

## A TECHNIQUE FOR APPLICATION OF THE ROSIWAL METHOD OF ANALYSIS TO BROKEN COAL STUDIES\*

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This is a report on a technique for utilizing the Rosiwal method of analysis to determine the percentage of various banded ingredients and other constituents in broken coal preparatory to more extensive studies. Thin-section and polished-surface studies have been used for many years to determine the percentage of banded ingredients and mineral matter in column samples and blocks of coal. Previous methods of studying broken coal were found to be inadequate for the detailed study planned.

Since 1898 petrologists have used the Rosiwal method of analysis, or some variation of it, to estimate the mineralogical composition of a rock by microscopic examination. This method consists of making parallel traverses across a specimen at regular intervals and recording the amount of each constituent observed. It is based on the assumption that, as stated by Grout, "the sum of the lengths recorded for each mineral may be assumed to be proportional to the volumetric occurrence of the mineral in the rock."<sup>1</sup>

Lincoln and Reitz<sup>2</sup> reviewed the Rosiwal method of analysis and found it to be valid. Chayes<sup>3</sup> has

recently evaluated the micrometric method of analysis and states that it is always a potentially accurate procedure. He stresses that "the inaccuracy of a micrometric analysis is never the result of a failure in the basic theory relating area to volume. It is always traceable to unsatisfactory technique or sampling which is either inadequate or inefficient."

Several foreign workers have used methods of linear measurement, based on the Rosiwal concept, in polished-surface and broken-coal studies of European coals. A recent publication of work by Abramski, Mackowsky, Mantel, and Stach<sup>4</sup> is an excellent reference for studies in anthracology using polished surfaces for either broken coal or column sample study. Hacquebard<sup>5</sup> and Lahiri have used a similar method in recent investigation on crushed coal samples in Nova Scotia.

The author wishes to express his appreciation to Dr. Gilbert H. Cady<sup>6</sup> for suggesting the application of the Rosiwal method of analysis as applied to broken coal and for his advice in developing the techniques.

### PROCEDURE

Thorough tests of the binocular stereoscopic microscope, previously

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<sup>1</sup> Grout, F. F., *Textbook of petrography and petrology*: New York, McGraw-Hill, p. 17, 1932.

<sup>2</sup> Lincoln, E. C., and Reitz, H. L., *The determination of the relative volumes of the components of rocks by mensuration methods*: Econ. Geology, vol. 8, pp. 120-139, 1913.

<sup>3</sup> Chayes, F., *The theory of thin-section analysis*: Jour. Geol., vol. 62, no. 1, pp. 92-101, Jan. 1954.

<sup>4</sup> *Atlas für Angewandte Steinkohlenpetrographie, Deutschen Kohlenbergbau-Leitung in Verbindung mit dem Amt für Bodenforschung, Verlag Glückauf, G.M.B.H.—Essen, 1951.*

<sup>5</sup> Hacquebard, P. A., personal communication.

<sup>6</sup> Cady, Gilbert H., Senior Geologist and Head of the Coal Division, Illinois State Geological Survey, Emeritus.

TABLE 1.—RESULTS OF COMPARISON TESTS BETWEEN BLOCK AND BROKEN COAL ANALYSIS

Coal		Vitrain	Clarain	Inertinite	Mineral matter
Virginia Imboden.....	Block count %.....	29.08	59.68	9.41	1.83
	Broken coal count %...	29.91	58.60	9.64	1.85
	Difference.....	0.83	1.08	0.23	0.02
	% difference.....	2.77	1.84	2.39	1.08
W. Ky. #9.....	Block count %.....	70.92	25.54	2.80	0.74
	Broken coal count %...	72.02	24.28	2.90	0.80
	Difference.....	1.10	1.26	0.10	0.06
	% difference.....	1.53	5.19	3.45	7.50
Illinois #6..... (sample #2)	Block count %.....	50.43	38.84	4.82	5.91
	Broken coal count %...	51.96	38.25	3.51	6.28
	Difference.....	1.53	0.59	1.31	0.37
	% difference.....	2.94	1.54	37.32	5.89
Illinois #6..... (sample #1)	Block count %.....	41.03	55.65	1.48	1.86
	Broken coal count %...	40.76	55.45	2.18	1.61
	Difference.....	0.27	0.20	0.70	0.25
	% difference.....	0.66	0.36	32.11	15.52
Illinois #7..... (sample #1)	Block count %.....	54.07	38.76	0.70	6.47
	Broken coal count %...	54.31	39.35	0.90	5.44
	Difference.....	0.24	0.59	0.20	1.03
	% difference.....	0.44	1.50	22.22	18.93
Illinois Cutler Rider?.. (Gallatin Co.)	Block count %.....	45.67	52.32	1.53	0.48
	Broken coal count %...	44.76	50.95	1.75	2.54
	Difference.....	0.91	1.37	0.22	2.06
	% difference.....	2.03	2.68	12.57	81.10
Illinois Reynoldsburg..	Block count %.....	59.30	39.53	0.39	0.78
	Broken coal count %...	59.71	39.63	0.14	0.52
	Difference.....	0.41	0.09	0.25	0.26
	% difference.....	0.69	0.23	178.5	50.0
Illinois #7..... (sample #2)*	Block count %.....	40.98	57.53	0.62	0.87
	Broken coal count %...	40.30	58.08	1.06	0.56
	Difference.....	0.68	0.55	0.43	0.31
	% difference.....	1.69	0.95	40.56	55.35
Illinois #5.....	Block count %.....	26.16	65.83	1.31	6.70
	Broken coal count %...	26.12	64.62	3.12	6.14
	Difference.....	0.04	1.21	1.81	0.56
	% difference.....	0.15	1.87	58.01	9.12

\* Sample that was sized.

TABLE 2.—PERCENT OF DIFFERENCE BETWEEN BLOCK AND BROKEN-COAL ANALYSES GIVING MAXIMUM, MINIMUM, AND AVERAGE DIFFERENCES

Coals	% difference in vitrain	
Illinois #6..... (sample 1)	0.66	Max. diff.—2.94
Illinois #6..... (sample 2)	2.94	
*Illinois #7..... (sample 1)	0.44	
*Illinois #7..... (sample 2)	1.69	Min. diff.—0.44
Illinois Reynoldsburg.....	0.69	
Illinois "Cutler Rider" (Gallatin Co.)*	2.03	
Illinois #5.....	1.65	
Virginia Imboden*.....	2.77	Av. diff.—1.60
Western Kentucky #9.....	1.53	

\* Samples in which more than 10 traverses were made.

used in broken coal studies, showed its inadequacy in this investigation because of limited magnification and methods of illumination. Coal components as small as 1 micron (1/1000 mm) may be distinguished by using a metallurgical-type microscope with vertical illumination and oil immersion objectives. Magnification up to 640 times for very detailed studies is possible; however a magnification of 200 times was found most satisfactory for ordinary quantitative microscopic analysis.

A high degree of polish is necessary in the identification of coal constituents. It is important to choose a binding material with sufficient adhesion to prevent broken coal from being torn loose in polishing and also with approximately the same hard-

ness as coal. Carnauba wax, Lumerith No. 12144,<sup>7</sup> Transoptic powder,<sup>8</sup> and bakelite under a pressure of 500 psi and a temperature of 250° F., were found to have sufficient adhesive qualities and a hardness comparable to that of coal. All of these mounting mediums have been used in this study, but the Transoptic powder was found to be the most satisfactory. Bakelite was used in earlier tests, but in most tests the broken coal was mixed with Transoptic powder in equal amounts by volume and solidified into 1¼-inch blocks in a specimen-mounting press at 285° F. and a pressure of 3,000 psi.

In this investigation a block of

<sup>7</sup> Celanese Celluloid Corp.

<sup>8</sup> A. I. Buehler Ltd.

coal, or a block containing broken coal, was first ground to a flat surface on a laboratory grinding-lap using carborundum powders of 220- and 400-grit. It was then ground by hand on glass plates, using 400-, 800-, 1200-, and 3200-mesh emery powder successively, which resulted in a flat, smooth, dull surface. The final highly polished surface was obtained in three additional steps by first using Goddard's silver polish on silk velvet, then distilled water (running) on a high quality billiard cloth, and finally, distilled water (running) on Microcloth.<sup>9</sup> A very light touch is necessary in the final polishing stages. Photomicrographs of samples prepared by the above method are shown in figure 1.

In order to test techniques of preparation, the results obtained from a Rosiwal method of analysis of polished blocks of coal and of the same coal after it had been broken and mounted were compared. Nine blocks were picked at random from seven different coal beds, but these blocks do not necessarily represent an average sample of the bed. These blocks, 2 cm. square and 1 cm. thick, were cut and polished, and the percentage of the various banded ingredients across the coal block perpendicular to banding was determined.

Seven of the blocks were broken individually with a mortar and pestle to pass a 28-mesh Tyler screen, mounted in a binding material, polished, and restudied under the microscope. Results of all the studies are given in table 1. Broken coal from the block of Reynoldsburg coal was divided equally into two parts

and these were mounted separately for comparative study. The remaining sample, sample 2 of the Illinois No. 7 coal, was broken and screened to five sizes—plus 35 mesh, 35 x 48M, 48 x 65M, 65 x 100M, and minus 100 mesh, and each size fraction was mounted and studied separately.

Ten parallel traverses, 2 cm. long, were run on each block. In the solid blocks the traverses were 4 mm. apart. On the broken coal blocks the traverses were 2 mm. apart, except that 12 traverses were made (6 in one direction and 6 perpendicular to them) on a broken coal sample that was screened and mounted in five separate bakelite blocks. The results of the 60 traverses on this sample were averaged and against these averages the solid-block count was checked. On the broken coal blocks of three samples—the Imboden coal, sample 1 of the No. 7 coal, and the "Cutler Rider" coal—an additional ten traverses were made perpendicular to the first ten to help in determining the proper number of traverses to give the least differential error. An average of 100 coal particles were intersected in each 2 cm. traverse, making an approximate total of 1000 particles of coal observed in each ten-traverse broken coal analysis, 2000 particles in the 20-traverse, and 6000 particles in the sized sample. From table 2 it can be seen that neither these additional traverses nor a detailed study of the sized sample was necessary for increased accuracy, as the average percentage difference for the nine samples is 1.60. Only one (sample 1 of the No. 7 coal) of the four tests in question gave a difference less than this. The results of this phase of the

<sup>9</sup> A. I. Buehler Ltd.

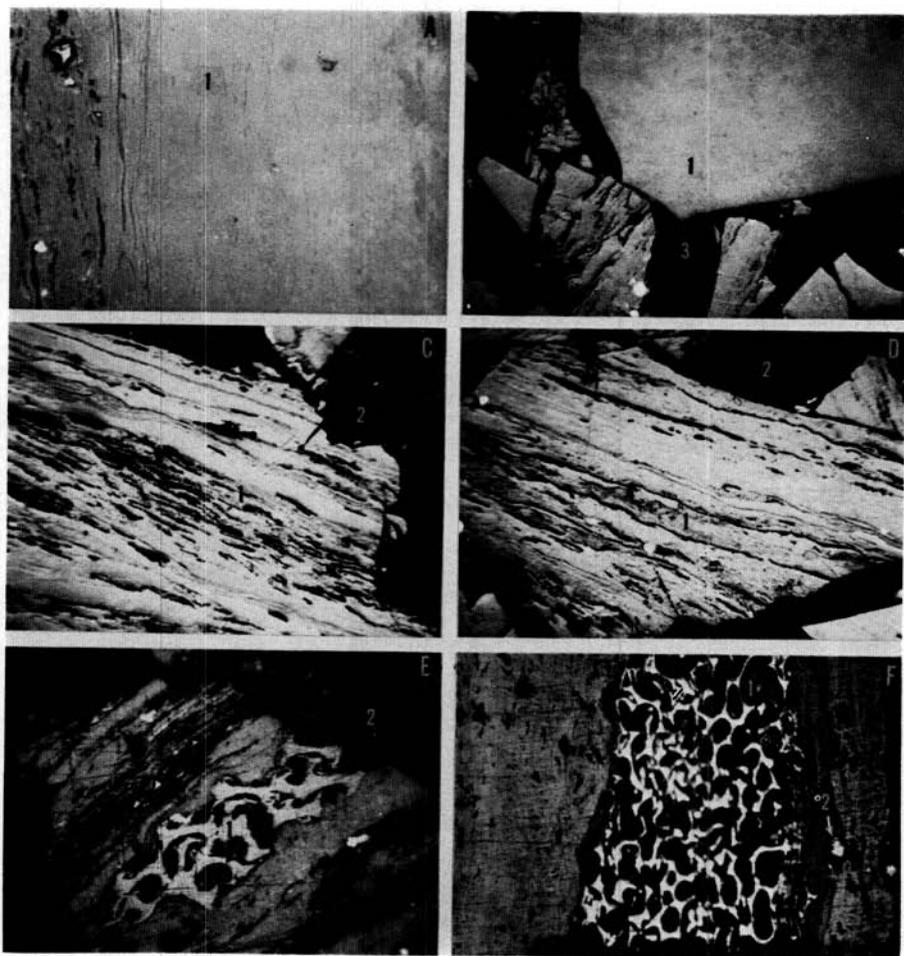


FIG. 1.—Microphotographs of coal (approx. x 200)

- A. 1. Vitrain. 2. Clarain.
- B. 1. Vitrain. 2. Clarain. 3. Mounting medium.
- C. 1. Clarain; the grey groundmass of vitrinite shows inclusion of rather black spores. 2. Mounting medium.
- D. 1. Clarain, spores, and a number of thin-toothed cuticles are included in the grey vitrinite groundmass. 2. Mounting medium.
- E. 1. An inertinite band in the clarain. Fusain shows "bogen structure" (cell walls broken and pushed into each other). 2. Mounting medium.
- F. 1. Inertinite; fusinite with semifusinite on the left. 2. A mass of small pyrite crystals between the semifusinite and vitrinite.

studies indicate that ten traverses are adequate to give an accurate analysis.

The terminology relative to banded ingredients follows that used by the German workers, with some modifications. Durain is generally absent in Illinois coals;<sup>10</sup> therefore, the maceral micrinite, where present, was included with fusinite and semifusinite under the term inertinite. An arbitrary lower limit of 25 microns was placed on vitrain. Pyrite, siderite, quartz, and other minerals were included under mineral matter.

Future work should include a maceral determination using no arbitrary limits, in addition to the banded-ingredient analysis. This method of representation of quantitative petrographic analysis would be similar to that proposed by van Krevelen.<sup>11</sup>

<sup>10</sup> McCabe, L. C., Changes in the constitution of Illinois coals through preparation processes, and the importance of these changes on utilization: Illinois Geol. Survey Circ. 23A, pp. 13-15, 1938.

<sup>11</sup> Van Krevelen, D. W., Representation of the quantitative petrographical analysis by means of a decimal code; A proposal made before the first session of the International Committee on Coal Petrology at Galeen (Holland) June 8 to 11, 1953.

## CONCLUSIONS

The results obtained in this study show that the techniques used in preparing the coal samples enable the Rosiwal analysis to be used in any anthracological study requiring this type of analysis. The data obtained in this specific application show that the percentage of the banded ingredients in a *small* block of coal compares favorably with the percentage of banded ingredients in the same coal after it has been broken and analyzed by the Rosiwal method.

Some studies that may be improved by utilizing these techniques are:

- (1) The accumulation of data on the effect of mining and preparation processes on the ingredient composition of commercially produced coals,
- (2) the accumulation of data bearing on the results of laboratory methods used for the separation and concentration of banded constituents,
- (3) the analysis of selected samples of coals which have unusual burning characteristics in stoker tests, and
- (4) information on the preparation of special samples of coal for stoker and coking tests under laboratory control.