INCREASING REFLECTANCE DIFFERENCE

interface readings (due to size) which skew its distribution, and, in some cases, may obscure totally its population. The semifusinite, fusinite, and micrinite populations likewise are skewed and distorted. All of this means that instead of the ideal reflectogram conceptualized in Figure 41, the actual product appears like Figure 32b. Even coals which have significant amounts of these fine-grained macerals (i.e. sporinite, micrinite, and fusinite with bogen structure) will not produce reflectograms with distinct peaks other than the vitrinite peak.

Any attempt to quantitatively interpret reflectograms for petrographic composition must consider all six factors which affect reflectogram resolution. The effect of dependent factors can be minimized by good operating technique throughout the analysis. However, the effects of the independent factors, particularly grain size, are difficult to evaluate, and undoubtedly will limit the amount of directly quantifiable information which can be gleaned from a reflectogram.

V. QUANTITATIVE INTERPRETATION OF RAPID SCAN REFLECTOGRAMS

A. Determination of Vitrinite Reflectance Parameters

The preceding section offers an explanation of how reflectograms are a complex composite of distorted reflectance populations. Quantitative interpretation of the reflectogram must avoid simplistic curve-stripping approaches. The computerized model to be described in this section is one approach to dealing with the reflectogram complexities on both a theoretical and empirical level.

As discussed previously, one important quantitative measure which can be derived from the reflectogram is the vitrinite reflectance. In the United States, the vitrinite reflectance value commonly measured by conventional photometry is a maximum reflectance determined by rotation of the vitrinite particle in polarized light (ASTM, 1979). Maximum reflectance is measured because it has a smaller standard deviation than either average random reflectance or minimum reflectance (de Vries et al., 1968; Pitt, 1975). It is easier to discriminate between several coals in a blend if the reflectance distribution is tighter (de Vries et al., 1968). In addition, the two major methods of coke strength prediction used in the United States utilize maximum vitrinite reflectance parameters.

Although vitrinite is not a crystalline solid, it does possess internal order and, in bituminous ranks, optically behaves somewhat like a uniaxial negative crystal (Cannon and George, 1943; Williams, 1953; de Vries et al., 1968). Any random surface sliced from a vitrinite particle in theory will pass through the circular section of the uniaxial negative indicatrix. Consequently, any surface will

display maximum reflectance at two locations approximately 180 degrees apart when the particle is rotated in the image plane. The surface also will display another characteristic reflectance value which may be equal to the maximum, intermediate between the maximum and the minimum, or equal to the minimum, depending on the orientation of the surface within the indicatrix (Figure 47).

Consider the effect of various orientations of surface A (Figure 47) on the reflection of vertically incident light. If the light is plane polarized, the magnitude of the reflected light will depend on the orientation of the polarization direction in relationship to R_w and R_E . Figure 48a shows the result of rotating surface A through 90 degrees starting with R_w parallel to the polarization plane of the incident light. As illustrated in this figure, as R_w is rotated out of alignment with the polarization plane of the incident light, the magnitude of the reflected light decreases until a minimum is reached when R_E is parallel to the polarization plane. Thus, a 360-degree rotation of the surface in plane-polarized light will yield two maximum reflectances and two minimum reflectances. It is common practice in the United States to connect a chart recorder or peak detector to the photometer and record the maximum reflectance value in order to characterize the vitrinite reflectance.

It mechanically is impractical to rotate the microscope stage during an automated reflectance analysis, and hence, maximum reflectance cannot be determined easily. Without the need for polarized light, the polarizer is removed from the optic path, increasing the luminous flux and improving the signal quality. When non-polarized light is directed onto a surface such as A in Figure 47, the magnitude of the

R_{ω} = Maximum Reflectance

R_{ϵ} = Minimum Reflectance

R'_{ϵ} = Apparent Minimum Reflectance

Figure 47. THE REFLECTANCE CHARACTERISTICS OF DIFFERENT SURFACES OF A UNIAXIAL NEGATIVE VITRINITE PARTICLE

Figure 48. A COMPARISON OF MAXIMUM AND AVERAGE REFLECTANCE

reflected light is controlled by the average of R_w and R'_e . As shown in Figure 48b, the orientation of surface Λ does not affect the magnitude of the reflected light. Because the reflectance is an average of R_w and the second principal reflectance, any surface cut through this vitrinite particle in theory will display an average reflectance dependent on the magnitude of R'_e . Any reflectance measured on a random surface, such as those present on the surface of a briquetted sample, will be an average random reflectance and will have a variance which will include the following components: the natural variation in the vitrinite; the experimental variation; and the variation due to the orientation of the surface and resulting bireflectance. Thus, the variance associated with measuring mean average random reflectance (MARR) will be higher than that associated with a mean maximum reflectance (which is subject to only the first two of the above sources of variation).

Other authors have chosen to use different terminologies to describe reflectance values measured in non-polarized light without stage rotation. De Vries et al. (1968), de Sousa (1972), and Ting and Lo (1978) have used the term "average" ("mittel" or "moyen") to refer to this type of reflectance reading. Davis and Vastola (1976) and the International Standards Organization (1979) used the term "random." In a further complication of terminology, Hevia and Virgós (1976) used random reflectance to refer to reflectance values determined in polarized light without stage rotation. Owing to the inconsistencies in the literature and the explanation given above, this report describes reflectance measured in non-polarized light without stage rotation as average random reflectance.

Because the relationship between mean maximum vitrinite reflectance and mean average random vitrinite reflectance (MARR) is dependent on the bireflectance present in the coal, an empirical equation can be developed to convert one value to the other. Hoover et al. (1976) and Davis and Vastola (1976) used this approach to convert MARR values, derived from Rapid Scan reflectograms, to mean maximum reflectances. This enabled them to obtain data from automated systems which could be compared to mean maximum values derived from conventional analyses or used directly in existing coal performance prediction procedures. Pitt (1975), Hower (1978), Ting (1978), and Gray et al. (1979a) also have developed empirical equations for converting visually-derived MARR values to mean maximum reflectances. All of the equations, with the exception of Hower (1978) and Gray et al., (1979a), were developed with sample sets restricted in size and areal extent.

In order to develop an equation applicable to a wide range of bituminous coals, a data set of over 600 analyses was assembled from the literature on coal reflectance, and the author's experimental data. Sources of the information gleaned from the literature search included: Hower (1978) (this was the source of much of the data, which were restricted to United States coals); Pitt (1975) (British coals); de Vries et al. (1968) (European coals); and ICCP (1978) (European and Canadian coals).

A linear regression analysis was performed on the data set, utilizing the computer program MINITAB II (Ryan, 1976), maintained on The Pennsylvania State University computation center IBM 370 computer. The results of the analysis, shown in Figure 49, included the following equation:

Figure 49. THE CORRELATION BETWEEN MEAN MAXIMUM VITRINITE REFLECTANCE AND MEAN AVERAGE RANDOM VITRINITE REFLECTANCE FOR VARIOUS COALS

$$\bar{R}_{\max} = \bar{R}_{\text{ar}} \times 1.061 \quad (15)$$

where,

$\bar{R}_{\max}$ = the mean maximum reflectance and

$\bar{R}_{\text{ar}}$ = the mean average random reflectance.

This equation agrees well with those of Ting (1978) ($\bar{R}_{\max} = \bar{R}_{\text{ar}} \times 1.066$) and Hower (1978) ($\bar{R}_{\max} = (\bar{R}_{\text{ar}} \times 1.066) - 0.01$), and can be utilized to convert MARR values to mean maximum reflectance values for bituminous coals from any basin.

An experiment then was designed to explore if coals from a single structural and sedimentological basin would have unique bireflectance properties. Twenty-two coals from western Pennsylvania were analyzed visually for mean average random vitrinite reflectance and for mean maximum vitrinite reflectance using the program CONAL3 (Appendix V). This program enables mean maximum reflectance or mean average random reflectance to be measured using the automated reflectance microscope installation in a conventional mode. Owing to the inherently higher variance of average random reflectance measurements the Student's t test (Griffiths, 1967) was used to evaluate the significance of the sample mean. It was determined that 250 measurements were necessary to generate a mean with equal statistical significance to one calculated from 100 maximum reflectance values. The results of a linear regression of these data are shown in Figure 50. The standard deviation of the regression is not significantly better than that in Figure 49, indicating that little improvement has been made by geographically restricting the sample set. Because the difference between the constants in the two equations (1.061 and 1.053) is within the experimental

Regression Equation - $\bar{R}_{\max} = \bar{R}_{\text{ar}} * 1.053$

Standard Deviation about the Regression Line = 0.023

* indicate individual data points

Numbers indicate number of superimposed data points

Figure 50. THE CORRELATION BETWEEN MEAN MAXIMUM VITRINITE REFLECTANCE AND MEAN AVERAGE RANDOM VITRINITE REFLECTANCE FOR WESTERN PENNSYLVANIA COALS

error inherent to the analysis, equation 15 has been used as a standard means for converting mean average random vitrinite reflectance values to mean maximum vitrinite reflectances.

Another aspect of this experiment sheds some light on the geologic history of the coal basin. It is considered generally that the bireflectance developed in vitrinite is a result of increasing aromaticity and orientation of aromatic molecules (van Krevelen, 1953; de Vries et al., 1968). The phenomenon of orientation parallel to the bedding can be considered as pressure-induced anisotropy, resulting from burial of the coal seam (Stach et al., 1975). Hence, vitrinite anisotropy has been suggested as an indicator of pressure and, therefore, depth (Wege, 1954; Teichmüller and Teichmüller, 1954; Hower, 1978).

If the depth of burial and rank were constant across a basin, one might expect little change in vitrinite anisotropy. Thus, the difference between mean maximum vitrinite reflectance and mean average random reflectance (one indicator of bireflectance) should be a unique value. However, if depth of burial was constant and rank increased across the basin, one would expect the relationship to change, but in a constant (or linear) manner. If both depth of burial and rank varied across the basin, then the relationship should change in a nonlinear manner.

The comparison of mean average random reflectance to mean maximum vitrinite reflectance for western Pennsylvania coals (Figure 50) shows a linear relationship. This supports Hower's conclusion that coals from this basin were buried to approximately the same depth (Hower, 1978). The scatter in the data points may be the result of experimental error,

or may result from localized pressure variations due to folding (Levine and Davis, 1979). A more sensitive indicator (less affected by experimental error) of anisotropy would be a comparison of mean minimum reflectance to mean maximum reflectance. However, one explanation of the linear relationship shown in Figure 49, is that most of these coals may have experienced similar tectonic histories.

As stated above, vitrinite reflectance values were determined from Rapid Scan Version I reflectograms by locating visually the mode of the vitrinite peak and reading its value from the χ axis of the plot. In order to remove any effect of human subjectivity and totally automate this procedure, programming was developed which could derive quantitative data directly from the reflectogram.

Researchers working in the field of spectral analysis (e.g. X-ray, infrared spectrometry) have developed several mathematical techniques for deconvoluting spectra (Koenig, 1975; Pitha and Jones, 1966; Morgan, 1977; and Vandeginste and De Galan, 1975). These techniques generally involve curve stripping using Gaussian or Lorentzian functions. Calculated curves are compared to the unknown distribution using a least squares method to find the minimal deviation. The most important requirements for this procedure as applied to infrared spectroscopy are that the number of absorption bands are known for the sample, and that the mathematical model gives a correct description of each band (Vandeginste and De Galen, 1975). This technique probably is not applicable to reflectograms because the number of reflectance populations, their position on the reflectogram, and their shapes generally are not known. Therefore, a modular program was written using curve stripping to determine vitrinite parameters (reflectance and amount), but

employing analysis of the residual populations to determine the proportions of other maceral groups.

The first program, VITFIT, was devised to calculate and match a "best fit" Gaussian distribution to the vitrinite peak on the reflectogram. A Gaussian distribution probably is an overly simplistic model of vitrinite reflectance because coals can contain more than one population of vitrinite, and the reflectance distribution can be distorted, as discussed in Section IV. The potential errors associated with minor departures from normality as a result of such distortions are not important to calculations of vitrinite reflectance parameters. Providing the coal does not contain large amounts of highly reflecting telocollinite ("pseudovitrinite"), the Gaussian distribution approximates the actual distribution quite well.

The computer fit is accomplished by a series of algorithms operating in the sequence of logic shown in Figure 51. An example of one such "fit" is shown in Figure 52 and indicates the degree of match that generally is achieved using this approach. Once the model of the vitrinite reflectance distribution is calculated, it is a simple matter to print out the mean, standard deviation, and population. The mean value, which is an estimate of the mean average random vitrinite reflectance, is substituted into equation 15 to calculate the mean maximum vitrinite reflectance.

In addition to the mean maximum vitrinite reflectance, several laboratories utilize a "V-type analysis" in their predictions of the coking behavior of coals. A V-type is designated by a one- or two-digit number, and represents a reflectance class interval of 0.10 percent reflectance. V-type 7, for instance, is a sum of all the

Figure 51. A FLOW CHART OF THE LOGIC USED IN VITFIT TO CALCULATE VITRINITE REFLECTANCE PARAMETERS FROM REFLECTOGRAMS

Figure 52. AN EXAMPLE OF THE PROGRAM VITFIT'S MATCHING OF A GAUSSIAN DISTRIBUTION TO THE VITRINITE REFLECTANCE PEAK

reflectance readings between 0.70 and 0.79 percent. Commonly each V-type is reported as a percentage of the total vitrinite. The concept behind such usage is that the various V-types represent significant compositional variations in the vitrinite of any coal. Certainly more than one population may be represented in the total vitrinite peak of a reflectogram, but it seems unlikely that a simple V-type distribution is effective in differentiating between vitrinite types.

The program V-TYPE was developed for coal reflectograms in an attempt to duplicate the kind of information contained within a conventional V-type analysis. This program takes the information generated by VITFIT and recalculates it into V-type classes. As stated above, the variance inherent in average random reflectance measurements is greater than that of maximum reflectance measurements. Therefore, before apportioning the readings into V-types, a correction factor must be applied to the variance of the average random reflectance distribution. The correction factor was determined from a data set of 100 coals of bituminous rank. A linear regression analysis was performed on the standard deviation of the mean average random vitrinite reflectance and the standard deviation of the mean maximum vitrinite reflectance. Although the resulting scatter of data is rather large (Figure 53) the regression equation:

$$\sigma_{\max} = \sigma_{\text{marr}} \times 0.842 \quad (17)$$

where,

$\sigma_{\max}$ = the standard deviation of the maximum reflectance distribution, and

σ_{marr} = the standard deviation of the mean average random reflectance distribution

Regression Equation $\sigma_{\max} = \sigma_{\text{marr}} \cdot 0.842$

Standard Deviation about the Regression Line = 0.007

* indicate individual data points.

Numbers indicate number of superimposed data points

Figure 53. THE CORRELATION BETWEEN MEAN MAXIMUM VITRINITE REFLECTANCE AND MEAN AVERAGE RANDOM VITRINITE REFLECTANCE

describes the underlying relationship and can be used to make the correction. The program V-TYPE recalculates the mean and standard deviations from the program VITFIT using equations 15 and 17. It then calculates a new Gaussian distribution (from these converted values), approximating one which would be expected from a mean maximum reflectance analysis. (See Figure 54 for an example of the recalculated distribution from Figure 52.) The program then sums the number of reflectance readings in each class interval, normalizes the total to 100 percent, and calculates V-type percentages.

Programs VITFIT and V-TYPE were incorporated into the larger reflectogram analysis program called PETAN. Program PETAN generates a standard two-page printout of vitrinite reflectance and compositional information derived from a reflectogram. Page one of the printout (Figure 55) first lists sample information and analysis conditions. This information is entered and stored on the magnetic disk at the time of the analysis. It automatically is recalled and printed along with the analytical data. The rest of page one represents the vitrinite reflectance parameters derived by VITFIT and V-TYPE.

In order to test the Rapid Scan reflectograms and program VITFIT for their accuracy in determining mean average random vitrinite reflectances, the same suite of western Pennsylvania coals used to determine equation 16 were scanned and analyzed. The results, shown in Table 19, indicate that in all but one case the absolute difference between the visually determined mean average random vitrinite reflectance and the value determined from the reflectogram utilizing VITFIT is equal to or less than 0.02 percent R_0 . Plus or minus 0.02 percent reflectance is the deviation allowable under the ASTM standard for

Figure 54. AN EXAMPLE OF THE APPORTIONING OF VITRINITE REFLECTANCE CLASSES BY PROGRAM V-TYPE

****PENN STATE COAL RESEARCH SECTION****
RAPID SCAN PETROGRAPHIC ANALYSIS

PAGE-1

SAMPLE DESIGNATION PSOC-276

DATE 11/ 9/1978

**** ANALYSIS CONDITIONS ****

FULL SCALE REFLECTANCE----- 2.00%
 STANDARDS USED----- 1.006%, 0.533%
 STAGE DRIVE SPEED (U/SEC.)-- 200.
 NUMBER OF DATA POINTS----- 50000.
 SPOTSIZE AREA IN MICRONS--- 4.
 TIME FOR ANALYSIS (MIN)---- 8.33
 COMMENTS----- PITTSBURGH SEAM

**** SUMMARY ANALYSIS DATA ****

VITRINITE REFLECTANCE PARAMETERS

MARR VALUE----- 0.64%
 STANDARD DEVIATION----- 0.046%
 VIT. DISTRIBUTION POPULATION--- 22875.

CALCULATED VITRINITE MEAN-MAX.--- 0.68%

VITRINOID TYPE TABLE

V4	V5	V6	V7	V8	V9	V10	V11
---	---	---	---	---	---	---	---
0.0	0.9	67.5	31.6	0.0	0.0	0.0	0.0
V12	V13	V14	V15	V16	V17	V18	V19
---	---	---	---	---	---	---	---
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

Figure 55. AN EXAMPLE OF PAGE ONE OF THE PRINTOUT FROM PETAN

Table 19

Comparison of Mean Average Random Vitrinite Reflectance

Determined Visually to Mean Average Random

Vitrinite Reflectance Determined by VITFIT

Sample (Rank)		Mean Maximum R ₀ %	MARR (%) (Visual)	MARR (%) VITFIT	Absolute Difference MARR _{VIS} - MARR _{VITFIT}
PSOC-194 (hvBb)	$\bar{X}$ σ	0.61 0.05	0.59 0.05	0.56 0.05	0.03
PSOC-276 (hvAb)	$\bar{X}$ σ	0.69 0.03	0.63 0.04	0.64 0.05	0.01
PSOC-1018 (hvAb)	$\bar{X}$ σ	0.79 0.05	0.75 0.06	0.73 0.07	0.02
PSOC-330 (hvBb)	$\bar{X}$ σ	0.80 0.04	0.76 0.05	0.74 0.06	0.02
PSOC-104 (hvAb)	$\bar{X}$ σ	0.82 0.05	0.76 0.05	0.75 0.06	0.01
PSOC-337 (hvAb)	$\bar{X}$ σ	0.87 0.06	0.84 0.06	0.84 0.08	0.00
PSOC-340 (hvAb)	$\bar{X}$ σ	0.88 0.05	0.84 0.06	0.86 0.06	0.02
JCH-70* (hvAb)	$\bar{X}$ σ	1.01 0.06	0.97 0.06	0.97 0.06	0.00
PSOC-1010 (mvb)	$\bar{X}$ σ	1.05 0.05	1.00 0.06	1.00 0.06	0.00
PSOC-324 (mvb)	$\bar{X}$ σ	1.20 0.04	1.16 0.05	1.16 0.05	0.00
PSOC-255 (mvb)	$\bar{X}$ σ	1.20 0.05	1.15 0.07	1.15 0.05	0.00
PSOC-257 (mvb)	$\bar{X}$ σ	1.22 0.05	1.15 0.05	1.15 0.06	0.00
PSOC-254 (mvb)	$\bar{X}$ σ	1.29 0.04	1.24 0.05	1.23 0.05	0.01

Table 19 (Continued)

PSOC-256 (mvb)	$\bar{X}$ σ	1.29 0.04	1.23 0.06	1.23 0.05	0.00
PSOC-258 (mvb)	$\bar{X}$ σ	1.35 0.05	1.31 0.05	1.31 0.06	0.00
PSOC-321 (mvb)	$\bar{X}$ σ	1.42 0.04	1.36 0.05	1.36 0.05	0.00
PSOC-619 (mvb)	$\bar{X}$ σ	1.52 0.08	1.41 0.07	1.41 0.07	0.00
PSOC-116 (lvb)	$\bar{X}$ σ	1.57 0.05	1.49 0.07	1.49 0.05	0.00
PSOC-127 (lvb)	$\bar{X}$ σ	1.62 0.05	1.49 0.07	1.50 0.08	0.01
PSOC-318 (lvb)	$\bar{X}$ σ	1.68 0.05	1.59 0.07	1.58 0.06	0.01
PSOC-876 (lvb)	$\bar{X}$ σ	1.65 0.05	1.59 0.07	1.61 0.08	0.02
PSOC-113 (lvb)	$\bar{X}$ σ	1.80 0.06	1.71 0.08	1.71 0.08	0.00

*Sample provided by James C. Hower.

mean maximum reflectance analysis (ASTM, 1979). The International Standards Organization has proposed that the repeatability of reflectance analysis, should be 0.06 percent R_0 (ISO, 1979). Following either standard, the mean average random vitrinite reflectance data generated by the program PETAN compares favorably with the results of visual analysis.

In order to test whether equation 15, as utilized in PETAN, was accurate for calculating mean maximum reflectance, 43 coals were analyzed using PETAN and their mean maximum reflectances were determined visually; these data then were subjected to linear regression analysis (Figure 56). The resulting equation:

$$\bar{R}_{\max} (\text{vis}) = \bar{R}_{\max} (\text{cal}) \times 1.00 \quad (18)$$

where,

$\bar{R}_{\max} (\text{vis})$ = the mean maximum vitrinite reflectance (visual analysis), and

$\bar{R}_{\max} (\text{cal})$ = the mean maximum vitrinite reflectance (calculated from automated analysis),

indicates no systematic bias between the calculated and determined mean maximum reflectances. The standard deviation of the regression line (0.02 percent R_0) shows that the PETAN program for interpreting reflectograms can yield accurate measurements of mean maximum vitrinite reflectance for typical bituminous coals.

B. The Determination of Petrographic Compositional Data

As was explained in Section IV, the use of a reflectogram as a quantitative measure of coal petrography is complicated primarily by the overlap in reflectance of components and by "misplaced"

Figure 56. A COMPARISON OF MEAN MAXIMUM VITRINITE REFLECTANCE (VISUAL) TO MEAN MAXIMUM VITRINITE REFLECTANCE (CALCULATED)

reflectance readings resulting from boundaries between components. Consequently, a simplistic curve-stripping analysis, based on unique reflectance distributions, is inadequate. Instead, a program employing both theoretical and empirical approaches was developed to interpret reflectograms.

The second section of the program PETAN begins by regenerating the normal distribution determined by VITFIT as a model of vitrinite reflectance population. This distribution is subtracted from the reflectogram and its population stored as a first approximation of the amount of vitrinite in the coal (Figure 57, step 2). The number of readings from zero reflectance to the mean of the vitrinite distribution (although no longer present in the reflectogram) is totaled as a first approximation of the liptinite content (SUMB). Then the number of readings from the mean of the vitrinite distribution to the end of the data array is totaled as an approximation of the inertinite content (SUMC). No subdivision of this region into inertinite and semi-inertinite is attempted because of the reflectance overlap of semifusinite and micrinite (Figure 40). The assumption is made that any semifusinite which might give reflectance readings within the vitrinite range is likely to behave in a manner similar to vitrinite. All higher reflecting material is classed automatically as inertinite.

The next section of the program involves correcting the liptinite approximation. The program first checks the vitrinite reflectance of the sample. If the reflectance is higher than 1.10 percent R_0 (MARR), the program assumes that the reflectance of the liptinite components is equal to or greater than the vitrinite reflectance. (See Figure 36.) When this happens, the program bypasses steps 6 through 11 (Figure 57)

Figure 57. A FLOW CHART OF THE LOGIC USED IN PETAN TO CALCULATE PETROGRAPHIC COMPOSITION FROM REFLECTOGRAMS

and assumes that the liptinite content is zero: The vitrinite population is adjusted upward to account for the reflectance readings skewed downward by boundary readings. If the vitrinite reflectance is lower than 1.10 percent R_0 , the program assumes that liptinite may be present in the sample.

Reflectance readings on epoxy, minerals, liptinite, coal/epoxy boundaries, and liptinite/other boundaries all report to the low reflecting region of the reflectogram and are included in SUMB. SUMB first is corrected by the subtraction of the average amount of epoxy in the briquette (23 percent). Next, the remainder is corrected for the amount of mineral present in the coal. This correction assumes that the volumetrically predominant minerals will be low-reflecting clays, quartz, and carbonates, which is true for most coals (Stach et al., 1975, p. 122). The correction factor is based on the dry ash and dry sulfur values of the proximate analysis of the sample. These weight percent data are calculated to volume percent using a modified version of the PARR formula (Figure 57, step 8). The volume percent of mineral in the coal is subtracted then from SUMB (Figure 57, step 9).

The formula used for calculating the mineral correction has at least one shortcoming: all of the sulfur in the coal is considered to occur as pyrite. Pyrite, with its high reflectance (R_0 of approximately 46 percent), does not contribute to the low-reflecting region of the reflectogram. Therefore, use of this equation results in a correction which is too high. For coals less than two percent total sulfur the discrepancy is insignificant, but for coals greater than this value, an equation which corrects only for low reflecting sulfate sulfur would be more accurate.

At this point the residual population is considered to represent the number of readings on liptinite, liptinite/coal boundaries, and coal/epoxy boundaries. As discussed in Section IV, the number of coal/epoxy boundaries is related directly to the number of particles intersected during the scan; this, in turn, is related to rank, composition, and size consist of the sample. From Table 17, it is evident that between four and six percent of the total readings on coal actually are coal/epoxy boundaries. The program corrects SUMB by subtracting the number of coal/epoxy boundaries expected for the sample rank. This correction makes the following assumptions: A standard preparation procedure is followed strictly. Only a small variation in coal particle size results from differing sample composition. The number of coal/epoxy boundaries is distributed between the three maceral groups in proportion to the volume of each group, and all these readings are contained in SUMB.

Finally, a correction is applied to adjust both the liptinite and the vitrinite approximations. As discussed in Section IV, boundary readings between two materials of differing reflectances will skew both populations toward one another. In this situation, the vitrinite distribution is skewed downward and an adjustment must be made to return these readings to SUMA (Figure 57, step 11).

The three transformed values now can be calculated as percentage data (Figure 57, step 12). The values are printed by PETAN on the second page of the printout, under the heading "Transformed Reflectogram Parameters" (Figure 58). Values are reported to one decimal place in order to lessen rounding errors, even though the decimal probably is not significant.

****PENN STATE COAL RESEARCH SECTION****
RAPID SCAN PETROGRAPHIC ANALYSIS

PAGE-2

SAMPLE DESIGNATION PSOC-276

DATE 11/ 9/1978

TRANSFORMED REFLECTOGRAM PARAMETERS

THE FOLLOWING PETROGRAPHIC DATA ARE VOLUME PERCENT

VITRINITE PERCENTAGE----- 77.1

LIPTINITE PERCENTAGE----- 4.6

INERTINITE PERCENTAGE----- 18.1

TOTAL REACTIVES----- 81.8

TOTAL INERTS----- 18.1

TOTAL REACTIVES-(MMC)----- 76.0

TOTAL INERTS-(MMC)----- 23.9

UNTRANSFORMED REFLECTOGRAM PARAMETERS

"INERT COEFFICIENT"----- 0.142

"INERT COEFFICIENT"-(MMC)---- 0.163

Figure 58. AN EXAMPLE OF PAGE TWO OF THE PRINTOUT FROM PETAN

The ability of PETAN to determine accurately the petrographic composition of coals from their reflectograms has been tested for 40 United States coals. The total "reactive" maceral contents (vitrinite plus liptinite) as determined by PETAN has been compared to equivalent values determined by the conventional visual technique. The results, shown graphically in Figure 59, show a fairly high degree of scatter. The regression equation:

$$\text{TRM}_{\text{vis}} = \text{TRM}_{\text{auto}} \times 1.0 \quad (19)$$

where,

TRM_{vis} = the total reactive macerals (visual analysis) and

TRM_{auto} = the total reactive materials (automated analysis)

shows no systematic bias in these results. The standard deviation about the regression line, 5.2 percent, indicates that the relationship between visual data and automated analysis data is not as favorable as desired.

Several factors may be contributing to the observed differences between the two techniques. First, it must be realized that automated reflectance systems utilize only one maceral property, i.e. reflectance, to effect a differentiation between macerals. The method of analysis, used by PETAN, involves the assumption that the three regions of the reflectogram are representative of the three maceral groups. A human operator, on the other hand, uses morphology and texture in addition to reflectance, to assign maceral group categories. The fundamental difference in analytical philosophy inevitably leads to some of the observed discrepancies between the two sets of results.

Regression Equation $TRM_{vis} = TRM_{auto} * 1.0$

Standard Deviation about the Regression Line = 5.2

* indicate individual data points

Numbers indicate number of superimposed data points

Figure 59. A COMPARISON OF TOTAL REACTIVE MACERALS (VISUAL) TO TOTAL REACTIVE MACERALS (AUTOMATED)

The proportioning by PETAN of semi-inertinite materials, especially semifusinite, may be a second source of discrepancy. In the United States it is common practice to assign arbitrarily 2/3 of the semifusinite to the "inert" maceral category; the automated system assigns the readings on reflectance only, again leading to possible differences.

Finally, the program PETAN must make a number of corrections to the reflectogram to account for misplaced or spurious (i.e epoxy) reflectance readings. These corrections, especially in the liptinite region, are subject to many assumptions. If, for instance, an unusual amount of epoxy is contained in the sample, or if the coal particle size is much smaller than expected, the liptinite estimation will be inaccurately high. Because liptinite generally is present in amounts of six percent or less and because it is subject to the greatest number of corrections, it has the largest coefficient of variation. However, even though its determination is subject to the largest variation, its contribution to the total "reactive" maceral content is relatively small.

Owing to the observed differences between visual and automated analysis, a completely new method of characterizing the petrographic composition of coal has been incorporated into PETAN. This method estimates the proportion of inertinite components in a manner which cannot be utilized directly in any existing coal performance prediction procedures. It simply determines the ratio of the number of readings higher in reflectance than the vitrinite population (SUMC in Figure 57) to the remainder of the readings minus the mineral content. The "Inert Coefficient", so determined, is printed as the

last item on page 2 of the PETAN printout under "Untransformed Reflectogram Parameters." Although highly simplistic and empirical, the "Inert Coefficient" avoids errors due to all the transformations necessary to estimate the proportion of inertinite. Providing the sample preparation technique has been standardized, the "Inert Coefficient" will yield a number indicative of the relative inertness of any subject coal. It also is useful in qualitative comparisons of the inertness of different samples. Section VII will discuss how the "Inert Coefficient" may be used directly in a modified coke strength prediction procedure.

In summary, Rapid Scan reflectograms can be interpreted quantitatively by the program PETAN. Vitrinite reflectance data determined from the reflectogram for average United States coals is as good as those determined visually. Petrographic compositional data determined from reflectograms do not always agree with visual results. Whether or not the observed differences are of sufficient magnitude to affect applied uses of coal petrographic data will be explored in the following section.

VI. APPLICATION OF RAPID SCAN PETROGRAPHIC COMPOSITIONAL DATA

A. Coke Strength Prediction

Historically, the greatest industrial application of coal petrographic data has been in the prediction of metallurgical coke properties from small coal samples. Of the various coke properties evaluated in this country, the ASTM Coke Stability Factor is the most recognized. The ASTM Stability Factor is a measure of the ability of the coke to resist abrasion in a standard drum test (ASTM, 1979). High coke strength (stability) is important for several reasons, including resistance to size degradation during handling, mechanical support of ore burden in the blast furnace, and maintenance of load permeability in the blast furnace. Some authors have related increased coke strength directly to increased blast furnace metal production (Matsuoka, 1975), which in turn relates to production profits. For modern blast furnace operation coal and coke engineers strive to produce strong metallurgical cokes with ASTM Stability Factors generally in excess of 55. Prior to the advent of applied coal petrography for coke strength prediction, coking coals were evaluated by actual carbonization testing, often requiring large samples (Wilson and Wells, 1950 p. 76).

The advent of techniques for predicting coke strength from coal petrographic data in the late 1950's greatly reduced the need for large-scale expensive carbonization tests. It also permitted coal petrologists to evaluate coal reserves from the limited sample sizes obtained from diamond drill cores. The success of the technique led

to larger and larger demands on coal petrographic laboratories for coal evaluation, and to the need for faster analyses (Thompson and Benedict, 1976).

In the United States, two systems for the prediction of coke stability, the United States Steel system (Schapiro et al., 1961) and the Bethlehem Steel system (Thompson et al., 1966), have found acceptance and widespread utilization. The two systems are similar in philosophy in that they employ a measure of the properties of the vitrinite in the coal (as a result of coalification) and a measure of the coal's petrographic composition to predict its behavior in the by-product coke oven. The primary difference between the two systems is that Bethlehem Steel recognizes two populations of vitrinite in coal, one of which may act partially as an inert maceral during carbonization. The remainder of this section will deal with utilizing data derived from the Rapid Scan in the United States Steel system of coke strength prediction.

The United States Steel system requires three types of information for a coke stability prediction: a V-type distribution, the total content of "reactive" and "inert" macerals, and a volume percent mineral content. These three analyses are employed to calculate two coal indices: the "composition-balance index" (CBI) and the "strength index" (SI). With these indices, the coal petrographer can predict the coke stability. It is important to note that certain variables in coke oven operation (i.e. particle size, bulk density, and heating rate) can have significant effects on the characteristics of the resulting coke and should be taken into consideration.

The composition-balance index is calculated from the total "inert" components, both organic and inorganic, present in the coal. The volume of mineral matter is calculated from the ash and sulfur data generated from a proximate analysis. This figure is included with the "inert" macerals to give the total "inert" components. The total "inert" content, in ratio form with the total "reactive" content, is then compared to the optimum inert/reactive ratio (the ratio which produces the strongest coke) calculated for the specific rank of the sample. The most favorable composition-balance index is equal to 1.0, with values less than 1.0 indicating an excess of "reactive" components, and values above 1.0 indicating an excess of "inert" components.

The strength index is related to the reflectance (as an indicator of structural and chemical properties) of the vitrinite in the coal and the maceral composition. The strength index increases with increasing vitrinite reflectance up to a vitrinite mean maximum reflectance of 1.90 percent. At this point, the ability of the vitrinite to bond particles and form strong coke walls begins to decrease with increasing rank, and the strength index decreases.

An experiment was devised to test the feasibility of employing data obtained by the program PETAN, from Rapid Scan reflectograms, to predict ASTM coke stability using the United States Steel method. A suite of 21 single-coal samples was compiled, for which 500-pound carbonization tests and ASTM coke stability tests were available. These 21 coals were subjected to conventional visual maceral and reflectance analysis and Rapid Scan petrographic analysis. Both sets of data were compiled and run through The Pennsylvania State University,

Coal Research Section's computerized coke strength prediction program (COKE4). COKE4 processes the input variables, calculates the composition-balance index and the strength index, and prints the results as a one-page printout. The composition-balance index and the strength index then are used to solve for the ASTM coke stability using an empirically derived set of curves (Schapiro et al., 1964).

The resulting coke strength predictions from the Rapid Scan data (Table 20) are shown, plotted against the experimentally determined values, in Figure 60. The standard deviation about the regression line is seven stability points, an unacceptably high deviation. This may indicate that Rapid Scan petrographic data are not appropriate for the prediction of ASTM coke stability. However, when the visually-derived petrographic data for the same coals were used to predict the coke strength, the prediction was not significantly better (Figure 61). In this data set the standard deviation about the regression line is six stability points, still an unacceptably high scatter.

There are at least three possible arguments which can be advanced to explain the observed deviations. The first is that the experimental coke stability determinations might be inaccurate. ASTM specifies that the standard deviation of stability tests should be 1.5 stability points (ASTM, 1979); however, interlaboratory "round robin" analyses have shown deviations as high as 10 stability points (Gray, 1979). A second possible factor affecting the prediction accuracy would be that both the automated and the visual petrographic data were in error. It can be shown that coal petrographers at the United States Steel Corporation consistently count higher inertinite maceral contents than do petrographers at The Pennsylvania State

Table 20

Comparison of Predicted Coke Stability (United States
Steel Method) to Measured Coke Stability

Sample Number	Measured ASTM Coke Stability (500 lb. oven)	Predicted ASTM Coke Stability (visual data)	Predicted ASTM Coke Stability (automated data)
CBCS-132	34	35	32
CBCS-133	38	45	49
CBCS-204	39	44	42
CBCS-214	39	42	45
CBCS-269	44	43	49
CBCS-177	45	52	52
CBCS-48	45	45	42
CBCS-205	46	42	55
CBCS-207	47	57	29
CBCS-176	48	58	58
CBCS-143	48	35	50
CBCS-112	49	46	48
CBCS-105	50	52	59
CBCS-142	51	61	43
CBCS-240	52	52	56
CBCS-72	52	50	38
CBCS-242	56	51	56
CBCS-155	57	60	65
CBCS-227	57	55	62
CBCS-178	58	54	64
CBCS-144	58	59	61

Figure 60. A COMPARISON OF PREDICTED COKE STABILITY (AUTOMATED DATA) TO MEASURED COKE STABILITY

Figure 61. A COMPARISON OF PREDICTED COKE STABILITY (VISUAL DATA) TO MEASURED COKE STABILITY

University (Hoover, In Preparation). This could explain some, but probably not all, of the observed deviations. The final, and probably the most important factor which could have affected the predictions, is the coals themselves. The fact that several coke stability prediction formulae exist for coals from different basins or coals which contain large amounts of high reflecting telocollinite ("pseudo-vitrinite"), indicates that the United States Steel system is not universally applicable. In addition, these test coals contained large amounts of semifusinite whose coking properties are not well understood, and are not accounted for adequately in any prediction technique. The utilization of a different prediction technique may have improved these results.

In summary, Rapid Scan petrographic data can be utilized directly in the United States Steel Corporation system of coke strength prediction. The accuracy of the predictions using automated data will have to be tested on a larger suite of well-characterized coals.

B. Evaluation of Coal Blends

Because of a shortage of single coals which can produce superior cokes, most by-product coke ovens utilize a blend of two or more coals. In this manner, high, medium and low volatile coals are blended to produce an optimum ASTM coke stability, while also optimizing economic considerations and operating parameters. Typical production blends contain 60 to 85 percent high volatile coal with 15 to 40 percent low and medium volatile coals (Gray et al., 1978).

One need of coke plant operators is a method of checking coal blends to ensure that the "mix" being charged into the ovens contains

the proper proportions of the component coals. Various approaches, such as marking coals of different rank by color, or determining bulk-blend chemistry, have not proved entirely satisfactory (Graham, 1979). Visual reflectance analysis has been used with some success; however, it is not a rapid test, since 500 or more reflectance readings are necessary to define the various vitrinite populations in the blend (Stach et al., 1975, p. 274).

The ability to collect many reflectance sample points in a short time suggests that the Rapid Scan could be an ideal instrument for characterizing blends. Early work by Hartmann (1976), to test this idea, showed that a Rapid Scan reflectogram of a two-coal blend displayed two characteristic vitrinite peaks.

Blends also were detected during routine scanning of samples. The reflectance profile in Figure 62 is of a sample which, when received, was labeled as a medium volatile bituminous coking coal. The gross-chemical volatile content, as determined in a proximate analysis, confirmed the sample as a medium volatile bituminous coal. However, the Rapid Scan reflectogram showed that the sample was predominantly low volatile bituminous coal with small amounts of high and medium volatile bituminous coals.

In order to understand better the resolution with which the Rapid Scan can characterize blends, a series of experiments was carried out on several three-coal blends prepared in the laboratory. The petrographic properties of the coals are shown in Table 21. These samples were selected because of their relatively high, and similar, vitrinite contents.

Figure 62. A REFLECTOGRAM FROM A THREE-COAL BLEND

Table 21

Petrographic Composition of Coals Used for Blending

Petrographic Parameter	Sample Number		
	PSOC-702	PSOC-720	PSOC-688
Maceral content, volume %, mineral free			
% Total Vitrinite	91.6	91.3	91.7
% Fusinite	1.4	1.8	0.7
% Sclerinite	3.8	2.6	4.1
% Macrinite	0.1	0.3	0.7
% Micrinite	1.0	3.4	1.8
% Exinite	1.8	0.6	0.0
% Resinite	0.3	0.0	0.0
Vitrinite Reflectance, mean maximum, %	0.82	1.41	1.63

The individual coals were scanned on the Rapid Scan, and their reflectograms are depicted in Figure 63. By generating individual reflectograms, it is possible to combine the profiles (using the computer program CBLEND (Appendix VI)) into simulated reflectograms. The program CBLEND takes three individual coal reflectograms and adds them together in proportions specified by the operator, creating a simulated blend reflectogram. This enables comparisons to be made between simulated and actual experimental blend reflectograms.

An example of a simulated blend of the three coals in equal proportions is shown in Figure 64, along with the comparable experimental blend reflectogram. From these profiles it is apparent that relative peak heights are not a sufficient criterion to interpret blend

Figure 63. THE REFLECTOGRAMS OF THE THREE
INDIVIDUAL COALS USED FOR BLENDING
EXPERIMENTS

Figure 64. ACTUAL AND SIMULATED REFLECTOGRAMS
FROM BLENDS OF THREE COALS IN EQUAL
PROPORTIONS

reflectograms. Even though both the simulated and actual blends contain approximately equal proportions of vitrinite from each of the coals, the peak of the vitrinite from the low volatile coal is depressed in comparison to the other two. Consequently, an interpretation that there is less low volatile coal in the blend, made on the basis of relative peak height, would be in error. Two factors lead to the depression of the low volatile bituminous coal peak. The first is a result of the increased bireflectance of the higher rank low-volatile vitrinite. Increased bireflectance will have the effect of broadening the average random vitrinite reflectance population, thus lowering the peak height. The second factor is related to the increased friability of low volatile bituminous coals. Such coals usually are characterized by a smaller particle size distribution in the sample briquette (Table 17) and hence, record more epoxy coal boundary reflectance readings (Section IV). These boundary readings come at the expense of reflectance readings on coal alone, and result in a lower vitrinite peak height.

At an earlier stage of this research it seemed feasible that a quantitative data interpretation program employing curve-stripping techniques, similar to PETAN, could be developed for the interpretation of blends. The peak height effect, described above, could be overcome by employing a rank-based adjustment. However, a more serious problem exists in deconvoluting superimposed blend reflectance profiles. Stated simply the semi-inertinite and low-reflecting inertinite components in a lower rank coal may have the same reflectance as the vitrinite of a higher rank coal (Figure 36). Therefore, in a reflectogram of a blend, two components with very

dissimilar technological properties would, on the basis of reflectance alone, be classified together. As a consequence, deconvolution techniques do not appear to be applicable to the interpretation of blend reflectograms.

An alternative approach is to employ an overall curve-matching technique. This type of method, already developed for single coals, (Frompovicz, 1978) entails the comparison of the sample blend reflectogram, visually or automatically, with computer stored blend reflectograms of the same constituent coals. Profile matches can be based on blend analyses from the actual sample blends, or on simulated computer-generated blends from the reflectograms of the individual coals. It should be noted, however, that simulated blends may not match actual sample blends exactly, due to differences in the preparation of the individual briquettes.

A series of scans on three-coal blends was carried out to test the sensitivity of Rapid Scan reflectograms to minor changes in blend proportions. The presence of three coals in a briquette increases the complexity of the sample above that of a single-coal preparation. The 50,000 data points routinely used to characterize single coals, were insufficient for blends, and, after the reflectograms of several blends had been analyzed, it was concluded that at least 150,000 data points were essential for the production of stable three-coal reflectograms.

Figure 65 shows two actual reflectograms of blends along with simulated reflectograms generated by the program CBLEND. Reflectograms a. and b. show the three coals listed in Table 21 combined in the following proportions:

Actual Blend
Reflectograms

Simulated Blend
Reflectograms

Figure 65. ACTUAL AND SIMULATED REFLECTOGRAMS OF SIMILAR BLENDS

PSOC-702 (high volatile A) - 70 percent

PSOC-720 (medium volatile) - 15 percent

PSOC-688 (low volatile) - 15 percent

Reflectograms c. and d. are of a second very similar blend, where the proportion of PSOC-702 was decreased to 65 percent and the proportion of PSOC-688 was increased to 20 percent. In reflectograms A and B the vitrinite peak of PSOC-702 is significantly truncated by the maximum frequency limit of the reflectogram, whereas in reflectograms C and D the vitrinite peak of PSOC-702 is almost within the scale.

The noticeable relative decrease of the peak height is evidence of the decrease of PSOC-702 in the blend. There also is a less noticeable increase in the size of the vitrinite peak for PSOC-688, as should be expected. The simulated reflectograms of blends also change in nearly the same manner, responding to the different coal proportions.

In summary, it is evident that the Rapid Scan is a useful tool for examining coal blends. Reflectograms of blends are sensitive to changes in coal proportions as low as five percent and could be used in quality control to monitor proportions in industrial operations. Quantitative interpretations of reflectograms of coal blends are hindered by reflectance overlap and differential peak heights; however, simulated reflectograms can be used in evaluating the proportions of the different coals by profile matching.

C. Application to Washability Studies

When coal is mined in either underground or in surface mines, the resulting product, known as run of mine (ROM) coal, often does not meet market specifications. Further processing of the product

may be as simple as size reduction, or may employ complex preparation procedures, generally aimed at reducing the mineral impurities. The techniques applied to improving coal quality, termed physical beneficiation, rely on the following properties: hardness, density, and particle surface characteristics. Fundamental differences in these properties, between the impurities in coal and the carbonaceous material, allow various processing schemes to separate the two phases.

Historically, physical beneficiation was applied primarily to coals destined for the production of metallurgical coke. Currently, these techniques are being used increasingly for improving coal quality for steam generation and are being evaluated for use in the synthetic fuels industry (Luckie, 1979). A result of physical beneficiation that is sometimes overlooked is that, as well as removing coal impurities, it also segregates (and sometimes removes) specific coal macerals. The fundamental differences in hardness, density, and surface properties which allow the separation of impurities apply also to coal macerals. Generally vitrinite is concentrated in the low density fractions; whereas inertinite macerals with high densities report to the refuse or middlings products (Bayer, 1960; Benedict, 1962). Detailed studies to determine the precise influence of physical beneficiation on petrographic composition have been hindered by lack of industrial interest, and by the expense of large numbers of petrographic analyses. Application of Rapid Scan analysis to studies of the effects of physical beneficiation could be one method of reducing the cost of such programs.

Laboratory-scale studies to evaluate the effects of physical beneficiation on coal composition, have been supported at The

Pennsylvania State University, first by the Federal Office of Coal Research and later by the Energy Research and Development Administration and the Department of Energy. The conceptual design of these projects was to selectively crush, size and fractionate by density separations various coals in a manner which might approximate methods feasible in commercial preparation plants. This design, shown schematically in Figure 66, had the potential to generate approximately 40 subsamples from each coal studied. The run of mine coal was separated into three hardness fractions based on its initial response to crushing and sizing. Each hardness fraction was separated into size fractions, two of which ($3/8$ inch by $1/4$ inch and $1/4$ inch by 16 mesh) were further separated into density fractions (the number of fractions and the specific gravity of the fluid varied depending on the sample characteristics).

Two coals were selected for testing from a group of 41 which had been processed by this procedure (Spackman et al., 1976). The object of this experiment was to test the application of the Rapid Scan in evaluating changes in coal composition as a result of density fractionation. Subsamples from group A and group B on Figure 66 were subjected to standard Rapid Scan analyses. Data generated by the Rapid Scan included reflectograms and petrographic analyses done with the program PETAN.

Figure 67 shows the reflectogram from the initial channel sample of PSOC-134 (Pratt seam, Alabama), and the reflectograms from the density fractions of group A and B subsamples. In both groups, the subsamples were arranged in order of increasing density of separation.

Several trends are evident in this figure. In group A, the area of the vitrinite peak decreases markedly with increasing density

Figure 66. A FLOW CHART OF THE COAL FRACTIONATION PROCEDURE (after Spackman et al., 1971)

Figure 67. REFLECTOGRAMS FROM THE DENSITY FRACTIONATION OF PSOC-134

of separation, indicating that the heavier fractions contain less vitrinite. Also, the reflectogram region higher in reflectance than the vitrinite peak, increases with increasing density of separation (up to the sink fraction). This indicates that as density of separation increases and the vitrinite decreases, there is, conversely, an increase in inertinite components. This trend is apparent in all the density fractions except the 1.40 sink fraction. In this reflectogram, the low-reflecting region has increased, indicating that this sample has a much higher mineral content; the vitrinite peak has increased and the inertinite region decreased, reversing the trend shown by the first three density fractions.

The trends evident in the group B subsamples are very similar to those in group A. The relationship between the vitrinite and inertinite regions is even more pronounced than in group A, indicating that even more enrichment of inertinite macerals has occurred. The primary difference between the groups is that in the 1.40 sink fraction of group B there is no apparent reversal of the inertinite enrichment trend, as in group A. This characteristic may be obscured by the greatly enriched mineral content present in the group B sink fraction (60 percent ash), as evidenced by the large increase in the area of the low reflecting region of the reflectogram.

Although qualitative information can be observed in Figure 67, it also was important to investigate whether quantitative petrographic measurements generated by the Rapid Scan would be comparable to visually derived data. The reflectograms shown in Figure 67 were analyzed by PETAN, and these data, together with the visual petrographic analyses are presented as part of a complete density fractionation analysis in Table 22. Both sets of petrographic analyses

Table 22
Density Fractionation Analysis of PSOC-134

		Pratt seam, Alabama										Medium Volatile Bituminous							
Specific Gravity		Direct Analysis										Cumulative Float							
		Wt. % Yield	Vol. % Yield	Ash Wt. %	Sul. Wt. %	Vis. Vit. %	R. S. Vit. %	Vis. In. %	R. S. In. %	Vis. Lip. %	R. S. Lip. %	Wt. % Yield	Vol. % Yield	Vis. React. %	R. S. React. %	Vis. In. %	R. S. In. %	Ash Wt. %	Sul. Wt. %
Group A	0.00 X 1.28	40.5	43	2.7	0.65	86.8	83	13.2	17	0.0	0.0	40	43	87	83	13	17	2.7	0.65
	1.28 X 1.30	13.8	15	4.3	0.74	81.8	72	18.2	28	0.0	0.0	54	58	86	80	14	20	3.1	0.67
	1.30 X 1.40	28.3	28	7.0	1.02	56.2	44	43.8	56	0.0	0.0	83	86	76	68	24	32	4.4	0.79
	1.40 Sink	17.4	14	34.9	4.38	68.6	55	31.4	45	0.0	0.0	100	100	75	66	25	34	9.7	1.42
Group B	0.00 X 1.28	5.5	7	2.6	0.58	83.2	82	16.8	18	0.0	0.0	6	7	83	82	17	18	2.6	0.58
	1.28 X 1.30	7.5	9	3.5	0.50	58.8	56	41.2	44	0.0	0.0	13	16	70	67	30	33	3.1	0.53
	1.30 X 1.40	23.3	25	6.4	0.68	44.8	42	55.2	58	0.0	0.0	36	41	55	52	45	48	5.2	0.63
	1.40 Sink	63.7	59	60.9	3.41	57.5	55	42.5	45	0.0	0.0	100	100	57	54	43	46	40.7	2.40

plotted in Figure 68. The trend of decreasing vitrinite content, with increasing density of fractionation, observed from Figure 67, is evident in both the Rapid Scan and the visual quantitative data. The agreement between the two sets of data is excellent for the group B subsamples, and although offset, shows the same trend for the group A subsamples. Note also that in Figure 68 the two sample groups respond differently to the same fractionation procedure. The vitrinite contents of group A decrease almost linearly with increasing density, whereas for group B the decrease is more exponential in shape. This may indicate that in group A the vitrinite and inertinite macerals are in close association, requiring higher densities to cause the particles to sink. In group B there would be fewer complex particles, and the more dense inertinite macerals could sink at lower fractionation densities. This fundamental difference in particle structure may indicate why the Rapid Scan results are more accurate for the petrographically simpler group B subsamples.

The second coal tested (PSOC-102, Pittsburgh seam, Pennsylvania) responded quite differently to the fractionation procedures. The reflectogram from the initial channel sample, and those of the density fractions, are shown in Figure 69. Unlike in Figure 67, the vitrinite peak retains approximately the same area with increasing density of separation up to the 1.40 sink fraction. Although minor changes are apparent in the high-reflecting inertinite region, they are not nearly as pronounced as those for PSOC-134. This would indicate, qualitatively, that there is little change in the petrographic composition of this coal as a result of density fractionation. Only in the 1.40 sink fraction, which is high in mineral content, does there appear to be any inertinite enrichment.

Figure 68. THE CHANGE IN SAMPLE COMPOSITION AS A RESULT OF DENSITY FRACTIONATION

Figure 69. REFLECTOGRAMS FROM THE DENSITY FRACTIONATION OF PSOC-102

A complete set of fractionation data, including visual and Rapid Scan petrographic analyses, is presented in Table 23. Figure 70 shows these data plotted in the same format as Figure 68. The small petrographic compositional change observed in Figure 69 is confirmed by the low slopes of trends plotted in Figure 70. There is even an increase in total "reactive" macerals of the group B subsamples in the 1.28 by 1.30 fraction. As with PSOC-134, the trends of the Rapid Scan petrographic data follow those of the visual data.

From these results, there appears to be a fundamental difference in the maceral associations present in the two coals. In PSOC-134 both groups of samples showed considerable inertinite enrichment with increasing density of separation. The lack of inertinite enrichment in PSOC-102 may be related to two different phenomena. One may be that the inertinite macerals are distributed evenly throughout the coal, and crushing has not destroyed this association. If this were the case, most of the coal particles would have similar densities, and little differentiation would be gained by density fractionation. The other possibility may involve the distribution of the low-density liptinite macerals in this coal. If the liptinite was associated intimately with the inertinite macerals, the resulting particles could have bulk densities similar to that of vitrinite. Once again, if this were the case, little differentiation could be gained by density fractionation.

These experiments have shown that the Rapid Scan is a useful device for studying petrographic compositional changes as a result of physical beneficiation. Qualitative changes easily can be observed in the samples' reflectograms. Quantitative petrographic data can be

Table 23
Density Fractionation Analysis of PSOC-102

		Pittsburgh seam, Pennsylvania										High Volatile A Bituminous							
Specific Gravity		Direct Analysis										Cumulative Float							
		Wt. % Yield	Vol. % Yield	Ash %	Sul. %	Vis. %	R. S. %	Vis. %	R. S. %	Vis. %	R. S. %	Wt. % Yield	Vol. % Yield	Vis. React. %	R. S. React. %	Vis. In. %	R. S. In. %	Ash %	Sul. %
Group A	0.00 X 1.28	22.5	24	2.5	0.91	83.0	87	15.4	10	1.6	3.0	23	24	85	90	15	10	2.5	0.91
	1.28 X 1.30	55.7	57	4.0	1.12	80.2	81	17.8	16	2.0	3.0	78	81	82	84	18	16	3.6	1.06
	1.30 X 1.40	13.9	14	9.3	1.77	78.2	78	20.0	18	1.8	4.0	92	95	80	82	20	18	4.4	1.17
	1.40 Sink	7.9	5	49.1	5.63	57.4	57	37.8	37	4.8	6.0	100	100	62	63	38	37	8.0	1.52
Group B	0.00 X 1.28	26.6	27	2.5	0.93	80.4	81	15.4	13	4.2	6.0	27	27	85	87	15	13	2.5	0.93
	1.28 X 1.30	48.7	50	4.2	1.05	86.0	84	11.0	11	3.0	5.0	75	77	89	89	11	11	3.6	1.01
	1.30 X 1.40	17.8	18	9.0	1.51	81.4	80	16.6	16	2.0	4.0	93	95	83	84	17	16	4.7	1.10
	1.40 Sink	6.9	5	51.9	3.22	64.8	60	31.8	35	3.4	5.0	100	100	70	65	30	35	7.9	1.25

Figure 70. THE CHANGE IN SAMPLE COMPOSITION AS A RESULT OF DENSITY FRACTIONATION

generated on suites of samples, although its accuracy may be affected by the concentration of petrographically complex particles, and by large amounts of minerals.

Data obtained by studying the changes in petrographic composition brought about by physical beneficiation may have application in the carbonization and synthetic fuels industries. An application of this type of data is shown in Figure 71. In this figure, the fractionation data for the group A subsamples, reported in Table 22, have been plotted in a standard "washability-curve" format. Laboratory data plotted in this manner can be used to predict the product characteristics of a given fractionation operation.

Suppose that this coal will be used for the production of metallurgical coke. By utilizing the United States Steel Corporation method of evaluating and predicting ASTM coke stability, one can determine that this coal should produce a maximum ASTM coke stability of 57, if it contained 82 percent vitrinite (MMF). Starting with a product specification of 82 percent vitrinite, one can construct the prediction matrix shown by dashed lines in Figure 71. The intercepts of the dashed lines on the four abscissae show that, to produce a product with 82 percent vitrinite, it would be necessary to float the sample at a specific gravity of 1.35. They also indicate that the product would contain 3.7 percent (by weight) ash and 0.75 percent (by weight) sulfur. The intercept on the ordinate indicates that this operation would yield 72 percent (by weight) of the feed material. These product and operation specifications are within the range of commercially available technologies. Whether or not this would be a

Figure 71. THE WASHABILITY CURVES FOR PSOC-134

practical undertaking would be controlled by the process economics and yields.

In the production of liquid synthetic fuels from coal, it is desirable to maximize the amount of vitrinite in the feed material (Davis et al., 1976). In the sample illustrated in Figure 71 (dotted line) a maximum of 86 percent vitrinite could be achieved by floating the coal at a specific gravity of 1.18. This type of processing, however, probably would not be commercially feasible due to the low specific gravity of operation (1.18) and the very low yield (40 percent).

In conclusion, the Rapid Scan can be used for qualitatively and quantitatively evaluating petrographic compositional changes resulting from physical beneficiation. It may be used to evaluate and monitor coal blends, either as a quality control device or in "post facto" investigations. Its utility for predicting ASTM coke stability, using current prediction techniques, will require more investigation with better experimental control of the reference samples. In general, it has been shown that the Rapid Scan is a rapid and sensitive device for evaluating coal petrographic composition, and should prove a useful tool to the industrial coal petrographer.

VII. SUMMARY, CONCLUSIONS, AND SUGGESTIONS FOR FUTURE RESEARCH

A. Summary and Conclusions

The following were the basic objectives of this research project:

1. To evaluate the need for, and the feasibility of, employing automated reflectance microscopy for coal petrographic analysis.
2. To develop a flexible and relatively inexpensive instrument for automated analysis.
3. To investigate the precision and resolution of both the instrument and the technique.
4. To examine, explain, and quantify the frequency distributions generated by the instrument.
5. To examine the implications and practical applications of the data.

Each of these objectives was discussed in the preceding sections and the following is a discussion of some of the salient results and conclusions.

Increased coal utilization has accelerated the demand for basic knowledge about coal properties and their relationships to coal processing. Coal petrographic analysis has been a useful method of coal evaluation and its applications can be expected to expand. Demands on present trained personnel, and difficulties in standardizing inter-laboratory analyses have stimulated interest in rapid, objective systems of petrographic analysis.

A reflectogram generated by automated reflectance microscopy changes in response to three geologic factors. The proportion,

distribution, and size of macerals (functions of conditions in the peat stage), are represented by populations in more or less distinct parts of the reflectogram. The contributions of minerals, introduced into the coal either as detrital material or as syngenetic or epigenetic growths, also can be assigned broadly to certain proportions of the reflectogram. Finally, the level of metamorphism (rank change) which the peat or coal material has undergone is represented quite precisely by the position of the vitrinite peak on the reflectance scale. Some previously published studies indicated that automated reflectance might be one method of differentiating between bituminous rank coal macerals.

This project showed that the reflectance of the liptinite group macerals, the vitrinite group macerals, and fusinite all were separated well. However, owing to their overlapping reflectance distributions, it is impossible for the instrument to discriminate between micrinite and semifusinite.

The various reflectance studies also have shed some light on the geologic history of the test coals. The mean reflectance values for fusinite showed considerable scatter and no distinct trend with rank (Figure 36). If fusinite originated from a unique biochemical process its optical properties should be more uniform. Thus, the high degree of scatter supports the multiple (or varied) origin theory for fusinite. These results also confirm previous observations that the optical properties of fusinite may show little change with increasing coalification. The mean reflectance values for sporinite show a convergent coalification path to that of vitrinite, up to a vitrinite reflectance of 1.10 percent mean average random reflectance. The

higher rank test coals have sporinite reflectances in excess of that of the vitrinite, suggesting divergent coalification.

Mean average random reflectance showed a linear relationship to mean maximum vitrinite reflectance for coals from western Pennsylvania. This implies that the development of pressure-induced anisotropy was uniform across the basin. Uniform vitrinite anisotropy may indicate similar depth of burial for the different rank coals.

During this project three generations of automated reflectance microscopes were developed and evaluated. Rapid Scan Version I was assembled from microscope and computer components already available at The Pennsylvania State University, Coal Research Section. It suffered from poor precision of reflectance measurement due to the optical-electronic shortcomings of the system hardware. Rapid Scan Version II incorporated an advanced microscope photometer and a more modern computer. In this system the precision of the reflectance measurement was limited by the continuous stage movement. The Rapid Scan Version III system utilizes a digital stepping stage to alleviate this problem and was developed at a system cost of less than \$50,000. It was assembled from commercially available hardware components, and is, in essence, a software-based instrument. Greater than 90 percent of the software was written in Fortran, limiting processor dependence and increasing system flexibility.

An evaluation of the instrument indicated that the primary controls on system precision were luminous flux and signal integration time. Effects on precision which stemmed from loss of focus and improper system calibration are controllable by operator diligence. The instrument precision for individual readings (as defined by

$\pm 2\sigma$) was found to be approximately ± 0.01 percent reflectance. Design considerations incorporated into the Version III system set the measurement precision at approximately ± 0.02 percent reflectance.

Although this work has shown that macerals can be differentiated by reflectance, reflectograms from the Rapid Scan resolved only two distinct reflectance populations (epoxy binder and vitrinite). Six factors were identified which affected the degree of maceral resolution in a reflectogram. The effect of size (and, hence, boundary readings) had, by far, the greatest influence on reflectograms.

Despite problems of resolution, reflectograms were shown to be highly reproducible, and the program PETAN was developed to interpret these curves. Results showed that PETAN-derived vitrinite reflectance measurements were quite similar to visually-produced values; the latter required approximately five times the amount of time to complete. Vitrinite reflectance values determined by the automated system may be better for the prediction of the technological behavior of coals because they represent a mean value for all the vitrinite types present. A comparison of total reactive macerals derived by PETAN to those done visually showed a standard deviation of 5.2 percent.

Data generated from PETAN were used to predict ASTM coke stability indices, employing the United States Steel Corporation prediction technique. The accuracy of the predictions was only marginally poorer than those done with visually-derived data. A larger, more controlled, sample set will be necessary for a comprehensive test of the system's capability to predict such coal behavior.

The Rapid Scan also was used for the detection and quantification of coal blends. It was shown that peak heights are insufficient for

quantifying blends, and that deconvolution techniques are not applicable due to overlaid reflectance populations. Overall curve comparison techniques using simulated blend reflectograms were shown to detect variations in blend composition as low as five percent.

Rapid Scan reflectograms and data from PETAN were used to investigate changes in coal composition resulting from density fractionations of coal samples. It was possible to detect differences in the reflectograms visually. Data from PETAN compared well with conventional maceral analysis data generated from the same samples.

In conclusion, it is the author's belief that this program successfully has achieved its initial research objectives. Automated reflectance microscopy is likely to become a useful tool in industrial laboratories where large numbers of samples are processed routinely. The technique also is potentially useful in research institutions as a screening tool to investigate large sample sets. In the future, automated reflectance systems will continue to be improved as new hardware becomes available, and new methods of data reduction are conceived.

B. Future Work

Several hardware improvements could be developed which may increase marginally the resolution of the instrument. As noted above, luminous flux is the primary control of measurement precision. If it were possible to increase the light intensity, either with higher intensity incandescent lamps or laser sources, one could improve the reliability of each reflectance measurement. Such a gain may improve the resolution of the reflectance populations of individual macerals in a reflectogram.

A second improvement would be the installation of a signal feedback system in the lamp source, to monitor and correct for variations in the intensity of light. Galbraith et al. (1975) and Gray et al. (1979a) have reported that such devices used in photon-counting photometers improve signal stability. Although drift generally is not a problem during a 15 minute Rapid Scan analysis, significant fluctuations in line voltage occasionally have affected stability.

Another hardware improvement which could have a significant impact on resolution would be a reduction in the size of the sampling spot (perhaps by one order of magnitude) without loss of measurement precision or increase in analysis time. Image analysis systems, with their high pixel density, approach this goal; however, they lack the precision of reflectance measurement. Optical systems are not likely to achieve these parameters owing to problems of light diffraction by small measuring slits and insufficient photon statistics.

Another area for future development is new methods of utilizing the data from current systems. As discussed in Section VI, one application of automated reflectance microscopy is a means of monitoring coal quality. Curve shape can be matched visually, or by algorithm, against target shapes so that the operator rapidly can detect differences between samples. Hampson et al. (1980) demonstrated that this approach enables operators to standardize and improve coke quality on a day-to-day basis at the Dominion Foundries and Steel Company of Canada. The United States Steel Corporation also has reported that it expects to utilize reflectograms in a similar manner (Gray, 1979).

Other future work could be concerned with the investigation of new ways of utilizing reflectograms in a quantitative manner.

It has been shown that, unlike visual petrographic analysis, reflectograms are very reproducible. Much of the discussion in Sections V and VI was about the generation of coal composition data which could be compared directly to conventionally measured values. A more direct approach would be to derive new compositional parameters directly from the reflectogram.

Consider the prediction of coke strength properties as an example. Formulae to do this were derived empirically by comparing measured coke stabilities to visual coal petrographic data. Figure 72 shows the relationship between coke stability and vitrinite reflectance for single coals varying in rank from high volatile B bituminous to low volatile bituminous. The relationship shows that for coals greater than approximately 0.85 percent reflectance, vitrinite reflectance alone explains much of the variation in the data set. A second parameter which could refine the relationship is a measure of the amount of inertinite present in the coal. It was suggested in Section V that a possible measure of the relative amount of "inert" to "reactive" macerals directly derivable from the reflectogram is the "Inert Coefficient." This value is a ratio of the number of coal reflectance readings greater than the vitrinite reflectance to the number of readings less than and including the vitrinite population. With a large enough data set one possibly could combine the Rapid Scan vitrinite reflectance values with the "Inert Coefficient" and generate an empirical coke strength prediction formula.

In a similar manner, equations also could be developed for predicting the conversion of coals into clean fuels. Statistical studies already have shown that the same two parameters (vitrinite reflectance and the proportion of inertinite or total reactive macerals) together

Figure 72. THE RELATIONSHIP BETWEEN MEAN MAXIMUM VITRINITE REFLECTANCE AND ASTM COKE STABILITY FOR SINGLE COALS

with the total sulfur in the coal can be used to predict coal conversion (Yarzac et al., 1980). The Rapid Scan also can measure the amount and size distribution of the pyrite (a major source of sulfur in coal and a potential catalyst) present in different coals (Kuehn and Davis, 1979). These parameters, together with the vitrinite reflectance and an index of petrographic composition, may enable Rapid Scan-derived data to be used directly for predicting coal conversion.

Finally, Rapid Scan is a rapid objective system for measuring coal petrographic composition. The use of only one criterion for classifying macerals (reflectance) limits the systems ability to generate compositional data which duplicates visual results. The improvement and utilization of data generated by automated systems are open to the imagination and creativity of all coal scientists, and should not be constrained by present paradigms.

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APPENDIX I
Computer Program PETR06
(Fortran IV)

```

C      PROGRAM PETRO VERSION 5 -- PETRO6 1/15/80
C      THIS PGM IS THE BASIC DATA ACQUISITION PGM FOR
C      THE RAPID SCAN SYSTEM-IT OPERATES THE DIGITAL STAGE
C      AND CALLS THE SUBROUTINES MOVE2 & MOVE3
C
C      AUTHOR DAVID S. HOOVER SEPTEMBER 1978
C
C      *****
C
C      MODULE-1
C
C      COMMON IARR(1024),RARR(20),ARR1(5)
C      INTEGER CH
C      DO 2 I=1,1024
2      IARR(I)=0
C      DO 731 ITR=1,5
731      ARR1(ITR)=' '
C      DO 709 IFE2=1,20
709      WRITE(4,915)
C      WRITE(4,705)
706      FORMAT(F12, '--- RAPID SCAN DATA ACQUISITION PROGRAM---')
C      DO 707 IFE1=1,5
707      WRITE(4,915)
C
C      MODULE-2
C
C      WRITE(4,701)
701      FORMAT( ' *** THIS PORTION IS TO ENTER THE OPERATING DATA ***')
C      DO 702 ITE=1,3
702      WRITE(4,915)
C      WRITE(4,700)
700      FORMAT( ' ENTER SAMPLE ID (10CHAR. MAX)      ', $)
C      READ(4,752)ARR1(1),ARR1(2)
752      FORMAT(2A6)
C      WRITE(4,710)
710      FORMAT( ' ENTER FULL SCALE REFLECTANCE (REAL)  ', $)
C      READ(4,755)RRFSC
C      WRITE(4,715)
715      FORMAT( ' ENTER PRIMARY STANDARD VALUE IN %    ', $)
C      READ(4,755)RPSD
C      WRITE(4,720)
720      FORMAT( ' ENTER SECONDARY STANDARD VALUE IN %  ', $)
C      READ(4,755)RSSD
C      WRITE(4,725)
725      FORMAT( ' ENTER STAGE DRIVE SPEED-U/SEC-(REAL) ', $)
C      READ(4,755)RSDSP
C      WRITE(4,730)
730      FORMAT( ' ENTER SPOTSIZE IN MICRONS (REAL)      ', $)
C      READ(4,755)RSPSZ
C      WRITE(4,50)
50      FORMAT( ' ENTER NUMBER OF READINGS              ', $)

```

```

      READ(4,60)IR
      WRITE(4,735)
735  FORMAT( ' ENTER ANY COMMENTS-18CHAR MAX-           ', $)
      READ(4,736)ARR1(3),ARR1(4),ARR1(5)
736  FORMAT(3A6)
750  FORMAT(A5)
755  FORMAT(F10.3)
C
C    MODULE-3
C
      DO 721 ITE4=1,2
721  WRITE(4,915)
      WRITE(4,901)
901  FORMAT( ' *** THIS PORTION STANDARDIZES THE COMPUTER***')
      RFS=RPSD
902  FORMAT(F8.4)
900  CALL SETADC(32,0)
      DO 915 IDO=1,3
      WRITE(4,915)
915  FORMAT(1X)
      WRITE(4,916)
916  FORMAT( ' REFLECTANCE VALUE FULL SCALE (PRIMARY STD)', )
      DO 905 I=1,20
      CALL FASTAD(12,ISS)
      CALL FASTAD(12,IS2)
      CALL FASTAD(12,IS3)
      CALL FASTAD(12,IS4)
      CALL FASTAD(12,IS5)
      CALL FASTAD(12,IS6)
      RSS=FLOAT(ISS)+FLOAT(IS2)+FLOAT(IS3)
      RS1=FLOAT(IS4)+FLOAT(IS5)+FLOAT(IS6)
      RSS=(RSS+RS1)/6.
      RD=1000./RSS
      RE=RD*RFS
      WRITE(4,910)RE
910  FORMAT(1X,F10.3)
      DO 908 IDL=1,100
908  CONTINUE
905  CONTINUE
      WRITE(4,920)
920  FORMAT( ' ARE YOU SATISFIED ?           ', $)
      READ(4,940)J1
940  FORMAT(A1)
      IF(J1.EQ.'N')GO TO 900
C
C    MODULE-4
C
      II=0
60  FORMAT(I6)
      KNT=0
      WRITE(4,51)
51  FORMAT( ' ENTER DELAY FACTOR           ', $)

```

```

      READ(4,60)IDL1
      WRITE(4,52)
52    FORMAT( ' ENTER NUMBER OF READINGS PER TRAVERSE ', $)
      READ(4,60)IPT
      WRITE(4,53)
53    FORMAT( ' ENTER LINE TO LINE DISTANCE IN STEPS ', $)
      READ(4,60)ISPZ
      ICTR=IR/IPT
      IRES=IR-(ICTR*IPT)
      KNTTR=1
C
C    MODULE-5
C
      CALL SETADC(32,0)
      IDIR=2
      DO 65 IDA=1,ICTR
      CALL MOVE2(IDIR,1,IPT,IDL1)
      WRITE(4,74)KNTTR
74    FORMAT( ' END OF DATA ACQUISITION TRAVERSE NUMBER ',14)
C
C    MODULE-6
C
      WRITE(4,73)
73    FORMAT( ' Y MOVEMENT IN PROGRESS ',)
      CALL MOVE3(0,1,ISPZ)
      KNTTR=KNTTR+1
      WRITE(4,72)KNTTR
72    FORMAT( ' BEGIN TRAVERSE ',14)
      IF(IDIR.EQ.2)GO TO 127
      IDIR=2
      GO TO 65
127   IDIR=5
65    CONTINUE
      IPT=IRES
      CALL MOVE2(IDIR,1,IPT,IDL1)
C
C    MODULE-7
C
      WRITE(4,128)
128   FORMAT(' DO YOU WANT TO FILTER THE DATA ?', $)
      READ(4,129)FIL
129   FORMAT(A1)
      IF(FIL.EQ.'N')GO TO 71
      DO 260 M=1,2
      DO 200 CH=1,1023
      IDIF=IABS(IARR(CH)-IARR(CH+1))
      IF (IDIF.LT.8)GO TO 200
      IAVE=(IARR(CH)+IARR(CH+1))/2
      IARR(CH)=IAVE
      IARR(CH+1)=IAVE
200   CONTINUE
260   CONTINUE

```

```

      IOLD=IARR(1)
      IARR(1)=(IARR(1)+IARR(2))/2
      DO 70 I=2,1023
      ITEMP=IARR(I)
      IARR(I)=(IOLD/2+ITEMP+IARR(I+1)/2)/2
      IOLD=ITEMP
70    CONTINUE
71    CONTINUE
C
C      MODULE-8
C
      DO 502 IER=1,20
502    RARR(IER)=0
      CALL DATE(IMON,IDA,IYR)
      RARR(2)=RSDSP
      RARR(3)=FLOAT(IMON)
      RARR(4)=FLOAT(IDA)
      RARR(5)=FLOAT(IYR)
      RARR(6)=RRFSC
      RARR(7)=RPSD
      RARR(8)=RSSD
      RARR(9)=FLOAT(IR)
      RARR(10)=RSPSZ
      RTIM=(FLOAT(IR)/(RSDSP/RSPSZ))/60
      RARR(11)=RTIM
501    WRITE(1,500) IARR
500    FORMAT(10I8)
      WRITE(1,527) RARR
527    FORMAT(5F10.3)
      WRITE(1,528) RARR
528    FORMAT(5A6)
      DO 526 ITV=1,4
526    WRITE(4,915)
C
C      MODULE-9
C
      WRITE(4,529)
529    FORMAT(T19,'***** ANALYSIS FINISHED *****')
      STOP
      END

```

```

SUBROUTINE MOVE2(IDIR,IUNIT,IPT,IDL1)
C THIS SUBROUTINE TELLS MICROPROCESSOR IUNIT TO
C MOVE IPT INCREMENTS IN DIRECTION IDIR WITH IDL1
C DELAY TIME BETWEEN STEPS
C THIS SUBROUTINE ALSO READS THE A/D CONVERTER
C AND INCREMENTS THE DATA ARRAY ELEMENT
C RECEIVE ACKNOWLEDGE
COMMON IARR(1024)
DO 500 IMST=1,IPT
CALL PUTB(6)
CALL PUTB(IUNIT)
CALL GETB(IDUMMY)
C SEND COMMAND
CALL PUTB(4)
CALL PUTB(IUNIT)
CALL PUTB(1)
CALL PUTB(IDIR)
CALL FASTAD(5,I)
DO 501 IDL=1,IDL1
501 CONTINUE
CALL FASTAD(5,I2)
I=(I+I2)/2
IARR(I)=IARR(I)+1
500 CONTINUE
RETURN
STOP
END

```

```

SUBROUTINE MOVE3(IDIR,IUNIT,ISPZ)
C THIS SUBROUTINE TELLS MICROPROCESSOR IUNIT TO
C MOVE ISPZ INCREMENTS IN DIRECTION IDIR
C RECEIVE ACKNOWLEDGE
DO 500 IMST=1,ISPZ
CALL PUTB(5)
CALL PUTB(IUNIT)
CALL GETB(IDUMMY)
C SEND COMMAND
CALL PUTB(4)
CALL PUTB(IUNIT)
CALL PUTB(1)
CALL PUTB(IDIR)
500 CONTINUE
RETURN
STOP
END

```



```

/      SUBROUTINE FASTAD(INR,ISS)
/      AUTHORS: FRED MARK SEAGAARD, THOMAS TRIMARCHI
/      /THE FOLLOWING IS A RALF FORTRAN IV SUBROUTINE TO READ THE ANALOG TO
/      /DIGITAL CONVERTER. TAKES INR**2 READINGS AND RETURNS AVERAGE IN ISS.
/      /THIS SUBROUTINE IS TO BE USED WITH SUBROUTINE SETADC.
/      1) INR          IS THE NUMBER OF READINGS TO BE AVERAGED.
/      THE NUMBER OF READINGS TAKEN IS 2**INR. THE FOLLOWING TABLE
/      MAY HELP YOU COMPUTE THE ACTUAL NUMBER OF AVERAGES.
/      VALUE PASSED AS ARGUMENT          NUMBER OF AVERAGES
/      0                                1
/      2                                4
/      4                                16
/      2) ISS          AN INTEGER VALUE REPRESENTING (2**INR) AVERAGED
/      READINGS TAKEN FROM CHANNEL SET BY SETADC.

      SECT8 FASTAD
      JA #ST
#XR,   ORG  .+10
      TEXT  +FASTAD+
#RET,  SETX #XR
      SETB  #BASE
      JA    .+3
#BASE, ORG  .+6
INR,   ORG  .+3
ISS,   ORG  .+3
      ORG   #BASE+30
      FNOP
      JA    #RET
      FNOP
#GOBAK, 0;0
#ARGS,  ORG  .+3
#RTN,   BASE #BASE
      JA    #GOBAK
#ST,    STARTD
      0210
      FSTA  #GOBAK,0
      0200
      SETX  #XR
      SETB  #BASE
      LDX   0,1
      FSTA  #BASE
      FSTA  #ARGS
      FLDA% #BASE,1+
      FSTA  INR
      FLDA% #BASE,1+
      FSTA  ISS
      STARTF
      FLDA% INR      /GET THE VALUE OF INR AND
      ATX      6      /PLACE IT IN INDEX REGISTER 6.
      TRAP4    GET2    /JMS TO PAL8 AND SET UP POINTER TO ARGUMENT INR.
      TRAP4    READIT  /JMS TO PAL8 ADC ROUTINE.
      TRAP4    GET1    /PICK UP THE FINAL VALUE FROM INDEX REG. 7

```

```

XTA      7      /GET READING FROM INDEX REGISTER 7.
FSTA%    ISS    /AND RETURN IT IN ISS.
JA        #RTN
ORG 162
GET1,    0000    /PAL8 ROUTINE TO RETURN ISS
CLA      /CLEAR AC.
TAD      FRED1  /PLACE AVERAGES INTO AC.
DCA      #XR+7  /NOW PUT IT INTO INDEX REGISTER 7.
CDF CIF 0    /GET READY TO GO TO RALF.
JMS% GET1    /BYE!!
GET2,    0000    /SET UP POINTER TO NUMBER OF AVERAGES ARG.
CLA      /CLEAR AC.
TAD      #XR+5  /PICK IT UP FROM INDEX REGISTER 6 (INR)
DCA      FRED1  /SAVE IT WHERE PAL CAN GET TO IT.
6532     /INITIATE CONVERSION FOR DOUBLE BUFFERING.
CDF CIF 0    /GET READY TO LEAVE.
JMS% GET2    /BYE!!!
FRED1,    0000    /THIS LOCATION CONTAINS INR.
ORG 200     /PLACE PAL8 CODE AT BEGINNING OF CORE PAGE.
READIT,  0000    /PAL8 ADC SUBROUTINE ENTRY POINT.
IOF      /TURN OFF ALL INTERRUPTS !!!!!!!!!!!!!!!
ISZ      INIT   /ON FIRST PASS THROUGH SUBROUTINE GET ADD OF INR
JMP      SKIP   /SKIP OVER THE FOLLOWING CODE.
FRED2,    JMS     FRED2 /GET ABSOLUTE ADDRESS +1 OF PRESENT LOCATION
CLA      /CLEAR AC GET READY FOR ARITH.
TAD      FRED2  / HERE IS WHERE WE SET UP A POINTER TO INR
TAD      OFFSET /THIS VALUE IS CRITICAL !!!!!!!!!!!!!!!
DCA      FRED2  / SAVE LOCATION OF INR IN FRED2
SKIP,     DCA     INIT /SET INIT TO 0 SO WE ONLY DO THIS ONCE.
TAD%     FRED2  /LOAD NUMBER OF AVERAGES TO BE TAKEN INTO AC.
CMA      /AND COMPLEMENT
DCA      INR1   /SAVE IT IN INR1 FOR FUTURE USE.
DCA      SUM1   /ZERO LOW ORDER WORD.
DCA      SUM2   /ZERO HIGH ORDER WORD.
TAD      INR1   /GET NUMBER OF READINGS AGAIN
DCA      NR2    /AND SAVE IT IN NR2 FOR COUNTER
/         THE FOLLOWING LINES OF CODE ARE A BINARY MULTIPLICATION
/         NR2=2**INR
C.L.L.    /MAKE SURE LINK IS CLEAR (0)
IAC
LOOP0,    ISZ      NR2
SKP
JMP
RAL
JMP      LOOP0
A12,     CIA
DCA      NR2
/         BEGIN TAKING READINGS OFF OF THE A/D.
READ,    6534     /WAIT FOR CONVERSION TO COMPLETE.
JMP      READ    /DONE YET?
6533     /YES! READ A/D BUFFER INTO AC
6535     /SKIP NEXT INSTRUCTION ON TIMING ERROR.

```

```

SKP          / NO ERROR.
JMP          ERROR /GO TO ERROR ROUTINE
6532         /CLEAR ALL FLAGS ON AD AND INITIATE CONVERSION.
TAD          MASK  /THIS IS DONE BECAUSE OF THE INTELLIGENT
/ WAY THIS MACHINE IS DESIGNED. THE ADC RETURNS INTEGER NUMBERS
/ -512 TO +511 AND SINCE THE ADDITION OF TWO NEGATIVE NUMBERS
/ TURNS THE LINK ON THIS CAN LEAD TO BIG PROBLEMS WHEN YOU ARE
/ AVERAGING MIXED SIGNED NUMBERS.
CLL
TAD          SUM2  /AND ADD SUM2.
DCA          SUM2  /SAVE SUM2.
SZL          /OVERFLOW LOW ORDER WORD?
ISZ          SUM1  /YES! INCREMENT HIGH ORDER WORD.
ISZ          NR2   /INCREMENT COUNTER.
JMP          READ  /CONTINUE READING
/ HERE IS WHERE WE DO A RATHER CLEVER DOUBLE PRECISION DIVISION
TAD          INR1  /LOAD COMPLEMENT OF THE NUMBER OF READINGS
DCA          CNT1  /INITIALIZE LOW ORDER WORD COUNTER.
JMP          LOOP1 /SKIP OVER ERROR ROUTINE.
ERROR, 7300 /CLA;CLL
6532         /CLEAR ALL ERROR FLAGS
JMP          READ  /GO BACK AND READ CHANNEL.
LOOP1, 7300 /CLA CLL
ISZ          CNT1  /CONTINUE DIVISION BY 2 ?
SKP          /YES!
JMP          NO1
TAD          SUM1
RAR
DCA          SUM1
TAD          SUM2
RAR
DCA          SUM2
JMP          LOOP1
NO1, TAD          SUM2  /LOAD FINAL DIVIDEND INTO AC.
DCA%         FRED2  /LOAD FINAL RESULT IN FRED1 INDIRECTLY.
CDF CIF 0     /RESET IF & DF FIELDS.
ION          /TURN INTERRUPT SYSTEM BACK ON!!!!!!
JMP%         READIT /RETURN TO RALF.
DECIMAL
MASK, 513     /THIS VALUE IS CHOSEN SO THAT A VALUE OF 0 IS NEVER
OCTAL
SUM1,        0
SUM2,        0
CNT1,        0
INR1,        0
INIT, 7777
OFFSET, 7772
NR2,         0

```

```

/ SUBROUTINE SETADC(IPOL, ICHAN) AUTHOR FRED MARK SEAGAARD
/ THE FOLLOWING SUBROUTINE IS A RALF FORTRAN IV SUBROUTINE TO SET
/ THE POLARITY AND CHANNEL TO THE ANALOG TO DIGITAL CONVERTER.
/ THIS PROGRAM IS TO BE USED WITH SUBROUTINE FASTAD.
/ 1) IPOL AN INTEGER VALUE OF 32 IS PASSED IF YOU WANT TO READ
/     VOLTAGES FROM 0 TO +5 VOLTS.
/     A ZERO IS PASSED IF YOU WANT TO READ FROM -2.5 TO
/     +2.5 VOLTS.
/ 2) ICHAN AN INTEGER VALUE OF 0 TO 15 IS PASSED HERE.
/ THIS IS THE CHANNEL YOU WANT TO READ.
SECT8 SETADC
JA #ST
#XR, ORG .+10 /RESERVE 10 OCTAL WORDS FOR INDEX REGISTERS.
TEXT +SETADC+
#RET, SETX #XR
SETB #BASE
JA .+3
#BASE, ORG .+6
IPOL, ORG .+3
ICHAN, ORG .+3
ORG #BASE+30
FNOP
JA #RET
FNOP
#GOBAK, 0;0
#ARGS, ORG .+3
#RTN, BASE #BASE
JA #GOBAK
#ST, STARTD
0210
FSTA #GOBAK,0
0200
SETX #XR
SETB #BASE
LDX 0,1
FSTA #BASE
FSTA #ARGS
FLDA% #BASE,1+
FSTA IPOL
FLDA% #BASE,1+
FSTA ICHAN
STARTF
FLDA% IPOL /GET POLATITY.
ATX 6 /STORE IT IN INDEX REGISTER 6.
FLDA% ICHAN /GET CHANNEL
ATX 7 /FIX AND STORE IN INDEX REGISTER 7.
TRAP4 SETUP /JMS TO PAL8 SUBROUTINE.
JA #RTN
SETUP, 0000 /PLACE RETURN ADDRESS HERE.
6530 /CLEAR ALL REGISTERS IN A/D.

```

```
CLA
TAD      #XR+6  /LOAD POLARITY REGISTER INTO
6536     /ENABLE REGISTER.
TAD      #XR+7  /LOAD CHANNEL REGISTER INTO
6531     /MULTIPLEX REGISTER.
CDF CIF 0      /RESET IF & DF FIELDS.
JMP%      SETUP /RETURN TO RALF.
```

APPENDIX II

Computer Program PLOTV1
(Fortran IV)

```

C      PROGRAM PLOT VERSION 1 --PLOTV1 1/15/80
C      THIS PROGRAM PLOTS A RAPID SCAN REFLECTOGRAM
C      WITH A SCALE OF 0 TO 2% AND A FREQUENCY OF 600
C
C      AUTHOR DAVID S. HOOVER MARCH 1978
C
C      *****
C
C      CALL PLOTS(.005,0)
C      WRITE(4,400)
400    FORMAT( ' ENTER SAMPLE ID. NUMBER ', $)
C      READ(4,410)NMBR
410    FORMAT(I5)
C      CALL XYPLOT(0.,0.,-3)
C      CALL XYPLOT(2.2,-2.5,-3)
C      CALL XYPLOT(-.1,0.,2)
C      CALL XYPLOT(0.,0.,3)
C      CALL FACTOR(.50)
C      Y=0
C      YT=0.
C      DO 10 IDO=1,10
C      DO 20 IDO2=1,9
C      YT=YT+(-.1)
C      CALL XYPLOT(0.,YT,2)
C      CALL XYPLOT(-.05,YT,2)
C      CALL XYPLOT(0.,YT,2)
20    CONTINUE
C      Y=Y+(-1.)
C      CALL XYPLOT(0.,Y,2)
C      CALL XYPLOT(-.1,Y,2)
C      CALL XYPLOT(0.,Y,2)
C      YT=YT+(-.1)
10    CONTINUE
C      YN=-8.8
C      Z1=1.80
C      CALL NUMBER(-.4,-9.5,.28,2.0,270.,1)
C      CALL NUMBER(-.4,-4.7,.28,1.00,270.,2)
C      CALL NUMBER(-.4,-.3,.28,0.00,270.,2)
C      CALL SYMBOL(-.9,-.7,.35,'AVG. RANDOM REFLECTANCE %',270.,25)
C      CALL XYPLOT(0.,0.,3)
C      CALL XYPLOT(0.,.1,2)
C      CALL XYPLOT(0.,0.,3)
C      XX=0
C      DO 21 IDOY=1,6
C      XX=XX+1.
C      CALL XYPLOT(XX,0.,2)
C      CALL XYPLOT(XX,.1,2)
C      CALL XYPLOT(XX,0.,2)
21    CONTINUE
C      CALL NUMBER(5.9,.85,.28,600,270,-1)
C      CALL NUMBER(2.9,.85,.28,300,270,-1)

```

```

      CALL SYMBOL(1.5,1.0,.35,'FREQUENCY',0.,9)
      CALL XYPLOT(0.,0.,3)
      DIMENSION IARR(1024),MARR(1002)
      DO 31 IAR4=1,1024
31      IARR(IAR4)=0
      DO 35 IAR2=1,1002
35      MARR(IAR2)=0
      READ(1,600) IARR
600      FORMAT(10I8)
      DO 30 IAR=1001,1024
30      IARR(IAR)=0
      DO 34 IAR5=1,1000
      IF(IARR(IAR5).GE.600) IARR(IAR5)=600
34      CONTINUE
      IARR(1)=600
      ISC=0
      DO 40 IAR3=1,1000
      ISC=ISC+1
      MARR(IAR3)=ISC
40      CONTINUE
      CALL PSCALE(IARR,6.,1000,1)
      CALL PSCALE(MARR,10.,1000,1)
      DO 50 IARN=1,1000
      MARR(IARN)=0-MARR(IARN)
50      CONTINUE
      CALL LINE(IARR,MARR,1000,1,0,0)
      CALL SYMBOL(5.,-6.5,.35,'PSOC-',270,5)
      CALL NUMBER(5.,-8.2,.35,NMBR,270,-1)
      CALL XYPLOT(7.3,0.,3)
      CALL XYPLOT(12.5,5.0,3)
      CALL PLEXIT
      END

```


APPENDIX III
Computer Program KOL03

```

C PROGRAM VERSION 3 -- KOLO3 1/10/80
C THIS PROGRAM READS TWO REFLECTOGRAMS FROM DISK AND
C CALCULATES THE KOLMOGOROV-SMIRNOV TWO-TAILED STATISTIC
C FOR LARGE SAMPLES. THE RESULT IS TESTED FOR SIGNIFICANCE
C AT VARIOUS ALPHA LEVELS.
C
C BASIC ALGORITHMS WRITTEN BY--K. KUEHN--MODIFIED BY
C DAVID S. HOOVER FOR APPLICATION TO REFLECTOGRAMS.
C MARCH 1979
C
C *****
C
C DIMENSION IARR(1024),RARR2(20),MARR(1024),RMARR2(20)
C DO 5 I=1,1024
C   IARR(I)=0
C   MARR(I)=0
C   DO 7 I=1,20
C     RARR2(I)=0.0
C     RMARR2(I)=0.0
C   READ(1,11)IARR
C   READ(2,11)MARR
C   11 FORMAT(10I8)
C   IDIF=0
C   DO 9 I=1,1000
C     IDIF=IDIF+IABS((IARR(I)-MARR(I)))
C     IC=0
C     ISUM=0
C     IS=1
C     IST=50
C     DO 100 IA=1,20
C     DO 51 IB=IS,IST
C       ISUM=ISUM+IARR(IB)
C       RAVG=FLOAT(ISUM)/50.0
C       IC=IC+1
C       RARR2(IC)=RAVG
C       IS=IS+50
C       IST=IST+50
C       ISUM=0
C   100 CONTINUE
C     ISUM=0
C     IS2=1
C     IST2=50
C     IC2=0
C     DO 200 IA2=1,20
C     DO 250 IB2=IS2,IST2
C       250 ISUM=ISUM+MARR(IB2)
C       RAVG=FLOAT(ISUM)/50.
C       IC2=IC2+1
C       RMARR2(IC2)=RAVG
C       IS2=IS2+50
C     275

```

```

IST2=IST2+50
ISUM=0
200 CONTINUE
RSUM4=0.0
DO 310 I=1,20
310 RSUM4=RSUM4+RARR2(I)
RSUM5=0.0
DO 320 I=1,20
320 RSUM5=RSUM5+RMARR2(I)
CUM1=0.0
CUM2=0.0
TEMP=0.0
ICAT=0
DO 10 I=1,20
IF(RARR2(I).EQ.0.AND. RMARR2(I).EQ.0.)GO TO 10
PCT1=RARR2(I)/RSUM4
CUM1=CUM1+PCT1
PCT2=RMARR2(I)/RSUM5
CUM2=CUM2+PCT2
ADIF=CUM1-CUM2
DIFF=ABS(ADIF)
IF(DIFF.LE.TEMP)GO TO 10
TEMP=DIFF
ICAT=I
10 CONTINUE
A05=1.36*(SQRT((RSUM4+RSUM5)/(RSUM4*RSUM5)))
A01=1.63*SQRT((RSUM4+RSUM5)/(RSUM4*RSUM5))
A001=1.95*SQRT((RSUM4+RSUM5)/(RSUM4*RSUM5))
DO 15 I=1,5
WRITE(4,12)
12 FORMAT(1X)
15 CONTINUE
WRITE(4,110)A05
110 FORMAT( ' THE A05 VALUE IS ',F10.5)
WRITE(4,17)TEMP
17 FORMAT( ' THE MAXIMUM D VALUE OBTAINED WAS ',F8.5)
WRITE(4,20)
20 FORMAT(1X)
WRITE(4,22)ICAT
22 FORMAT( ' THE CATEGORY WHERE THE MAXIMUM D VALUE OCCURRED
1WAS',I5)
WRITE(4,26)
26 FORMAT(1X)
IF(TEMP.GT.A05)GO TO 30
WRITE(4,35)
35 FORMAT( ' THIS D VALUE IS NOT SIGNIFICANT AT THE .05 LEVEL.')
```

```
STOP
```

```
30    IF(TEMP.GE.A01)GO TO 40
      WRITE(4,45)
45    FORMAT( ' THIS D VALUE IS SIGNIFICANT AT THE .05 LEVEL.')
      STOP
40    IF(TEMP.GE.A001)GO TO 50
      WRITE(4,55)
55    FORMAT( ' THIS D VALUE IS SIGNIFICANT AT THE .01 LEVEL.')
      STOP
50    WRITE(4,60)
60    FORMAT( ' THIS D VALUE IS SIGNIFICANT AT THE .001 LEVEL.')
      STOP
```

APPENDIX IV
Computer Program PLOTV2
(Fortran IV)

C
C
C
C
C
C
C
C
C

PROGRAM PLOT VERSION 2 --PLOTV2 1/18/80
 THIS PROGRAM PLOTS CONVENTIONAL REFLECTANCE PROFILES
 IT PARTIONS THE DATA INTO 1/2 VSTEPS
 IT ASKS FOR MAX Y SCALE INPUT AND MAX RO

AUTHOR DAVID S. HOOVER JULY 1978

```

CALL PLOTS(.005,0)
NMBR=3
IMXY=12
IMXRO=10
CALL XYPLOT(0.,0.,-3)
CALL XYPLOT(2.2,-2.5,-3)
CALL XYPLOT(-.1,0.,2)
CALL XYPLOT(0.,0.,3)
CALL FACTOR(.50)
Y=0
YT=0.
DO 10 IDO=1,10
DO 20 IDO2=1,9
YT=YT+(-.1)
CALL XYPLOT(0.,YT,2)
CALL XYPLOT(-.05,YT,2)
CALL XYPLOT(0.,YT,2)
20 CONTINUE
Y=Y+(-1.)
CALL XYPLOT(0.,Y,2)
CALL XYPLOT(-.1,Y,2)
CALL XYPLOT(0.,Y,2)
YT=YT+(-.1)
10 CONTINUE
RHM=FLOAT(IMXRO)/2.0
CALL NUMBER(-.4,-9.3,.28,IMXRO,270.,1)
CALL NUMBER(-.4,-4.7,.28,RHM,270.,2)
CALL NUMBER(-.4,0.3,.28,0,270.,2)
CALL SYMBOL(-.9,-.7,.35,'AVG. RANDOM REFLECTANCE %',270.,25)
CALL XYPLOT(0.,0.,3)
CALL XYPLOT(0.,.1,2)
CALL XYPLOT(0.,0.,3)
XX=0
DO 21 IDOY=1,6
XX=XX+1.
CALL XYPLOT(XX,0.,2)
CALL XYPLOT(XX,.1,2)
CALL XYPLOT(XX,0.,2)
21 CONTINUE
CALL NUMBER(5.9,.85,.28,IMXY,270.,-1)
CALL SYMBOL(1.5,1.0,.35,'FREQUENCY',0.,9)
CALL XYPLOT(0.,0.,3)
DIMENSION IARR(1024),MARR(1002),ARR(5)

```

```

DO 31 IAR4=1,1024
31  IARR(IAR4)=0
DO 35 IAR2=1,1002
35  MARR(IAR2)=0
    READ(1,600) IARR
    READ(1,601) ARR
601  FORMAT(5A6)
600  FORMAT(10I8)
    ITOT=0
    IS=1
    IST=5
    DO 310 IGA=1,200
    DO 320 IGB=IS,IST
    ITOT=ITOT+IARR(IGB)
320  CONTINUE
    DO 315 IGC=IS,IST
    IARR(IGC)=ITOT
315  CONTINUE
    IS=IST+1
    IST=IST+5
    ITOT=0
310  CONTINUE
    DO 30 IAR=1001,1024
30  IARR(IAR)=0
    DO 34 IAR5=1,1000
    IF(IARR(IAR5).GE.IMXY) IARR(IAR5)=IMXY
34  CONTINUE
    IARR(1)=IMXY
    ISC=0
    DO 40 IAR3=1,1000
    ISC=ISC+1
    MARR(IAR3)=ISC
40  CONTINUE
    CALL PSCALE(IARR,6.,1000,1)
    CALL PSCALE(MARR,10.,1000,1)
    DO 50 IARN=1,1000
    MARR(IARN)=0-MARR(IARN)
50  CONTINUE
    CALL LINE(IARR,MARR,1000,1,0,0)
    CALL SYMBOL(4.06,-6.5,.28,ARR,270,13)
    CALL SYMBOL(4.55,-5.66,.28,'1/2 V-TYPE PLOT',270,15)
    CALL SYMBOL(5.,-6.5,.35,'PSOC-',270,5)
    CALL NUMBER(5.,-8.2,.35,NMBR,270,-1)
    CALL XYPLOT(7.3,0.,3)
    CALL XYPLOT(12.6,5.2,3)
    CALL PLEXIT
    END

```

APPENDIX V
Computer Program CONAL3
(Fortran IV)


```

C      PROGRAM CONAL VERSION 3 -- CONAL3 1/15/80
C      THIS PROGRAM ENABLES CONVENTIONAL REFLECTANCE ANALYSIS
C      TO BE CARRIED OUT USING THE ARM INSTALLATION
C      IT IS CAPABLE OF MEASURING AVERAGE RANDOM REFLECTANCE OR
C      MAXIMUM REFLECTANCE FOR VITRINITE AND PSEUDOVITRINITE
C      THIS VERSION HAS SPECIAL DATA PROCESSING ALGORITHMS
C      FOR THE DETERMINATION OF MAXIMUM REFLECTANCE
C
C      AUTHOR DAVID S. HOOVER JUNE 1978
C
C      *****
C
C      DIMENSION IARR(1024),MARR(1024)
C      DO 750 IZ=1,1024
750    IARR(IZ)=0
C      DO 755 IZZ=1,1024
755    MARR(IZZ)=0
C      KNTV=0
C      J1='N'
C      JJ1='N'
C      ITPVIT=0
C      KNTP=0
C      CALL BLANK
C      WRITE(4,600)
600    FORMAT(T15,' ** COAL RESEARCH MANUAL REFLECTANCE PROGRAM **')
C      DO 10 I=1,3
10     WRITE(4,610)
610    FORMAT(1X)
C      WRITE(4,615)
615    FORMAT(' DO YOU WANT TO MEASURE MAX. REFLECTANCE      ',5)
C      READ(4,620)J1
620    FORMAT(A1)
C      WRITE(4,610)
C      IF(J1.EQ.'Y')GO TO 15
C      WRITE(4,625)
625    FORMAT(' ** I GUESS YOU WANT TO MEASURE MARR THEN **')
C      WRITE(4,610)
15     WRITE(4,630)
630    FORMAT(' DO YOU WISH TO MEASURE PVIT.      ',5)
C      READ(4,620)J2
C      WRITE(4,610)
C      WRITE(4,635)
635    FORMAT(' ENTER NUMBER OF READINGS VITRINITE---',5)
C      READ(4,640)ITPVIT
640    FORMAT(I4)
C      IF(J2.EQ.'N')GO TO 20
C      WRITE(4,610)
C      WRITE(4,645)
645    FORMAT(' ENTER NUMBER OF READINGS PVIT-----',5)
C      READ(4,640)ITPVIT
20     CALL BLANK

```

```

        WRITE(4,650)
650   FORMAT(T15,' ***** TIME TO STANDARDIZE MICROSCOPE *****')
C     STANDARDIZATION ROUTINE
      IDEC=(ITPVIT+ITVIT)/2
      WRITE(4,610)
      WRITE(4,610)
      WRITE(4,610)
      WRITE(4,903)
903   FORMAT( ' ENTER FULL SCALE RO ---REAL---      ', $)
      READ(4,902)RES
      WRITE(4,901)
901   FORMAT( ' ENTER GLASS STD. RO-----      ', $)
      READ(4,902)RFS
902   FORMAT(F8.4)
900   CALL SETADC(32,0)
      DO 915 ISTD=1,3
      WRITE(4,915)
915   FORMAT(1X)
      WRITE(4,916)
916   FORMAT( ' REFLECTANCE FULL SCALE  ')/
      IF(J1.EQ.'Y')GO TO 906
      DO 905 ISTD1=1,20
      CALL FASTAD(12,ISS)
      CALL FASTAD(12,IS2)
      CALL FASTAD(12,IS3)
      CALL FASTAD(12,IS4)
      CALL FASTAD(12,IS5)
      CALL FASTAD(12,IS6)
      RSS=FLOAT(ISS)+FLOAT(IS2)+FLOAT(IS3)+FLOAT(IS4)
      RS1=FLOAT(IS5)+FLOAT(IS6)
      RSS=(RSS+RS1)/6.
81    RD=1000./RSS
      RE=RD*RFS
      WRITE(4,910)RE
910   FORMAT(1X,F10.3)
      DO 908 IDL=1,100
908   CONTINUE
      IF(J1.EQ.'Y')GO TO 907
905   CONTINUE
904   CONTINUE
      WRITE(4,920)
920   FORMAT( ' ARE YOU SATISFIED?  ', $)
      READ(4,940)JJ1
940   FORMAT(A1)
      IF(JJ1.EQ.'N')GO TO 900
      CALL BLANK
      WRITE(4,655)
655   FORMAT( ' YOU ARE NOW READY TO BEGIN THE ANALYSIS  ')
      DO 25 I1=1,5
25    WRITE(4,610)
      KNTV1=0
      KNTPL=0

```

```

30      CONTINUE
        WRITE(4,660)
660     FORMAT(' WHEN READY TO MEASURE A READING HIT -RETURN-',$)
        READ(4,620)JDUM
        IF(J1.EQ.'Y')GO TO 82
        CALL FASTAD(12,ISV)
        CALL FASTAD(12,ISV2)
        ISV=(ISV+ISV2)/2
        GO TO 34
906     DO 907 ISM=1,10
        IF(JJ1.EQ.'N')GO TO 909
82      IRST=200
        GO TO 33
909     IRST=100
33      IMAX=0
        IKP=0
        IKO=0
        DO 32 IR=1,IRST
41      CALL FASTAD(9,ISV)
        IF(IKO.EQ.1)GO TO 38
        IF(IKO.GE.2)GO TO 39
        IOLD1=ISV
        IKO=IKO+1
        GO TO 32
38      IOLD2=ISV
        IKO=IKO+1
        GO TO 32
39      IOLD3=ISV
        ISV=(IOLD1+IOLD2+IOLD3)/3
        IOLD1=IOLD2
        IOLD2=IOLD3
        IF(ISV.GT.IMAX)IMAX=ISV
32      CONTINUE
        ISV=IMAX-3
        RSS=FLOAT(IMAX)
        IF(JJ1.EQ.'N')GO TO 81
907     CONTINUE
        IF(JJ1.EQ.'N')GO TO 904
34      RE1=RES/1000.
        RE2=RE1*FLOAT(ISV)
        WRITE(4,670)RE2
670     FORMAT(' REFLECTANCE VALUE THIS POINT= ',F7.3)
        CALL BELL
        CALL BELL
        WRITE(4,675)
675     FORMAT(' DO YOU WANT TO RECORD THIS READING ? ', $)
        READ(4,620)J4
        IF(J4.EQ.'N')GO TO 30
        IF(J2.EQ.'N')GO TO 40
        WRITE(4,665)
665     FORMAT(' WAS THIS READING PVIT ` ', $)
        READ(4,620)J3

```

```

IF(J3.EQ.'N')GO TO 40
MARR(ISV)=MARR(ISV)+1
KNTP=KNTP+1
KNTPl=KNTPl+1
IF(KNTP.GE.ITPVIT)GO TO 35
CALL BLANK
WRITE(4,681)KNTV
681  FORMAT(' NUMBER OF READINGS ON VIT SO FAR = ',I5)
WRITE(4,680)KNTP
680  FORMAT(' NUMBER OF READINGS ON PVIT SO FAR = ',I5)
WRITE(4,610)
WRITE(4,610)
WRITE(4,610)
IF(KNTPl.EQ.IDEC)GO TO 44
GO TO 30
37  CALL BELL
GO TO 30
35  WRITE(4,685)
685  FORMAT(' YOU HAVE REACHED YOUR GOAL FOR PVIT ')
CALL BELL
IF(KNTV.LT.ITVIT)GO TO 30
IF(KNTPl.EQ.25)GO TO 20
40  IARR(ISV)=IARR(ISV)+1
KNTV=KNTV+1
KNTPl=KNTPl+1
IF(KNTV.GE.ITVIT)GO TO 45
CALL BLANK
IF(J2.EQ.'N')GO TO 689
WRITE(4,688)KNTP
688  FORMAT(' NUMBER OF READINGS ON PVIT SO FAR = ',I5)
689  WRITE(4,690)KNTV
690  FORMAT(' NUMBER OF READINGS ON VIT SO FAR = ',I5)
WRITE(4,610)
WRITE(4,610)
WRITE(4,610)
IF(KNTPl.EQ.IDEC)GO TO 44
GO TO 30
44  CALL BELL
IF(J2.EQ.'N')GO TO 810
WRITE(2,696)MARR
WRITE(1,696)IARR
DO 830 IZRP=1,1024
MARR(IZRP)=0
830  CONTINUE
GO TO 820

```

```
810  WRITE(1,696)IARR
820  DO 825 IZR=1,1024
      IARR(IZR)=0
825  CONTINUE
      GO TO 30
45   WRITE(4,695)
695  FORMAT( ' YOU HAVE REACHED YOUR GOAL FOR VIT. ')
      DO 733 IBLL=1,20
      CALL BELL
733  CONTINUE
      ISUMT=0
      DO 735 ISUM=1,1024
735  ISUMT=ISUMT+IARR(ISUM)
      WRITE(4,736)ISUMT
736  FORMAT(I5)
      IF(J2.EQ.'N')GO TO 49
      IF(KNTP.LT.ITPVIT)GO TO 30
      WRITE(5,696)MARR
49   WRITE(3,696)IARR
696  FORMAT(10I8)
      STOP
      END
```

APPENDIX VI
Computer Program CBLEND
(Fortran IV)

```

C      PROGRAM CBLEND VERSION 1 -- CBLEND 1/18/80
C      THIS PROGRAM IS DESIGNED TO READ THREE DATA ARRAYS FROM
C      DISK AND COMBINE THEM IN DESIGNATED PERCENTAGES TO GENERATE
C      A BLEND DATA ARRAY WHICH WILL CONTAIN 150,000 DATA POINTS
C
C      AUTHOR DAVID S. HOOVER DECEMBER 1979
C
C      *****
C
C      DIMENSION IAR1(1024), IAR2(1024), IAR3(1024), IAR4(1024)
C      READ(1,10) IAR1
C      READ(2,10) IAR2
C      READ(3,10) IAR3
C      WRITE(4,20)
20     FORMAT( ' ENTER PERCENT OF FIRST COAL IN BLEND', $)
C      READ(4,25) RPT1
C      WRITE(4,30)
30     FORMAT9 ' ENTER PERCENT OF SECOND COAL IN BLEND', $)
C      READ(4,25) RPT2
C      WRITE(4,40)
40     FORMAT( ' ENTER PERCENT OF THIRD COAL IN BLEND', $)
C      READ(4,25) RPT3
25     FORMAT(F10.3)
10     FORMAT(10I8)
C      RFAC1=RPT1*3.0
C      RFAC2=RPT2*3.0
C      RFAC3=RPT3*3.0
C      DO 15 I=1,1024
C      IAR1(I)=IFIX(FLOAT(IAR1(I))*RFAC1)
C      IAR2(I)=IFIX(FLOAT(IAR2(I))*RFAC2)
C      IAR3(I)=IFIX(FLOAT(IAR3(I))*RFAC3)
C      IAR4(I)=IAR1(I)+IAR2(I)+IAR3(I)
15     CONTINUE
C      WRITE(5,10) IAR4
C      STOP
C      END

```

APPENDIX VII
Data for Coals Analyzed

Sample	State	Seam	Rank	% R ₀	% Vit.	% Lip.	% Sin.	% In.	% Ash	% Sul.	% VM (dmf)	% C (dmf)
PSOC-002C	KY	UPPER ELKHORN #3	HVA	0.87	47.4	21.3	6.0	31.4	4.1	0.6	39.4	85.0
PSOC-002D	KY	UPPER ELKHORN #3	HVA	0.91	30.5	39.3	4.6	30.1	6.8	0.6	37.6	86.6
PSOC-003	KY	UPPER ELKHORN #3	HVA	0.85	41.9	21.7	5.0	36.4	4.6	0.6	37.4	85.6
PSOC-003D	KY	UPPER ELKHORN #3	HVA	0.83	27.8	32.4	8.8	39.8	3.7	0.6	39.4	86.1
PSOC-004	KY	UPPER ELKHORN #3	HVA	0.85	72.1	13.2	1.4	14.8	2.1	0.9	39.0	84.4
PSOC-006D	KY	UPPER ELKHORN #3	HVA	0.95	18.4	46.9	13.4	34.6	6.1	0.7	40.4	86.2
PSOC-012	KY	KENTUCKY C	HVA	0.92	47.2	21.1	5.2	31.7	2.6	0.6	39.4	85.6
PSOC-022	IL	ILLINOIS #5		0.49	88.4	4.4	1.4	7.2	10.1	2.6	41.9	80.8
PSOC-056	AZ	ARIZONA #2	HVC	0.57	93.9	3.3	4.0	12.8	6.2	0.4	47.3	77.4
PSOC-060	CO	WADGE	HVC	0.58	84.7	2.3	3.1	13.0	11.2	0.5	45.1	77.5
PSOC-070	WA	BIG #2	HVB	0.78	89.0	9.3	0.8	1.8	25.7	0.4	41.4	81.0
PSOC-084	PA	PENNSYLVANIA #8	SEMI-AN	2.15	70.8	0.0	20.2	29.2	25.3	1.2	8.0	92.9
PSOC-085	PA	PENNSYLVANIA #8--LEADER	AN	2.82	97.5	0.0	0.0	2.5	8.3	1.1	6.5	92.5
PSOC-102	PA	PITTSBURGH	HVA	0.84	82.5	3.9	4.3	13.6	7.1	1.4	38.6	84.6
PSOC-104	PA	PITTSBURGH	HVA	0.80	80.7	6.6	3.4	12.7	9.5	3.4	38.8	84.8
PSOC-106	IN	INDIANA #1 BLOCK	HVB	0.63	24.0	28.2	11.4	47.6	14.4	0.6	39.0	84.2
PSOC-109	PA	PITTSBURGH	HVA	0.83	84.6	4.8	1.8	10.6	5.1	0.6	37.4	85.3
PSOC-113	PA	LOWER KITTINGING	LOW VOL	1.80	86.9	0.0	3.9	13.1	10.6	3.1	18.4	90.9
PSOC-116	PA	LOWER FREEPORT	LOW VOL	1.47	85.8	0.0	3.5	14.2	12.9	4.0	21.4	88.6
PSOC-125	PA	LOWER FREEPORT	HVA	1.17	91.4	1.3	1.4	7.4	10.0	1.9	33.0	88.8
PSOC-127	PA	LOWER KITTINGING	LOW VOL	1.51	92.1	0.0	3.9	7.9	5.7	0.8	20.0	90.4
PSOC-128	PA	LOWER KITTINGING	LOW VOL	1.56	77.4	0.0	2.7	22.6	13.6	2.1	19.4	89.5
PSOC-132	WV	POCAHONTAS #3	MED VOL	1.47	71.4	0.0	6.4	28.5	3.4	0.7	22.4	91.0
PSOC-134	AL	PRATT	MED VOL	1.31	78.6	0.0	4.4	21.4	4.6	1.0	24.8	90.2
PSOC-142	OK	HARTSHORNE	MED VOL	1.45	90.5	0.0	1.0	9.5	10.2	1.1	22.2	90.4
PSOC-145	OK	HARTSHORNE	LOW VOL	1.57	81.1	0.0	3.6	18.9	5.3	0.8	19.6	90.3
PSOC-146	OK	HARTSHORNE RIDER	LOW VOL	1.49	84.7	0.0	4.5	15.3	6.4	0.8	15.0	92.2
PSOC-149	OK		HVB	0.54	71.9	11.0	5.4	16.9	16.1	6.8	41.5	83.2
PSOC-151	NM	LOWER SPLIT OF BLUE	HVC	0.40	78.6	10.6	2.3	10.8	5.1	0.4	45.4	78.4
PSOC-157	CO	COLORADO B	MED VOL	1.43	92.0	0.1	0.5	7.9	6.7	0.6	26.0	89.9
PSOC-159	CO		SEMI-AN	3.10	92.4	0.0	0.8	7.6	7.1	0.8	13.2	92.5
PSOC-163	MO	BAXTER	HVA	0.73	79.8	3.9	2.5	16.3	13.1	6.1	41.6	85.4
PSOC-173	WV	PITTSBURGH	HVA	0.78	81.4	4.9	1.8	13.7	6.7	2.2	41.3	85.8
PSOC-176	WV	PITTSBURGH	HVA	0.82	76.3	4.4	3.2	19.3	6.5	2.9	42.2	85.8
PSOC-194	OH	OHIO #5	HVB	0.60	85.9	5.4	2.3	8.7	9.2	3.2	41.9	80.4
PSOC-211	OH	OHIO #3	HVB	0.52	84.3	3.9	1.5	11.8	16.0	4.9	42.9	77.9
PSOC-233	CO	WADGE	HVC	0.61	88.8	4.7	1.6	6.5	9.1	0.5	41.8	78.2
PSOC-236	CO	COLORADO BASIN B	MED VOL	1.44	92.2	0.0	2.0	7.8	8.0	0.5	24.2	90.3
PSOC-242	WY	DIETZ	SUBBIT B	0.43	90.6	3.8	3.3	5.6	5.9	0.6	46.0	74.5
PSOC-254	PA	LOWER KITTINGING--TOP BENCH	MED VOL	0.0	85.8	0.0	2.4	14.2	10.2	1.9	27.2	0.0

Sample	State	Seam	Rank	% R ₀	% Vit.	% Lip.	% Sin.	% In.	% Ash	% Sul.	% VM (dmmf)	% C (dmmf)
PSOC-255	PA	LOWER KITTANNING--BOTTOM BENCH	MED VOL	0.0	76.0	0.2	6.4	23.8	19.0	5.3	26.8	0.0
PSOC-256	PA	LOWER FREEPORT	MED VOL	0.0	82.2	0.0	3.9	17.8	6.7	1.8	27.0	0.0
PSOC-257	PA	UPPER FREEPORT	MED VOL	0.0	88.1	3.5	2.2	8.4	12.6	1.6	29.5	0.0
PSOC-258	PA	LOWER KITTANNING	MED VOL	0.0	73.5	0.0	5.0	26.4	10.1	4.0	22.7	0.0
PSOC-266	VA	UPPER BANNER	HVB	0.72	21.4	0.3	11.9	78.3	23.6	3.0	32.6	85.3
PSOC-270	AL	AMERICAN	HVA	1.02	51.1	13.8	5.6	35.1	2.2	0.8	34.1	77.8
PSOC-274	WV	SEWELL	MED VOL	0.98	71.3	5.8	4.6	22.9	15.7	2.3	35.8	85.9
PSOC-276	OH	OHIO #3	HVA	1.14	68.6	4.8	2.9	26.6	8.7	0.8	30.5	89.1
PSOC-307	OH	OHIO #12A	HVA	0.65	80.7	3.7	4.7	15.6	11.2	3.5	43.7	83.9
PSOC-309	NM	NEW MEXICO #3	HVC	0.76	87.0	2.9	2.6	10.1	19.3	2.7	43.5	83.8
PSOC-318	PA	UPPER FREEPORT	LOW VOL	0.48	73.4	1.8	11.0	24.8	20.4	0.8	46.0	79.1
PSOC-319	PA	UPPER KITTANNING	LOW VOL	1.65	92.1	0.2	3.5	7.7	6.5	1.6	19.3	90.9
PSOC-320	PA	PITTSBURGH	MED VOL	1.55	77.5	0.1	7.6	22.4	16.2	6.0	18.6	91.5
PSOC-321	PA	PITTSBURGH	MED VOL	1.30	92.5	0.1	5.0	7.4	15.8	1.2	24.3	90.3
PSOC-324	PA	MIDDLE KITTANNING	MED VOL	1.35	90.4	0.2	5.7	9.4	15.0	2.6	23.3	90.0
PSOC-330	PA	MIDDLE KITTANNING	HVB	1.18	89.6	2.5	3.1	7.9	34.8	3.3	27.1	89.0
PSOC-336	PA	UPPER FREEPORT	MED VOL	0.76	84.2	3.4	2.8	12.4	6.1	2.8	39.5	83.7
PSOC-337	PA	LOWER KITTANNING	HVA	1.24	88.5	0.0	2.9	11.5	10.8	2.6	25.9	89.3
PSOC-340	PA	LOWER KITTANNING	HVA	0.89	75.1	16.4	2.8	8.4	8.5	1.5	37.5	85.3
PSOC-341	PA	CLARION	HVA	0.92	90.7	2.6	2.4	6.7	9.5	1.2	36.0	85.8
PSOC-345	PA	LOWER KITTANNING	HVA	0.75	68.1	18.5	4.3	13.3	11.5	4.5	42.4	85.6
PSOC-347	PA	LOWER KITTANNING	HVA	0.79	75.0	16.0	2.9	8.9	12.8	4.2	41.0	85.7
PSOC-351	PA	LOWER CLARION	HVA	0.78	80.0	15.0	0.8	4.9	7.8	3.6	42.1	85.4
PSOC-354	PA	LOWER KITTANNING	HVA	0.71	95.1	1.2	0.4	3.7	6.7	3.4	45.0	84.1
PSOC-359	KY	LOWER ELKHORN	HVA	0.81	75.3	15.4	4.0	9.4	12.9	1.4	38.0	84.6
PSOC-360	KY	LOWER ELKHORN	HVA	1.03	64.1	20.0	7.2	15.8	29.7	0.3	33.8	88.1
PSOC-363	KY	HAZARD #5A	HVA	1.05	76.9	7.1	6.4	16.0	8.0	0.6	33.7	87.2
PSOC-366	KY	HAZARD #5A	HVA	0.79	60.3	18.7	8.4	21.1	9.5	0.7	39.1	83.9
PSOC-369	KY	HAZARD #7	HVA	0.77	60.7	11.3	11.9	27.5	9.9	0.7	40.0	83.9
PSOC-375	KY	HAZARD #9	HVA	0.81	63.8	18.0	7.4	18.1	6.5	0.6	38.2	84.3
PSOC-394	TX	UNNAMED	HVC	0.78	75.8	12.0	2.5	12.2	14.2	1.1	38.9	84.4
PSOC-400	OK	FORT SCOTT	HVA	0.45	91.8	5.2	2.1	3.0	5.1	2.0	45.9	73.1
PSOC-403	OK	STIGLER	MED VOL	0.59	89.8	3.0	2.0	7.1	8.7	3.7	45.2	83.5
PSOC-405	OK	LOWER HARTSHORNE	LOW VOL	1.23	91.0	1.2	2.4	7.7	9.6	0.5	28.5	88.3
PSOC-407	OK	LOWER HARTSHORNE	LOW VOL	1.68	81.2	0.0	7.3	18.8	8.2	0.7	18.8	89.8
PSOC-452	CO	COLORADO UPPER B		1.62	85.7	0.0	5.5	14.3	7.6	1.4	18.5	90.3
PSOC-462	UT	LOWER SUNNYSIDE	HVA	1.39	95.8	0.0	2.1	4.2	0.0	0.0	0.0	0.0
PSOC-618	PA	KELLY	LOW VOL	0.63	74.4	5.0	8.8	20.7	6.0	1.0	40.7	80.9
PSOC-619	PA	BARNETT	MED VOL	1.55	89.1	0.0	7.0	10.9	32.2	1.1	21.8	91.3

Sample	State	Seam	Rank	% R ₀	% Vit.	% Lip.	% Sin.	% In.	% Ash	% Sul.	% VM (dmmf)	% C (dmmf)
PSOC-620	PA	TWIN	LOW VOL	1.54	92.7	0.0	2.7	7.3	13.3	0.6	23.4	91.5
PSOC-646	AR	PHILPOTT	LOW VOL	1.79	82.0	0.0	8.2	18.0	14.1	2.0	17.6	92.0
PSOC-688	WV	ELK LICK	LOW VOL	1.77	85.0	0.0	7.7	15.0	3.8	0.7	16.6	91.1
PSOC-702	WV	PITTSBURGH	HVA	1.63	92.7	0.0	4.1	7.3	18.6	2.7	17.3	90.5
PSOC-720	WV	UPPER KITTANNING	HVA	0.82	91.6	2.1	3.8	6.3	6.8	2.7	42.2	84.5
PSOC-735	WV	UPPER KITTANNING	MED VOL	1.14	91.3	0.6	2.6	8.1	11.3	2.3	40.8	88.5
PSOC-773	OH	OHIO #5	HVB	1.00	30.1	16.3	41.0	53.6	18.2	0.6	30.2	86.8
PSOC-876	PA	BLOSS	LOW VOL	1.71	89.2	0.0	6.9	3.9	19.44	3.6		
PSOC-379	PA	ONE FOOT	LOW VOL	1.82	88.2	0.0	7.3	4.5	22.48	0.9		
PSOC-1010	PA	LOWER KITTANNING	MED VOL	1.11	85.5	0.6	9.3	4.6	16.77	3.2		
PSOC-1018	PA	LOWER KITTANNING	HVA	0.82	85.4	3.8	5.6	5.2	12.30	5.2		
PSOC-1023	PA	LOWER FREEPORT	HVA	0.92	57.7	15.1	20.1	7.0	18.33	1.8		
PSOC-1099	PA	PITTSBURGH	HVA	0.89	87.4	1.4	6.2	5.0	11.35	1.4		
PSOC-1100	PA	PITTSBURGH	HVA						9.52	1.2		
PSOC-1101	PA	PITTSBURGH	HVA	0.90	81.8	3.2	5.8	9.2	5.43	0.7		
PSOC-1102	PA	PITTSBURGH	HVA	0.89	81.0	2.3	7.9	8.8	7.72	1.9		
PSOC-1103	PA	PITTSBURGH	HVA	0.80	69.4	9.8	11.4	8.8	4.73	1.9		
PSOC-1104	KY	UPPER ELKHORN	HVA	0.77	77.0	9.7	4.9	8.3	3.43	1.8		
CBCS-132			HVA	0.90	77.4	6.0	5.6	11.0	7.2	0.6		
CBCS-133			HVA	0.95	71.3	7.6	10.6	10.5	6.6	0.8		
CBCS-204			HVA	0.94	74.2	5.0	9.6	11.2	6.2	0.9		
CBCS-214			MED VOL	0.96	74.5	5.9	6.5	13.1	5.7	1.0		
CBCS-259			HVA	1.00	75.9	5.9	8.4	9.8	6.0	0.9		
CBCS-177			MED VOL	1.07	68.8	8.1	9.6	13.5	3.2	0.6		
CBCS-48			HVA	0.99	73.6	5.4	8.5	12.5	5.1	0.8		
CBCS-205			MED VOL	1.08	83.3	4.7	5.4	6.6	3.4	0.6		
CBCS-207			MED VOL	1.10	67.0	1.0	23.9	8.1	10.4	0.5		
CBCS-176			MED VOL	1.16	79.8	2.5	7.3	10.4	4.4	0.7		
CBCS-143			HVA	0.98	79.3	7.2	4.0	9.5	5.7	0.8		
CBCS-112			HVA	1.09	81.0	6.3	2.3	10.4	4.9	0.9		
CBCS-105			LOW VOL	1.70	78.3	0.0	8.0	13.7	5.9	0.8		
CBCS-142			LOW VOL	1.71	88.1	0.0	4.2	7.5	7.5	0.7		
CBCS-240			MED VOL	1.09	77.4	5.6	5.4	11.6	5.4	0.9		
CBCS-72			LOW VOL	1.68	72.4	0.2	15.1	12.3	5.4	0.9		
CBCS-212			MED VOL	1.09	75.6	7.0	4.8	12.6	4.6	0.8		
CBCS-155			MED VOL	1.35	78.2	0.8	10.4	10.6	3.9	0.7		
CBCS-227			MED VOL	1.20	94.4	3.1	5.4	7.1	3.2	0.8		
CBCS-178			MED VOL	1.14	60.8	0.7	25.6	12.9	8.2	0.9		
CBCS-144			MED VOL	1.15	79.1	1.8	8.5	10.6	5.9	0.7		