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URBANA

REPORT OF INVESTIGATIONS — NO. 81

DETERMINATION OF ASH IN COALS UNUSUALLY
HIGH IN CALCITE AND PYRITE

BY

O. W. REES and W. A. SELVIG

IN COOPERATION WITH
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DETERMINATION OF ASH IN COALS UNUSUALLY HIGH IN CALCITE AND PYRITE

BY

O. W. REES¹ AND W. A. SELVIG²

IN DETERMINING ash in coals containing unusually large amounts of calcite and pyrite, difficulty in obtaining satisfactory results by the standard A.S.T.M. procedure (3)³ may be experienced, because of the varying amounts of sulfur that are retained as calcium sulfate in the ash. Variations in the heating procedure used in the ash determination influence the amount of sulfur retained. Lower ash and lower sulfur in the ash are obtained by slow rates of heating.

This paper is a report of cooperative work done by the Analytical Laboratory of the Illinois State Geological Survey and the Pittsburgh Laboratory of the United States Bureau of Mines for the purpose of studying modified procedures for the determination of ash in such troublesome coals.

EXPERIMENTAL TESTS

For this work five sizes of coal prepared by screening from a large sample of 1¼ inch × 0 screenings from the No. 2 bed, Woodford County, Ill., were used. The larger sizes were crushed to pass a No. 4 sieve and after mixing and riffing all sizes, two 1-quart samples of each were cut out. One set of samples was sent to the U. S. Bureau of Mines Laboratory and one set was retained in the Illinois State Geological Survey Laboratory.

The samples are designated as follows:

Sample	Size	Sample	Size
1	1¼ × ¾ inch	4	10 × 48-mesh
2	¾ × ¾ inch	5	-48-mesh
3	¾ inch × 10-mesh		

Each laboratory prepared its own samples for analysis by the usual A.S.T.M. procedure (1). Analyses in the two laboratories indicated that the two samples of each size were satisfactory duplicates, with the exception of sample 3. Because the two 1-quart portions differed in ash content by too large an amount, the ash values reported by the Geological Survey laboratory were obtained on the 60-mesh coal prepared in the Pittsburgh laboratory.

The coals were analyzed for total sulfur (Eschka method), and for forms of sulfur and carbon dioxide by methods of the U. S. Bureau of Mines (7). Moisture at 105°C. was determined by each laboratory on the samples according to the A.S.T.M. standard method (2).

BUREAU OF MINES DETERMINATIONS

METHOD A. The dried samples were heated on the hearth of a hot muffle furnace for 8 minutes to drive off volatile matter, then heated at 725°C. to constant weight (within 0.001 gram).

METHOD B. The dried samples were placed in a cold furnace and heated to 725°C. in 1.5 hours. The temperature was kept at 725°C. to constant weight.

METHOD C (Parr's sulfated ash method, 5, 6). The dried samples were burned by Method A and after cooling were moistened with a few drops of 1 to 1 sulfuric acid and dried on an air bath until the fumes were largely driven off. The samples were then heated at 725°C. to constant weight. This treatment produces a sulfated ash in which it is assumed that all calcium is present as the sulfate. To correct to a calcium oxide

¹ Illinois State Geological Survey, Urbana, Ill.
² U. S. Bureau of Mines, Pittsburgh, Penna.
³ Italic numbers in parentheses refer to literature cited at the end of the report.

basis the percentage of sulfur trioxide, coal basis, determined in the sulfated ash is subtracted from the sulfated ash or 1.82 times the mineral carbon dioxide, coal basis, is subtracted from the sulfated ash. In the latter method of correction it is assumed that the mineral carbon dioxide is a measure of the calcium carbonate present in the original mineral matter of the coal.

TABLE I. SULFUR FORMS AND CARBON DIOXIDE

	Sample 1 %	Sample 2 %	Sample 3 %	Sample 4 %	Sample 5 %
Sulfate sulfur					
U. S. B. M.	0.06	0.08	0.07	0.11	0.12
I. G. S.	0.01	0.02		0.04	0.07
Pyritic sulfur					
U. S. B. M.	0.67	1.03	1.30	2.28	3.14
I. G. S.	1.02	1.10		2.56	3.50
Organic sulfur					
U. S. B. M.	0.72	0.69	0.58	0.55	0.52
I. G. S.	0.53	0.49		0.40	0.19
Total sulfur					
U. S. B. M.	1.45	1.80	1.95	2.94	3.78
I. G. S.	1.56	1.61		3.00	3.76
Carbon dioxide					
U. S. B. M.	0.29	0.53	1.59	4.24	3.63
I. G. S.	0.47	0.64		4.10	3.64

METHOD D. The dried samples were placed in a cold furnace and heated to 400°C. in 0.5 hour. They were held at this temperature for a further 0.5 hour, then transferred to another furnace at 725°C. and heated to constant weight.

One ash sample from each test was analyzed for total sulfur by the sodium carbonate fusion method (7). The duplicate ash was tested for sulfide sulfur by the evolution method used for coke (8). Sulfate sulfur was determined in the hydrochloric acid solution from the sulfide tests. Ammonium hydroxide was added in slight excess and the precipitated iron together with any insoluble ash was removed by filtration. The filtrate was made slightly acid and the sulfur precipitated in the regular manner.

GEOLOGICAL SURVEY DETERMINATIONS

METHOD E. The dried samples were heated on the hearth of a hot muffle furnace for 10 minutes to drive off volatile matter, moved just inside the furnace for 5 minutes, and then moved back to

the hot portion of the furnace where they were heated to constant weight at 750°C.

METHOD F. The dried samples were placed in a cold muffle furnace and heated to 750°C. in 1.75 hours. They were heated to constant weight at this temperature.

METHOD G (Parr's sulfated ash method). The dried samples were burned by Method F and after cooling were treated with 1 to 1 sulfuric acid, the excess acid was fumed off on a hot plate, and the samples were heated to constant weight at 750°C.

The ashes obtained by all three methods were analyzed for sulfur trioxide by extraction with dilute hydrochloric acid, precipitation and removal of R_2O_3 with ammonium hydroxide, with subsequent precipitation of sulfate with barium chloride in acid solution. Sulfide sulfur determinations were made on the ashes obtained by Methods E and F by the procedure mentioned above (8).

The effect of using furnaces of different sizes, in which the rate of change of atmosphere varied, for duplicate determinations was studied in the Geological Survey laboratory. One duplicate was ashed in the larger Hoskins F. D. 204 furnace whose heating chamber is 7½ inches wide, 5¼ inches high, and 14 inches long while the other was ashed in the smaller Hoskins F. D. 202 furnace whose heating chamber is 4¼ inches wide, 3 inches high, and 10 inches long. Both furnaces were equipped with thermocouples placed loosely through the back, so that air flow through the furnace would take place.

All tests in both laboratories were made in electrically heated muffle furnaces.

RESULTS OF TESTS

Results of analyses of the coals for forms of sulfur and mineral carbon dioxide are given in Table I. Ash values as determined by the various methods, together with amounts of sulfur as sulfur trioxide retained in the ashes, are listed in Tables II and III. Sulfide sulfur was found only in ashes obtained by Methods A and E. Table

IV presents additional data showing the effect of a slower rate of heating and ashing in furnaces of different sizes in which air circulation was different.

Table V presents a comparison of average results obtained by different methods.

TABLE II. DETERMINATION OF ASH
(Per cent of dry coal)

Sample	Ash	SO ³ in Ash	Sulfide in Ash	SO ³ - and S-Free Ash	Ash	SO ³ in Ash	Sulfide in Ash	SO ³ - and S-Free Ash
Data by Method A								
1	9.28	0.46	8.82	9.40	0.29	0.04	9.09
	9.27	0.40	0.00	8.87	9.50	0.30	0.02	9.19
Av.	9.28			8.85	9.45			9.14
2	14.37	0.62	13.72	13.80	0.22	0.02	13.57
	14.20	0.57	0.03	13.60	13.66	0.31	0.03	13.34
Av.	14.29			13.66	13.73			13.46
3	18.59	1.52	17.06	18.06
	18.70	1.56	0.01	17.13	17.87
Av.	18.65			17.10	17.97		
4	31.27	3.64	27.61	29.26	1.73	0.02	27.52
	31.15	3.49	0.02	27.64	28.92	1.28	0.02	27.63
Av.	31.21			27.63	29.09			27.58
5	34.93	3.14	31.73	34.83	1.86	0.32	32.81
	35.12	3.03	0.06	32.03	34.65	1.59	0.27	32.92
Av.	35.03			31.88	34.74			32.87
Data by Method B								
1	9.13	0.29		SO ³ -Free Ash 8.84	9.32	0.23		SO ³ -Free Ash 9.09
	9.09	0.26		8.83	9.33	0.24		9.09
Av.	9.11			8.84	9.33			9.09
2	14.09	0.40		13.69	13.77	0.23		13.54
	14.06	0.34		13.72
Av.	14.08			13.71	13.77			13.54
3	17.87	0.85		17.02	17.53
	17.80	0.68		17.12	17.85
Av.	17.84			17.07	17.69		
4	29.04	1.39		27.65	28.81	0.70		28.11
	28.91	1.07		27.84	28.14	0.68		27.46
Av.	28.98			27.75	28.48			27.79
5	33.19	1.01		32.18	34.73	1.45		33.28
	33.01	0.83		32.18	34.03	1.24		32.79
Av.	33.10			32.18	34.38			33.04
Data by Method D								
1	9.12	0.29		8.83				
	9.10	0.24		8.86				
Av.	9.11			8.85				
2	14.03	0.38		13.65				
	14.05	0.34		13.71				
Av.	14.04			13.68				
3	17.58	0.53		17.05				
	17.71	0.61		17.10				
Av.	17.65			17.08				
4	28.53	0.86		27.67				
	28.49	0.76		27.73				
Av.	28.51			27.70				
5	32.95	1.02		31.93				
	33.16	0.95		32.21				
Av.	33.06			32.07				

DETERMINATION OF ASH IN COALS

TABLE III. DETERMINATION OF ASH
(Per cent of dry coal)

Sample	Sulfated Ash	SO ³ in Sulfated Ash	SO ³ -Free Ash	Sulfated Ash Less 1.82 × Mineral CO ²	Sulfated Ash	SO ³ in Sulfated Ash	SO ³ -Free Ash	Sulfated Ash Less 1.82 × Mineral CO ²
	Results by Method C				Results by Method G			
1	9.81	1.05	8.76	9.28	10.15	0.76	9.39	9.29
	9.82	1.00	8.82	9.29	10.23	0.84	9.39	9.37
Av.	9.82		8.79	9.29	10.19		9.39	9.33
2	15.15	1.54	13.61	14.22	14.86	1.21	13.65	13.70
	15.18	1.50	13.68	14.25	14.99	1.25	13.74	13.83
Av.	15.17		13.65	14.24	14.93		13.70	13.77
3	20.43	3.43	17.00	17.56
	20.49	3.49	17.00	17.62
Av.	20.46		17.00	17.59
4	35.62	8.09	27.53	27.88	35.95	7.85	28.10	28.49
	35.68	8.15	27.53	27.94	35.71	7.77	27.94	28.25
Av.	35.65		27.53	27.91	35.83		28.02	28.37
5	39.38	7.42	31.96	32.78	40.68	7.16	33.52	34.06
	39.59	7.50	32.09	32.99	40.67	7.21	33.46	34.05
Av.	39.49		32.03	32.89	40.68		33.49	34.06

DISCUSSION

The tolerances for permissible differences between ash determinations in the same laboratory and between different laboratories by the A.S.T.M. standard method (4) are 0.3 and 0.5 per cent, respectively, on coals containing carbonates. For coals with more than 12 per cent ash containing carbonate and pyrite these are 0.5 and 1.0 per cent, respectively.

For the most part results obtained by the various methods checked within these tolerances, both in the same laboratory and between the two laboratories (Tables II and III), particularly the results corrected for retained sulfur. In a few cases the results did not check within these tolerances but, with one exception, these results were for the last two samples (samples 4 and 5) which are high in mineral matter containing large amounts of mineral carbon dioxide and pyritic sulfur. The ash of these samples is considerably higher than would normally be encountered in commercial samples.

The ash values obtained by Methods A and E, uncorrected for retained sulfur, are higher than those obtained by other methods with the exception of the

Parr sulfated ashes. Methods A and E correspond to a more rapid rate of heating and the ashes contain considerably greater amounts of retained sulfur than those obtained by Methods B, F, and D. Results obtained by Method D probably show the best agreement between duplicate determinations, with comparatively lower amounts of sulfur being retained in the ashes. Results obtained by the Parr sulfated ash method check reasonably well for the most part, but this procedure is rather long and requires mineral carbon dioxide values for use in correcting the sulfated ash obtained.

In Table IV are presented further data on the effect of different rates of heating, not corrected for sulfur retained in the ash. Lower results are obtained with the slower rate of heating. In addition, information is presented on the use of different sized furnaces for ashing. The higher results obtained in the smaller F. D. 202 furnace indicate that sulfur trioxide as formed was not removed as rapidly as in the larger furnace and was therefore fixed in the ash to a larger extent. The adequate removal of sulfur trioxide therefore becomes important. Use of the Parr sulfated ash method appears to smooth out these differences to some extent.

HIGH IN CALCITE AND PYRITE

TABLE IV. COMPARISON OF ASH VALUES

Obtained by varying time necessary for furnace to reach 750° C., by ashing in different sized furnaces, and by applying the modified Parr method where first burning off took place in different sized furnaces. Data by Illinois Geological Survey.

Sample	Per Cent of Ash Starting with Cold Furnace, Heating to 750° C.		Per Cent Ash, Starting with Cold Furnace, Heating to 750° C. in 1.75 Hours		Per Cent Ash, Sulfated Modified Parr Method		Per Cent Ash, Modified Parr, Sulfated Ash Less 1.82 × Mineral CO ²
	1.5 hours	2.25 hours	Large furnace, Hoskins F. D. 204	Small furnace, Hoskins F. D. 202	Large furnace, Hoskins F. D. 204	Small furnace, Hoskins F. D. 202	
1	9.14	9.08	9.00	9.24	9.59	9.53	9.03
2	14.08	14.05	14.02	...	14.76	14.89	13.87
3	17.89	17.60	17.46	17.73	20.08	20.15	17.23
4	29.07	28.52	28.66	29.91	35.46	35.70	27.87
5	33.28	32.98	32.76	33.16	39.16	39.39	32.68

Table V presents a comparison of average ash values obtained by the procedures tried. For those samples containing larger amounts of ash and mineral carbon dioxide the slower rates of heating give definitely better results when compared to those obtained by the Parr sulfated ash method. However,

differences appear which are outside A.S.T.M. tolerances and in such cases the ash values should be corrected for retained sulfur or determined by the modified Parr method. It is not likely that such samples would be encountered in commercial samples but they might be encountered in certain special studies.

TABLE V. COMPARISON OF AVERAGE ASH VALUES OBTAINED BY METHODS A, B, D, E, AND F WITH METHODS C AND G
(Per cent of dry coal)

Sample	Data by U. S. Bureau of Mines				Difference from Method C		
	Method A, hot muffle	Method B, cold muffle	Method D, two muffles	Method C, Parr's method ^a	Method A	Method B	Method D
1	9.28	9.11	9.11	9.29	-0.01	-0.18	-0.18
2	14.29	14.08	14.04	14.24	+0.05	-0.16	-0.20
3	18.65	17.84	17.65	17.59	+1.06	+0.25	+0.06
4	31.21	28.98	28.51	27.91	+3.30	+1.07	+0.60
5	35.03	33.10	33.06	32.89	+2.14	+0.21	+0.17

	Data by Illinois Geological Survey			Difference from Method G	
	Method E, hot muffle	Method F, cold muffle	Method G, Parr's Method ^a	Method E	Method F
1	9.45	9.33	9.33	+0.12	0.00
2	13.73	13.77	13.77	-0.04	0.00
3	17.97	17.69	17.23	+0.74	+0.46
4	29.09	28.48	28.37	+0.72	+0.11
5	34.74	34.38	34.06	+0.68	+0.32

^a Parr's sulfated ash less 1.82 × mineral CO₂.

SUMMARY

The preliminary hearth heating method (A and E) gave results within the A.S.T.M. tolerances for all duplicates obtained in the same laboratory. Checks between different laboratories within A.S.T.M. tolerances were obtained for coals containing up to about 3.6 per cent mineral carbon dioxide, but these ashes contained larger amounts of retained sulfur.

The determination of ash by the cold furnace method (B and F) gave duplicate results within A.S.T.M. tolerances for all samples in the U. S. Bureau of Mines laboratory and for samples up to about 1.6 per cent mineral carbon dioxide content in the Illinois Geological Survey laboratory. Checks between average values from the two laboratories were within A.S.T.M. tolerances for all samples.

Results obtained by the modified Parr method were all well within A.S.T.M. tolerances for duplicates in the same laboratory. With the exception of results on one sample (5), the checks between the two laboratories were within A.S.T.M. tolerances.

The determination of ash by Method D gives duplicates checking within the 0.3 per cent tolerance for all samples.

The adequate removal of sulfur trioxide from the furnace during ashing is important.

As compared to results obtained by the modified Parr procedure; Methods A and E gave results within the A.S.T.M. 0.3 per cent tolerance for samples containing up to 0.6 per cent of carbon dioxide; Method B for coals up to 3.6 per cent of carbon dioxide; Method F for coals up to 4.2 per cent of carbon dioxide (with one exception); and Method D for coals containing up to 3.6 per cent of carbon dioxide.

Sulfur trioxide-free ash values were similar for all methods.

The slow heating method starting with a cold furnace appears to be satisfactory for determining ash in commercial samples of coal containing unusually large amounts of calcite and pyrite.

CONCLUSIONS

Methods A and E are not recommended for determining ash in coals high in calcite and pyrite, because too much sulfur is retained in the ashes.

Methods B, D, and F give most consistent results, with Method D apparently giving the best results of the three.

Methods C and G appear to give good results, especially for coals high in calcite and pyrite where other procedures studied are not so satisfactory. This procedure requires more work than other procedures.

Adequate removal of sulfur trioxide from the furnace in which ashing takes place is necessary.

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