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were rejected because they failed to meet rock-dusting requirements. These samples included disintegrated slag, spent bentonite, calcium carbide residue, trap rock, and fly ash from boiler furnaces.

Fusain determination by a chemical oxidation method on two coals from the Palau No. 5 mine, Coahuila, Mexico, and from the Upper Elkhorn No. 3 bed, Knott County, Ky., showed moderate amounts of 1.7 percent fusain in the Mexican coal and 2.5 percent in the Kentucky coal.

Analyses of four soot-remover compounds showed that they contained 70 to 90 percent sodium chloride. Three of the samples contained zinc, and one sample contained 20 percent of copper chloride.

Chemical analyses of the 58 other miscellaneous materials were made in connection with investigations of complete gasification, coke production, coal hydrogenation, coal combustion, and mine accidents.

#### Constitution, Properties, and Analytical Methods

##### Extractable Waxes from Lignitic and Subbituminous Coals of the United States

Investigation of the possible extraction of montan wax, similar to that extracted commercially from certain European brown coals, from low-rank coals of the United States was continued during the year. As preliminary tests had indicated that Arkansas lignites might be a potential source of this wax, a survey was made of representative lignites of this State. Thirteen channel samples, collected by the Bureau of Mines and the Geology Division of the Arkansas Resources and Development Commission from 10 outcrop exposures and abandoned strip mines in Clay, Dallas, Hot Spring, Ouachita, Poinsett, and Saline Counties, were examined. The coals from these sources occur in the Wilcox formation and are classified as attrital lignites.

The yields of wax were determined by solvent extraction, using benzene and a benzene-alcohol mixture, the solvents reported to have been used in commercial extraction of German brown coal. Low yields of wax were obtained from two bench samples of Dallas County lignite which had very high ash contents and from the Clay County sample. The yields of wax from the other sources ranged from 5.7 to 11.1 and 9.6 to 16.4 percent on an air-dried-lignite basis, using benzene and benzene-alcohol, respectively, as solvent.

Tests of the chemical and physical properties of the benzene extracts showed that they are similar in many respects to the Riebeck brand of crude montan wax formerly imported in large amounts from Germany. Probably the most significant difference is the greater "resin" content of the wax from the Arkansas samples. In this respect they more closely resemble a commercial grade of montan wax imported from Czechoslovakia. The benzene-alcohol extracts had higher acid and saponification values than the benzene extracts and when heated appeared only to soften rather than completely fuse.

Petrographic analyses were made of column samples of lignite from Arkansas, Texas, and North Dakota in connection with an investigation of montan wax in American lignite. The results of the thin-section study showed that lignites giving relatively high yields of extractable wax were attrital in composition, and the low-yield lignites were mainly composed of woody tissue. The Arkansas lignites examined were typically attrital and resembled some of the German brown coal in

petrographic composition. The Texas and North Dakota lignites examined were xyloid (chiefly of woody tissue), with considerably less yellow translucent attritus than the bright attrital lignite from Arkansas.

#### Electron-Diffraction and Electron-Microscope Studies of Fischer-Tropsch Catalysts

Electron-diffraction techniques were utilized to determine the effective surface compounds in catalysts for the Fischer-Tropsch synthesis. X-ray diffraction methods are of value in revealing the nature of the bulk of the catalyst material, but they may not detect changes that take place at or near the surface of the catalyst. Electron diffraction is better-adapted to analyzing these surface compounds because electrons, not being as highly penetrating as X-rays, are diffracted in the surface layers.

One of the most widely held theories of the mechanism of the Fischer-Tropsch synthesis postulates that a carbide of the iron or cobalt catalyst is formed and that this compound is the effective catalytic agent. X-ray diffraction studies by the Bureau of Mines have indicated that cobalt carbide is not a factor in the synthesis on cobalt catalysts. Electron-diffraction results supported this finding. Patterns of cobalt carbide were readily obtained with intentionally carbided catalysts but not with those used in the synthesis. These showed patterns that are probably largely that of cobalt oxide (CoO), with possibly some metallic cobalt. Figure 3A, shows this type of pattern.

With iron catalysts, X-ray studies have shown indications of iron carbide in used catalysts, but electron-diffraction patterns have not shown the carbide. Nearly every used iron catalyst analyzed has shown a strong pattern of magnetite,  $Fe_3O_4$ , as illustrated in figure 3B. Thus, although not as yet conclusive, electron-diffraction evidence shows that oxides of iron and cobalt rather than carbides are the effective catalytic compounds.

Electron-microscope studies of Fischer-Tropsch catalysts<sup>13/</sup> revealed the effects of the presence in catalysts of promoters, such as magnesia and thoria, and of carriers, like kieselguhr, in inhibiting the sintering of cobalt on reduction, thus preserving a large proportion of the surface area upon which the catalytic activity is so dependent. Much smaller particle size of the cobalt results when the reduction is carried out in the presence of the promoters thoria and magnesia. Addition of a kieselguhr carrier also decreases the size of the cobalt particles. Measurements of the average particle sizes showed: Cobalt basic carbonate, 0.01 micron; reduced cobalt basic carbonate, 0.22 micron; reduced carbonate and promoters, 0.011 micron; reduced carbonate and kieselguhr, 0.028 micron. Surface-area measurements by a nitrogen-adsorption method confirm this beneficial behavior of promoters and kieselguhr.

A peculiar crystalline aggregation occurred in cobalt-metal powder reduced from cobalt oxide used in Fischer-Tropsch catalysts.<sup>14/</sup> The oxide particles sintered into larger smooth droplets of cobalt that were aggregated into thin, hexagonal platelets. The hexagons vary in diameter from 7 to 25 microns, and their thickness

<sup>13/</sup> Anderson, Robert B., McCartney, James T., Hall, W. Keith, and Hofer, L. J. E., Kieselguhrs. Suitability as Carriers in Catalysts: Ind. Eng. Chem., vol. 39, 1947, pp. 1618-1628.

<sup>14/</sup> McCartney, J. T., and Anderson, R. B., Crystalline Aggregation of Cobalt Powder: Jour. Appl. Phys., vol. 18, 1947, pp. 902-903.

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is apparently of the order of the diameter of the component particles. X-ray diffraction analysis also shows that the crystal phase present is hexagonal cobalt. These hexagonal aggregates may be formed by forces similar to those operating in normal crystal formation, but since the component particles do not lose their identity and the aggregates are easily fractured, they can hardly be classed as true crystals.

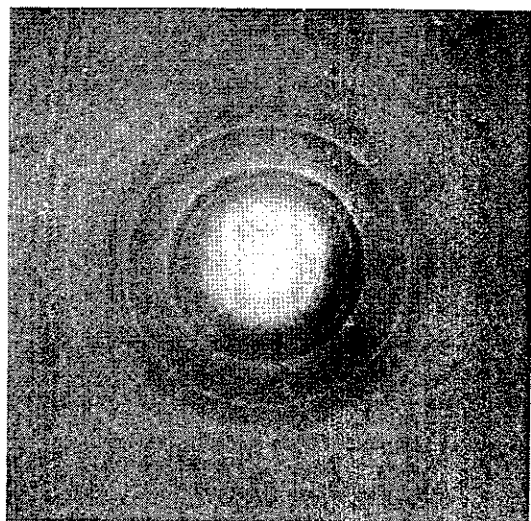
#### Electron-Microscope Studies of a Micro-Organism Occurring in Acid Mine Drainage

Research groups working on the problem of acid mine drainage and its contribution to stream pollution have devoted considerable attention to the role of bacteria in the formation of acid waters. Evidence has been given in the literature that an unidentified micro-organism appears to influence the oxidation of ferrous salts to less soluble ferric compounds that cause the objectionable rusty color of mine drainage and add to its acid content. Bureau of Mines electron-microscope studies have shown some interesting features of this micro-organism. Figure 4 is an electron micrograph of a pure culture of this bacterium. Figure 5 illustrates an apparent micro-organism that has been observed in natural and synthetic mine waters. The lower oval body in figure 4, displaying a dense nucleus or inclusion, is the common type found in the pure culture. At the top of figure 4 there is an apparent splitting of this organism or possibly a union of two, with disintegration of opposite rounded ends. Comparison of figures 4 and 5 indicates that this evolution probably results in the peculiar shape of the micro-organism found in mine drainage.

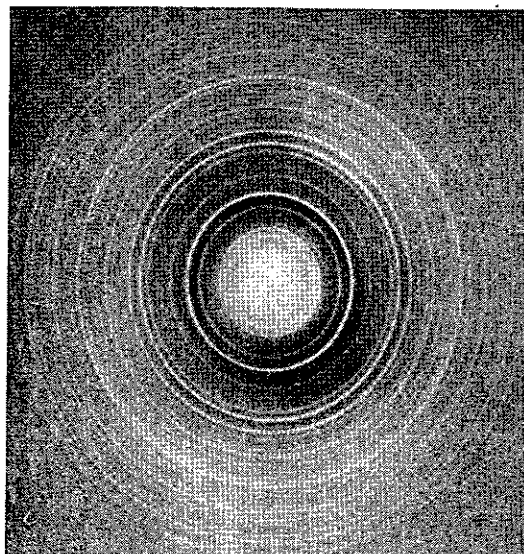
#### Petrographic Studies of Coal

Petrographic examinations were made of 627 feet of coal cores, and on the basis of the findings the coal was selectively sampled for special chemical and physical tests. The cores were recovered from the Bureau of Mines drilling projects in the Castleman Basin, Md., and at Eska, Alaska, and from the Bureau of Reclamation's drilling project at Moorhead Dam in the Powder River field, Wyo. Detailed strip logs were prepared of all cores studied to aid in field correlation of coal beds encountered in the test drilling.

A complete petrographic analysis by thin section study was made of a column sample of coal from the Fairview bed, Soot Creek mine, St. Clair County, Ala. The results of this analysis, showing the amounts of various components and types of coal, are presented in graphic form in figure 6. The same type of analysis was made of a drill-core sample of Fairview coal recovered from a test drill hole 1-1/2 miles from the Soot Creek mine. A comparison of the amounts of coal components determined in the analyses of the coal core and column sample is shown in table 1. This table shows a fusain content for the column sample almost double that of the coal core from the drill hole. The large difference in fusain content determined for two coal samples from the same coal bed is explained by the fact that the coal core recovered from the drill hole was in a badly broken condition, and much of the soft fusain had been pulverized to dust and lost in recovering and handling the core. The lower part of the table shows the coal components of the two samples recalculated on a fusain free basis and the results here indicate essentially the same petrographic composition for the Fairview coal at the two localities.



A



B

Figure 3. - Electron diffraction patterns of cobalt (A) and iron (B) catalysts that have been used in the Fischer-Tropsch synthesis.

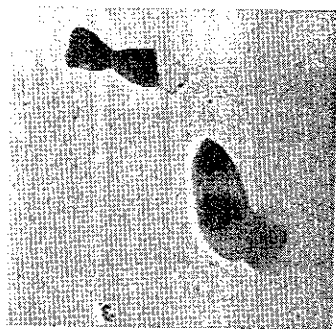


Figure 4. - Electron micrograph of a pure culture of a micro-organism found in acid mine water. 10,300X.



Figure 5. - Electron micrograph of micro-organisms in synthetic acid mine water. 10,300X.

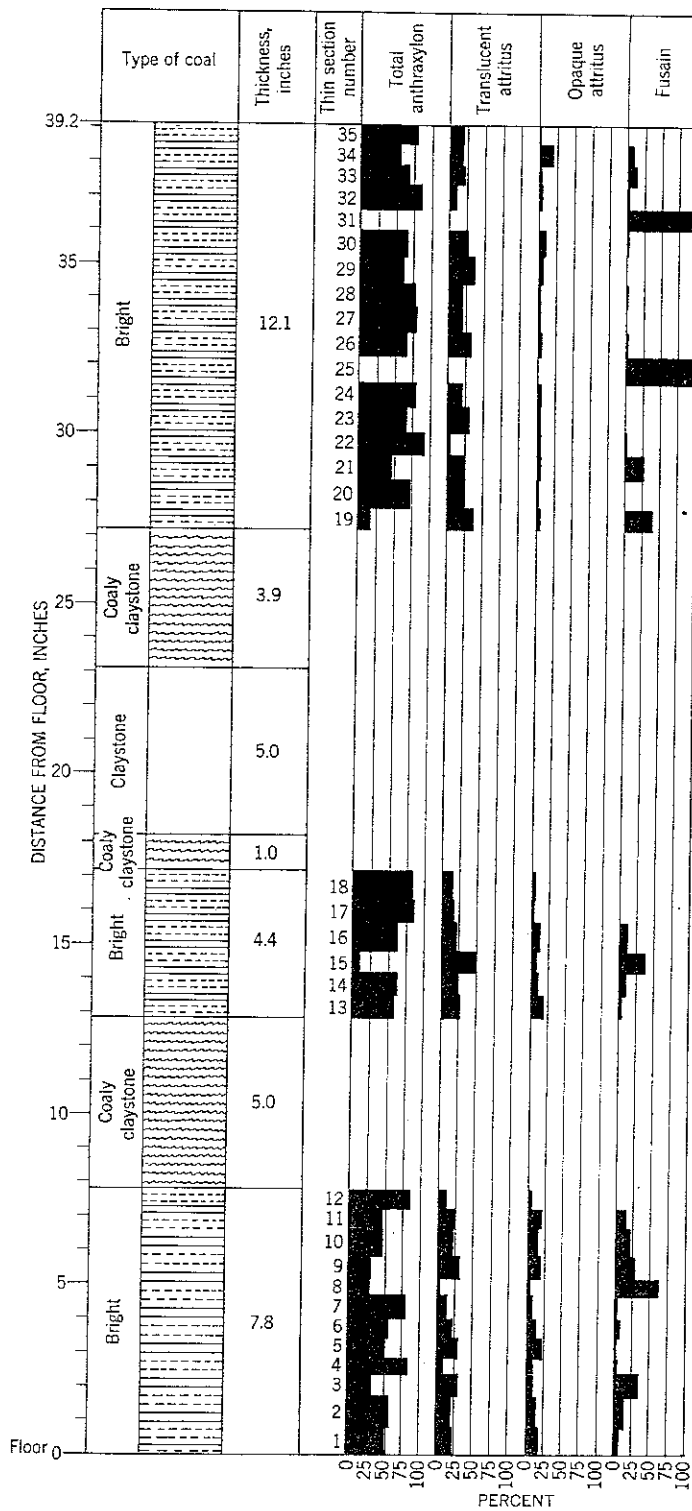


Figure 6. - Types of coal and percentage of components in Fairview Bed, Soot Creek mine, St. Clair County, Ala.



Specific-gravity fractions	Percent petrographic components						Ash of coal moisture-free basis percent
	Anthraxylon	Bright attritus	Dull attritus	Fusain	Mineral matter	Anthraxylon and bright attritus	
Float - 1.25	46.5	53.5	-	-	-	100	0.95
1.25 - 1.26	40.2	59.8	-	-	-	100	.90
1.26 - 1.27	32.0	68.0	-	-	-	100	1.15
1.27 - 1.28	37.2	62.8	-	-	-	100	1.53
1.28 - 1.29	36.6	59.8	3.2	0.4	-	96.4	2.31
1.29 - 1.30	25.1	67.6	6.5	.8	-	92.7	3.75
1.30 - 1.31	22.4	63.8	12.9	.9	-	86.2	5.00
1.31 - 1.32	19.8	60.1	18.7	1.4	-	79.9	5.69
1.32 - 1.33	21.2	53.4	20.9	2.2	0.3	76.6	6.74
1.33 - 1.34	18.8	49.1	27.0	4.9	.2	67.9	7.60
1.34 - 1.35	16.4	51.1	27.3	4.9	.3	67.5	9.18
1.35 - 1.37	14.1	38.8	41.1	5.5	.5	52.9	11.65
1.37 - 1.40	11.9	29.2	48.6	9.2	1.1	41.1	15.79
1.40 - 1.50	7.5	20.8	56.0	13.8	1.9	28.3	20.61
1.50 - 1.60	4.8	15.6	56.2	19.6	3.8	20.4	25.76
Sink - 1.60	1.4	4.0	44.8	13.6	36.2	5.4	64.49

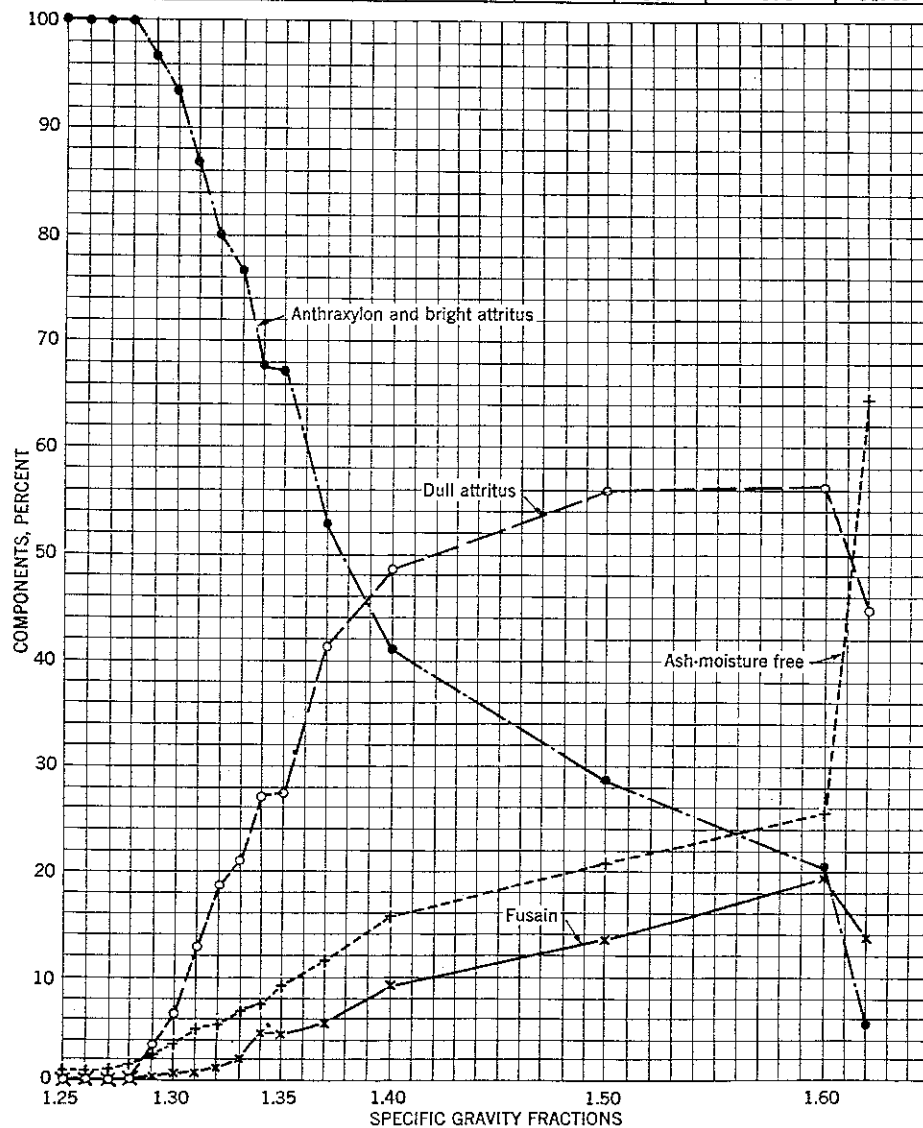


Figure 7. - Percentage of petrographic components of float-and-sink fractions. Rock Springs No. 3 bed coal, Sweetwater No. 2 mine, Sweetwater County, Wyo.

TABLE 1. - Comparison of petrographic composition of column sample and coal core from the Fairview bed, St. Clair County, Ala.

Coal	Sample	Percent of coal components			
		Anthraxylon	Translucent attritus	Opaque attritus	Fusain
Fairview	Column sample from mine .....	57.2	20.0	8.3	14.5
Do.	Coal core from drill hole .....	63.0	20.1	9.1	7.8
Do.	Column sample, fusain-free basis.	67	23	10	
Do.	Coal core, fusain-free basis .....	68	22	10	

Studies were made of the petrographic composition of 40 coal samples from coal-preparation investigations for synthetic liquid fuels research. The samples consisted of crushed coal of size range 14- by 100-mesh. These samples represented various specific gravity fractions from float at 1.25 sp. gr. to sink at 1.60 sp. gr. Quantitative petrographic analyses of the various gravity fractions were made by particle identification and count using a low-power stereoscopic microscope and accessories. Table 2 shows the source of the coals and petrographic analysis of the specific gravity fractions. Figure 7 shows by graph and table the petrographic composition of 16 specific-gravity fractions of Rock Springs No. 3-bed coal from the Sweetwater No. 2 mine, Sweetwater County, Wyo. All these analyses showed a common characteristic in concentration of anthraxylon and bright attritus in the light-gravity fractions and concentration of dull attritus and fusain in the heavy-gravity fractions. This suggests that gravity separation methods may be useful for obtaining a coal product high in anthraxylon and translucent attritus. These components of coal are hydrogenated more easily to liquid fuels than are the opaque attritus and fusain.

A petrographic study of meta-anthracite occurring in Newport and Providence Counties, R. I.,<sup>15</sup> revealed a preponderance of extremely fine grained minerals identified by X-ray analysis as consisting of large parts of quartz, mica, and chlorite. Mineral graphite, kaolinite, and dolomite also were noted. The minerals found in the meta-anthracite are common to highly metamorphosed rocks and are a secondary indication of the extreme metamorphism which has well-advanced the original coal material toward becoming a true graphitic rock. The high content of dispersed mineral matter strongly suggested that ash reduction by preparation methods would improve the meta-anthracite very little, and it could be considered an extremely low quality fuel at best.

<sup>15</sup> Toenges, Albert L., Turnbull, Louis A., Neale, Arthur, Schopf, J. M., Abernethy, R. F., and Quinn, Alonzo, Investigation of Meta-Anthracite in Newport and Providence Counties, R. I.; Petrography, Chemical Characteristics, and Geology of Deposits: Bureau of Mines Rept. of Investigations 4276, 1948, 37 pp.

TABLE 2. - Analytic summary of petrographic components of coal samples prepared in connection with coal-preparation studies for synthetic liquid fuels

Sample No.	Specific-gravity fraction	Anthrax-ylon	Bright attritus	Dull attritus	Fusain	Mineral matter	Source of coal
1	F. 1.25, S. 1.25, F. 1.31 .....	36.2	45.5	9.2	9.1	-	Indiana No. 4 coal, Saxton mine, Vigo County, Ind.
2	S. 1.31, F. 1.50	14.6	29.6	31.8	24.0	-	
3	S. 1.50 .....	3.2	5.7	32.0	41.8	17.3	
4	F. 1.25, S. 1.25, F. 1.33 .....	18.2	58.9	12.8	10.1	-	Mannington coal, Williams mine, Hopkins County, Ky.
5	S. 1.33, F. 1.50	2.6	13.2	23.7	60.5	-	
6	S. 1.50 .....	1.3	6.7	16.5	67.7	7.8	
7	F. 1.27, S. 1.27, F. 1.35 .....	26.9	54.9	16.0	2.2	-	Rosebud coal, Rosebud mine, Rosebud County, Mont.
8	S. 1.35, F. 1.50	3.3	21.2	50.9	24.6	-	
9	S. 1.50 .....	1.0	4.2	61.8	18.5	14.5	
10	F. 1.25, S. 1.25, F. 1.34 .....	33.2	59.0	6.4	1.4	-	Middle Kittanning coal, Castner mine, Jefferson County, Ohio
11	S. 1.34, F. 1.50	15.9	52.8	11.2	20.1	-	
12	S. 1.50 .....	10.3	24.1	21.3	38.6	5.7	
13	F. 1.25, S. 1.25, F. 1.29 .....	31.6	66.0	2.4	-	-	Sunnyside coal, Sunnyside No. 1 mine, Carbon County, Utah
14	S. 1.29, F. 1.50	7.3	51.1	39.7	1.9	-	
15	S. 1.50 .....	.6	8.4	57.5	16.9	16.6	
16	F. 1.25, S. 1.25, F. 1.31 .....	25.0	53.5	18.5	3.0	-	No. 5 Block coal, Colcord No. 5 mine, Raleigh County, W. Va.
17	S. 1.31, F. 1.50	5.5	28.9	42.2	23.4	-	
18	S. 1.50 .....	.8	5.3	75.3	17.1	1.5	
19	F. 1.29, S. 1.29, F. 1.31 .....	31.5	57.7	6.5	4.3	-	Monarch coal, Sheridan mine, Sheridan County, Wyo.
20	S. 1.31, F. 1.50	19.3	32.6	17.5	30.6	-	
21	S. 1.50 .....	3.7	6.7	18.4	64.5	6.7	
22	F. 1.29, S. 1.29, F. 1.31 .....	32.8	64.5	2.5	.2	-	Mammoth coal, Elkol mine, Lincoln County, Wyo.
23	S. 1.31, F. 1.37	24.7	63.9	9.3	2.1	-	
24	S. 1.37 .....		4.5	10.4	3.4	81.7	

Thin sections of the Beaver Hill coal bed in the Coos Bay coal field were examined microscopically and some significant petrographic characteristics were found.<sup>16/</sup> About 30 percent of the coal consisted of anthraxylon bands wide enough to be distinguished with the unaided eye. Fusain was not present, and total opaque matter in no instance exceeded 1 percent of the coal. Clay minerals and pyrite, finely dispersed in the coal, were the principal ash-forming ingredients recognized with the microscope.

<sup>16/</sup> Toenges, Albert L., Dowd, James J., Turnbull, Louis A., Schopf, J. M., Cooper, H. M., Abernethy, R. F., Yancey, H. F., and Geer, M. R., *Minable Reserves, Petrography, Chemical Characteristics, and Washability Tests of Coal Occurring in the Coos Bay Coal Field, Coos County, Oreg.*: Bureau of Mines Tech. Paper 707, 1948, 56 pp.



Heating-Value Loss During Air Drying of Coal

The standard method for analyzing coal specifies that all wet coal be air-dried before final preparation of the laboratory sample. As there has been some criticism of the possible loss of heating value from excessive air drying, a series of tests was made of coals of several ranks to evaluate the degree of oxidation, or loss of heating value, caused by air-drying according to standard practice. The results of the investigation<sup>17/</sup> showed that:

The percentage of heating-value loss depends on the rank of coal, since changes in low-volatile bituminous coals were not detected, and high-volatile C bituminous and subbituminous B coals were oxidized slightly, with resulting slight loss in heating value;

The heating-value loss due to 1 or 2 days' drying beyond the time when the air-dry loss is less than 0.1 percent per hour, is negligible;

The heating-value determination of low-rank coals should be made as quickly as possible after collection of samples.

Determination of Moisture in Coal

A series of tests was made to compare the percentages of moisture in coal obtained by the A. S. T. M. standard procedure with those using the Brabender Semi-automatic Moisture Tester, an instrument that has been used to determine rapidly the moisture content of textiles, cellulose, feeds, foodstuffs, etc. The results<sup>18/</sup> showed that the moisture values for coal obtained with the Brabender apparatus are within the permissible tolerances of those obtained by the A. S. T. M. procedure, except for anthracite. The drying rates by the A. S. T. M. and Brabender procedures were similar, and it was found that the drying time for high-rank coals could be reduced for both procedures.

Float-and-Sink Testing of Lignite

Since float-and-sink tests applied to bituminous coals give erratic results on air-dried lignite, an investigation was made of the effect of moisture in the float-and-sink testing of lignite. Tests<sup>19/</sup> made on North Dakota lignite showed that (1) the yield of float material in a float-and-sink test is greatly influenced by the moisture content as tested; (2) air-dried lignite differs from bituminous coal in that it cannot be restored to its original state by saturating with water; (3) saturating air-dried lignite with a gasoline-carbon tetrachloride mixture has no effect on the results; (4) lignite coarser than 14-mesh can be tested in organic solutions without air-drying; and (5) order in which a sample is tested through a series of solutions is without effect on the results.

<sup>17/</sup> Cooper, H. M., Tarpley, E. C., and Abernethy, R. F., Heating-Value Loss of Coal on Air-Drying: Bureau of Mines Rept. of Investigations 4197, 1948, 6 pp.

<sup>18/</sup> Cooper, H. M., Tarpley, E. C., and Abernethy, R. F., Determination of Moisture in Coal with the Brabender Semiautomatic Tester: Bureau of Mines Rept. of Investigations 4304, 1948, 15 pp.

<sup>19/</sup> Cooper, H. M., Tarpley, E. C., and Abernethy, R. F., Effects of Moisture on the Float-and-Sink Testing of Lignite: Bureau of Mines Rept. of Investigations 4184, 1947, 8 pp.