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DETERMINING THE FUSAIN CONTENT OF ILLINOIS COALS

A Comparison of Chemical and Petrographic Methods

BY

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DETERMINING THE FUSAIN CONTENT OF ILLINOIS COALS

A Comparison of Chemical and Petrographic Methods

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B. C. Parks, G. W. Land[1], and O. W. Rees

The method commonly referred to as microscopic count or petrographic analysis used for quantitatively determining the different ingredients of banded coal has been standard practice for several years in the coal laboratory of the Illinois State Geological Survey. However, this type of analysis is long and tedious, is subject to personal errors, and does not permit concurrent duplicate determinations. Because of these disadvantages a less time-consuming and less laborious method for determining fusain is desirable. This investigation, therefore, was for the purpose of determining whether the chemical method described by Fuchs, Gauger, Hsiao, and Wright (*2*) would provide values for fusain in sufficiently close agreement with those obtained by the petrographic method to justify its exclusive use.

Petrographic Method

The procedure involves two distinct series of steps after preparation of the sample and riffling a desired amount (approximately 500 grams). In the first step the sample selected by riffling is weighed and separated into size classes by screening through a series of Tyler standard sieves. Twelve sieves generally are used and these are grouped into two nests of six sieves each for convenience in using the Ro-Tap shaker. The sieves range from No. 8 mesh to No. 300 mesh. The size of the openings in the individual sieves decreases in the ratio of the square root of two. The openings of the No. 8 mesh screen are 2.36 mm. square, and of the No. 300 mesh are 0.046 mm. square. The material retained on eleven of the screens, after shaking through the first nest (8- to 35-mesh) for 10 minutes and the second nest (48- to 300-mesh) for 15 minutes, consists of sorted particles, each group having a relatively small size range. In addition to the sized material there are two unsorted batches, the 2.36-mm. material retained on the No. 8 mesh screen and the contents of the pan which passed through the No. 300 mesh screen (Table I).

The essential purpose of the screening is to facilitate the second step of manually separating and counting the component particles. For this work an ore-dressing type of binocular microscope is used.

Table I.—Petrographic Estimation of Fusain Content in a Fine Coal

⅜-inch screenings (raw carbon)[a]

Screen Mesh No.	Particle Sizes Retained on Screens *Mm.*	Screening Analysis *Grams*	*%*
8	+2.36	7.40	3.87
10	−2.36 +1.651	22.10	11.56
14	−1.651+1.17	26.50	13.86
20	−1.17 +8.33	24.40	12.76
28	−8.33 +0 590	21.40	11.19
35	−0.590+0.417	18.30	9.57
48	−0.417+0.300	14.20	7.43
65	−0.300+0 208	10 50	5.49
100	−0.208+0 150	10.10	5.28
150	−0.150+0.104	6.10	3.19
200	−0.104+0.075	6.50	3.40
300	−0.075+0.046	3.30	1.73
Pan	−0.046	20.40	10.67
		191.20	100 00

[a] Sample from preparation plant of Madison County, Ill., mine.

[1] Present address, Battelle Memorial Institute, Columbus, Ohio.

TABLE II.—PETROGRAPHIC ESTIMATION OF FUSAIN CONTENT IN A FINE COAL

⅜-inch screenings, microscopic count

Screen Mesh No.	Particle Sizes Retained	Separation and Count			Distribution of Fusain		
		Fusain	Non-fusain	Total	Fusain and non-fusain	Fusain	%
	Mm.				*Grams*	*Grams*	
8	+2.36	104	1700	1804	7.40	0.43	5.76
10	—2.36 +1.651	142	2021	2163	22.10	1.45	6.56
14	—1.651+1.17	151	1950	2101	26.50	1.91	7.19
20	—1.17 +0.833	181	2196	2377	24.40	1.87	7.67
28	—0.833+0.590	210	2348	2558	21.40	1.76	8.21
35	—0.590+0.417	251	2489	2740	18.30	1.68	9.16
48	—0.417+0.300	234	2125	2359	14.20	1.41	9.92
65	—0.300+0.208	333	2584	2917	10.50	1.20	11.42
100	—0.208+0.150	322	2058	2380	10.10	1.30	12.90
150	—0.150+0.104	298	1400	1698	6.10	1.07	17.55
200	—0.104+0.075	264	800	1064	6.50	1.61	24.81
300	—0.075+0.046	408	500	908	3.30	1.48	44.93
Pan	—0.046	560	180	740	20.24	15.44	75.67
Total weight of sample, grams		191.20					
Weight of nonfusain, grams		158.59					
Weight of fusain, grams		32.61					
Content of fusain in sample, %		17.06					

In the separation and counting of fusain particles use is made of the distinctly different appearance of fusain and of the other constituents. In contrast to particles of the other constituents fusain particles are flaky and needle-shaped with surface striations and pits often characteristic. These characteristic differences are maintained throughout the entire range of sizes used in the petrographic method of estimation.

In making fusain estimations the number of particles separated and counted in order to arrive at the correct fusain percentage in each size class is dependent upon the frequency of distribution of fusain in the particular class. For the size classes containing less than 10 per cent fusain a count of 2500 particles gave results which could be duplicated within 0.2 per cent by different operators. For size classes which had in excess of 10 per cent fusain good results were obtained by counting as few as 1000 particles in some classes (Table II).

It is obvious that the fusain percentages obtained by separating and counting particles of coal in this manner are not true weight percentages. If all particles in the two separated groups of fusain and nonfusain were spheres of the same size and specific gravity, true weight percentages could be calculated from the count data. It is known, however, that the ingredients of banded coal do not break down into spherical particles and that specific gravity is variable. Notwithstanding this, it was assumed, for practical purposes in obtaining satisfactory results, that every particle of coal separated and counted could be regarded as having the same size and specific gravity. The validity

TABLE III.—COMPARISON OF FUSAIN VALUES BY COUNT AND BY WEIGHT

Microscopic method

Grade Size (Screen Mesh)	Per Cent by Weight	Per Cent by Count
8-10	2.3	2.6
10-14	2.8	2.5
14-20	4.0	3.6
20-28	3.9	3.9
28-35	4.1	3.9
8-10	1.5	2.1
10-14	2.2	1.8
14-20	2.4	2.3
20-28	2.9	2.4
10-14	2.7	2.6

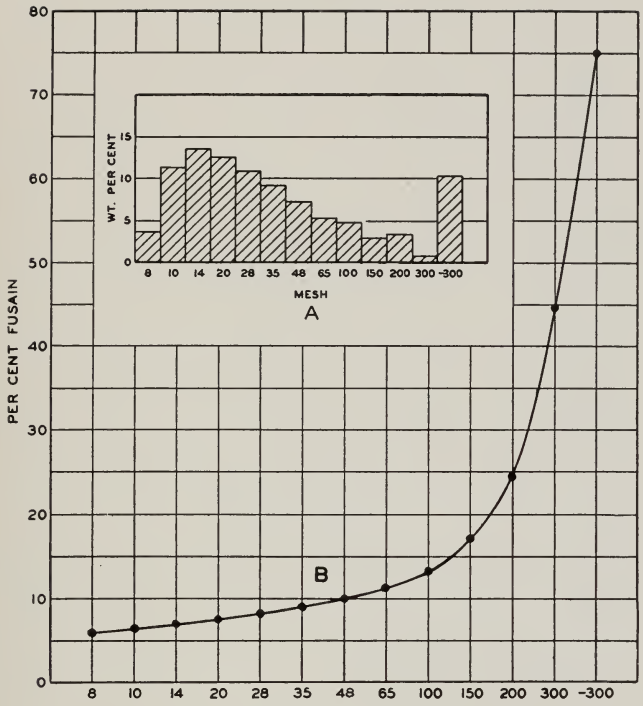


FIGURE 1.—DISTRIBUTION OF FUSAIN

A. Histogram showing distribution of coal by weight percentages on screens after Ro-Tap operation of ten minutes for +35-mesh and 15 minutes for —35-mesh screens. Sample same as Table I

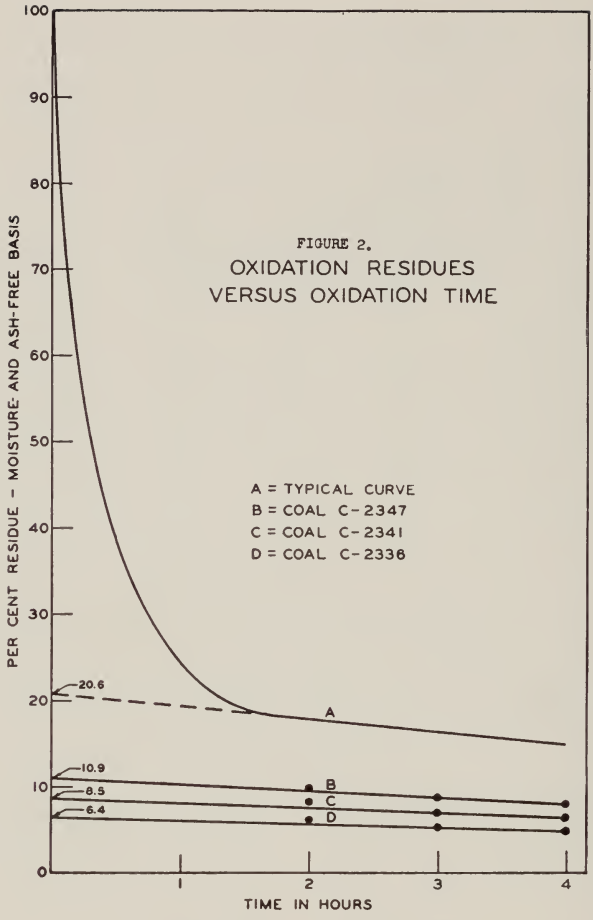
B. Direct plot curve showing distribution of fusain by weight percentages in coal retained on screens in *A*

of this assumption was tested by weighing the fusain and nonfusain of ten size classes, calculating true weight percentages, and comparing these with results obtained from count data. The results so obtained are in reasonably close agreement (Table III).

The estimation of fusain by the petrographic method here described is probably subject to unavoidable human errors and is time-consuming. The identification and separation of fusain from nonfusain in the fine sizes are particularly difficult. Eye and muscular fatigue are probably the chief causes of error in identifying, separating, and counting the extremely fine particles. An experienced petrographic observer working carefully can complete an analysis of a coal sample in about one week, and there is no short cut in making a duplicate count. An advantage afforded by petrographic analysis lies in the data obtained with which curves can be drawn showing size frequency distribution of fusain in the sample (Figure 1).

CHEMICAL METHOD

The chemical method as described by Hsiao and co-authors (*2*) depends upon the relative susceptibility to oxidation of fusain and nonfusain coal material. Briefly, this procedure consists of oxidizing 1-gram portions of —200-mesh coal by boiling in 8 *N* nitric acid for 2-, 3-, and 4-hour periods. After proper washing with sodium hydroxide, hydrochloric acid, and water the residues are dried at 105°C., weighed, and ashed, and the weights of ash are deducted from the weights of oxidation residues. These



ash-free residues are then calculated to per cent of moisture- and ash-free coal and plotted against oxidation time. A straight line is drawn through these points (through the last two points if impossible to draw through all points) and extrapolated to cut the zero time axis, and the corresponding residue value is taken as the fusain value. Moisture and ash determinations were made by A. S. T. M. procedures (*1*).

Figure 2 presents curves for three of the samples studied, showing their extrapolation to the zero time axis, as well as a typical oxidation curve (curve *A*) for times from 10 minutes to 4 hours. This curve appears to be made up of two parts, the first or curvilinear portion representing the oxidation of nonfusain coal material and the second flat portion representing the oxidation of the fusain. The curved portion appears to represent a first-order reaction while the flat portion appears to represent a zero-order reaction where extrapolation is easily possible.

Results

Thirteen coal samples were analyzed for fusain content independently by both methods. A comparison was made of the results and certain incidental factors were also observed, such as length of time required for the analyses and the probability of errors. The fusain content of the thirteen samples as determined chemically and microscopically is given in Table IV. The values check reasonably well. In only four of the samples was the difference in the percentage figures more than 2 per cent. In the other nine samples the difference between chemical and microscopic results varied from 0.0 to 1.8 per cent.

Table IV.—Results of Tests

Lab. No.	Moisture in Coal %	Ash in Coal %	Fusain		
			Chemical method[a] %	Petrographic method %	Difference %
C-2336 (1)	5.1	32.2	6.4	6.4	0.0
C-2337 (2)	6.5	24.2	5.4	7.2	+1.8
C-2338 (3)	5.5	41.5	14.2	9.4	—4.8
C-2339 (4)	7.1	25.6	18.0	15.2	—2.8
C-2340 (5)	2.7	18.8	19.0	19.5	+0.5
C-2341 (6)	5.1	24.2	8.5	7.5	—1.0
C-2342 (7)	6.1	19.3	10.1	7.4	—2.7
C-2343 (8)	5.2	11.0	11.5	12.6	+1.2
C-2344 (9)	5.9	23.2	12.8	10.4	—2.4
C-2345 (10)	6.2	19.2	15.9	14.4	—1.5
C-2346 (11)	5.1	10.9	4.2	4.0	—0.2
C-2347 (12)	7.6	13.4	10.9	10.8	—0.1
C-2348 (13)	5.5	9.8	13.8	12.1	—1.7

[a] Moisture- and ash-free basis.

The time necessary for conducting the chemical determinations, including duplicate tests of each sample, was 2 days, as compared to 5 to 10 days required for the petrographic estimations.

Certain of the residues from the chemical analyses were saved and examined carefully under the microscope. No organic material other than fusain could be found. The fusain in these residues was unusually easy to identify.

Conclusions

These tests indicate that in the majority of cases practically the same results are obtained by each method. Where different values are obtained, the greater complexity of the petrographic method and its greater dependence on personal judgment seem to favor the results obtained by the oxidation method.

The advantages of using the chemical method as indicated by these tests are summarized as follows: A considerable saving of time is effected. Duplicate analyses can be made simultaneously. There is less liability of human error. The values obtained represent fusain on an ash- and moisture-free weight basis.

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