

The increased efficiency obtained by igniting the mixture at the bottom of the cavity over that resulting from ignition at other points is exactly what would be expected, and the increase might have been greater, if the box could have been made more rigid at the bottom.

It will also be observed that the efficiency resulting from the use of an inside cover was about ten times that from an outside one. The increase varied considerably with the perfection of fit of the cover and also with the load, heavier loads showing increased efficiency. Unfortunately all this power could not usually be measured, as an excessive load almost invariably resulted in the rupture of the box and consequent loss of energy.

To sum up, therefore, it appears from the foregoing results that, under ordinary conditions of explosion and covering, such as might occur in practice, an efficiency of about one one-hundredth of one per cent. is all that could be expected, that this efficiency increases with the excellence of the fit of the covering, with the load on the cover, to be raised, and also seems to increase if the point of ignition be near the bottom rather than near the top of the cavity. With an inside cover which has to be raised through a distance equal to the thickness of the cover, before vent is given to the box, the efficiency is about ten times as great.

The results obtained in the foregoing paper agree well with the experience which one of us, (A. H. G.) and also a colleague, has had in a series of experiments independently carried out on the large scale. Some fifty explosions were made, using boxes buried in the earth and also with covered pits from 80 to 7000 cubic feet capacity.

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## A STUDY OF THE LIGNITES OF THE NORTHWEST. PARTS I. MOISTURE AND HYGROSCOPICITY.

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As a result of an investigation of the lignites from the west, northwest and north, the writer has reached the conclusion from experience extending over several years that in many respects they differ from bituminous coal to such an extent as to call for somewhat different methods of analysis and different interpretation of results. This seems to be largely due to the difference in composition and more especially to the large amount of moisture present. It has been found from analyses of a large number of samples ranging from the poorest to the best lignites of the country and from samples fresh from the mines to those exposed to the atmosphere for several months, that the moisture may vary from 6 to 40 per cent. and in a single instance 44.2 per cent. This variation is due partly to the condition under which the lignite exists in the seam and

partly to the nature of the lignite itself. For instance, the brown lignite of North Dakota contains more moisture than the dark lignite farther west and north. The writer has never found the freshly mined lignite of Dakota to run below 30 per cent. of moisture. A sample direct from the Klondike<sup>1</sup> region gave only 22 per cent.

Freshly mined lignite loses moisture so rapidly that it is with the greatest difficulty that accurate determinations are made. As will be seen from data which follow, loss of moisture is so very rapid that as high as ten per cent. is frequently lost in the process of preparing the sample for analysis, unless precautions are taken. This fact is mentioned by the Committee on Coal Analyses<sup>2</sup> although freshly mined samples of lignite were probably not examined<sup>3</sup>.

Loss on exposure to the air continues until an equilibrium is reached. Thoroughly dried lignite is very hygroscopic, absorbing moisture rapidly until an equilibrium is reached. This hygroscopicity is likewise characteristic of the lignites, for the increase in weight of thoroughly dried coal seems to be entirely due to water, there being only slight indications of the oxidation that is frequently observed in bituminous coals, either by drying at 104° to 107° or on standing exposed to the air after drying.

As a practical illustration of the rapid loss of moisture, a sample of lignite from Dakota left the mine, containing 39.3 per cent. of moisture. On arriving at its destination several days later, it contained 28 per cent., a loss of 11.3 per cent. during transportation. This loss of moisture continues more or less rapidly, depending upon the fineness of the coal, until from 10 to 12 per cent. is reached. At this point, it ceases to lose more unless the coal is badly "slaked," in which case the per cent. of moisture may drop to between 5 and 9 per cent. depending upon the fineness and the nature of the lignite and upon atmospheric conditions.

*The Rate of Loss of Moisture on Exposure to the Air.*—While the rapidity of the drying of coal is mentioned in the Report of the Committee on Coal Analysis and in the U. S. Geological Survey Reports and the importance of limited time taken in preparing samples for analysis emphasized, detailed results concerning the lignites are at the present time not complete. In this paper I have recorded some of the peculiar characteristics of the lignites, incidentally emphasizing the fact that modified methods of analysis are necessary in order to obtain the best results.

In order to compare results obtained by using different kinds of recep-

<sup>1</sup> The sample was thirty-four days in transportation but was packed in a close-fitting box.

<sup>2</sup> This Journal 22, 1119.

<sup>3</sup> Since this paper was written, a Report of the Coal Testing Plant of the U. S. Geological Survey Professional Paper No. 48 has been received. In this report Dr. Hillebrand gives a mass of facts along the same lines covered in this paper.

tacles, some of these determinations were made in ground stoppered weighing bottles 50 mm. high and 25 mm. in diameter, some in regulation size crucibles and some in flat capsules. In each case the temperature and relative humidity were noted and the time taken for pulverizing was exact to the minute.

Samples of North Dakota lignite direct from the mine were pulverized requiring different lengths of time for the process, and the moisture determined in weighing bottles under exactly the same conditions. Samples in each case were put through a No. 100 sieve. The following per cents. were obtained :

Time in pulverizing . . . . .	1 minute	30 minutes	1 hr.	2 hrs.
Moisture . . . . .	37.61	34.22	32.43	31.42

The loss of moisture was greater during the first half hour pulverizing, than during the next hour and a half. That more data might be obtained, many determinations were made by exposing coal to the air for short periods of time and determining accurately the rate of loss. Experiments showed that for about ten minutes, the rate of loss per minute is practically constant ; after that time the rate diminishes, in some samples slowly and in others quite rapidly. An examination into this fact showed that the rate of loss of moisture in lignite when exposed to air depends not upon the time of exposure alone but upon the physical conditions of the coal, whether pulverized or in lump ; upon the temperature of the air, the relative humidity and upon the form of receptacles in which the coal is exposed and upon the amount of sample taken. The rate of loss was found to be greater in crucibles than in ordinary weighing bottles and greater in flat capsules than in crucibles. Samples of 100-mesh coal containing 31.17 per cent. of moisture were exposed to the atmosphere in a capsule 30 mm. in diameter and 5 mm. deep, in porcelain crucibles and in glass stoppered weighing bottles. The temperature was 25° and the relative humidity 29. The receptacles were arranged so that the sample could be protected from the air while weighing. In this, as in other experiments, as near two grams as possible was taken.

The following results were obtained :

Loss after	1 minute exposure	Bottle	Crucible	Capsule	per cent.
“ “	2 “	0.13	0.16	0.196	“ “
“ “	3 “	0.26	0.34	0.364	“ “
“ “	4 “	0.39	0.48	0.533	“ “
“ “	5 “	0.43	0.65	0.701	“ “
“ “	6 “	0.57	0.78	0.860	“ “
“ “	7 “	0.69	0.94	1.020	“ “
“ “	8 “	0.91	1.10	1.190	“ “
“ “	9 “	1.02	1.24	1.370	“ “
“ “	10 “	1.15	1.38	1.568	“ “
“ “	20 “	1.27	1.56	1.740	“ “
“ “	30 “	2.00	2.53	2.81	“ “
“ “	30 “	2.72	3.69	3.84	“ “

It will be seen that the rates of loss in these cases were very nearly constant for the first ten minutes. The loss was slightly less during the next ten minutes, gradually decreasing with time of exposure as indicated in the above tables. The variation in the results with the crucible is unquestionably due to the fact that it could not be closed perfectly during the weighing as in the case of the weighing bottles and capsules. I am, therefore, inclined to recommend weighing bottles in preference to crucibles for the accurate determination of moisture.

It is evident from the above data that widely varying results may be obtained from the same sample of lignite by varying the time of pulverizing and exposure, preparatory to analysis. In fact, even the preserving in stoppered bottles does not entirely prevent the loss of moisture. As an illustration of this fact, a sample of about one pound of lignite containing 39 per cent. of moisture was placed in a large bottle with a glass stopper and allowed to stand for nine weeks. At the end of that time an analysis showed only 31 per cent. of moisture.<sup>1</sup>

Many samples of different quality and from different localities have been examined. The following samples, two from Alaska mines and two from different parts of the Dakota fields have been selected as representing different grades of lignites :

No. 1. Beaver Hill, Yukon Valley, containing 11.77 per cent. moisture. Fresh from the mine, sample contained 26.8.

No. 2. Klondike, Yukon Valley, containing 10.47 per cent. moisture. Fresh from mine, sample contained 25.1.

No. 3. Kenmare, North Dakota, six weeks in air, 11.83 per cent. moisture. Fresh from mine, sample contained 37.6.

No. 4. Wilton, North Dakota, exposed for a few days, 31.16 per cent. moisture. Fresh from mine, sample contained 38.20.

The samples were dried in baths arranged with thermometers and a thermostat so that a constant temperature could be maintained. Toluene and xylene baths were likewise used in some of these experiments. Special air and hydrogen baths were constructed with asbestos and glass walls and a thermostat, so that a temperature varying within one degree could be maintained.

*Loss by Drying Above 107°.*—From a great number of determinations it was found that while lignite loses moisture very rapidly, a temperature of 104°-7° is apparently not sufficient to remove all of it. In the following tables the four above mentioned samples were taken. Just five minutes in each case were required to pulverize and pass through a 20-mesh sieve. Exactly 15 minutes more were taken in putting parts of the

<sup>1</sup> The Com. on Coal Analysis recommended a sealed fruit jar. A ground glass stopper is never air tight unless lubricated. Editor.

same samples through a 100-mesh sieve. These samples were dried to constant weight under exactly the same conditions.

Dried 2 hrs. at

	No. 1 Fineness		No. 2 Fineness		No. 3 Fineness		No. 4 Fineness	
	No. 20	No. 100	No. 20	No. 100	No. 20	No. 100	No. 20	No. 100
104-7°	10.94	10.74	10.54	9.41	11.34	10.61	30.67	30.17
112-14	11.91	11.77	11.59	10.43	12.20	11.82	31.16	31.17
125-6	12.10	11.76	11.70	10.51	12.53	11.83	31.86	31.17
135-6	12.12	11.76	11.76	10.48	12.50	11.83	31.87	31.26
loss above 107	1.18	1.02	1.22	1.09	1.16	1.22	1.20	1.11

I have dried two hours instead of one. In only one or two cases has the second hour's drying made any difference. In each case I have dried one hour and weighed, then dried a second hour and reweighed. The per cent. of moisture in the 20-mesh was greater than in the 100-mesh samples. These results may be explained by the difference in the length of time required in pulverizing. The 100-mesh samples were exposed 15 minutes longer to the air than the 20-mesh samples. Furthermore, the 100-mesh samples lose moisture more rapidly than do the 20-mesh samples.<sup>1</sup>

A distinct loss in weight occurred above 107°. Over one per cent. of loss resulted in the raising of the temperature to 135°. Most of the loss above 107°, however, occurred below 114, although a temperature of about 135° seemed necessary to obtain constant results. The question then arose as to what this loss represented, whether due to moisture or to some other cause.

Determinations were therefore made on the above samples by heating at a temperature of 135° in an atmosphere of hydrogen, and collecting whatever was driven off by the heat, in calcium chloride tubes. The loss in the weight of coal was compared with the gain of the weight of the calcium chloride tube. The apparatus finally adopted as giving best results consisted of an ordinary sized air bath with glass walls. The opposite glass ends were removed and replaced by asbestos cards. Holes were made in the asbestos cards and a combustion tube about 18 inches long inserted. One end of the tube was connected with a hydrogen generator arranged with proper apparatus for purifying and drying the gas. The other end was connected with a weighed calcium chloride tube. The weighed coal was placed in a boat and inserted in the tube. A slow but constant stream of hydrogen was passed through the tube. The bath was then heated to the desired temperature :

The following results were obtained with this apparatus :

	No. 1	No. 2	No. 3	No. 4
Loss by drying in				
air at 107° C	10.74	9.42	11.31	30.29
in hydrogen at 135°	11.82	10.69	12.23	31.29
Gain CaCl <sub>2</sub> Tube	11.80	10.75	12.34	31.35

<sup>1</sup> See Report of Coal Testing Plant at St. Louis.

These results indicate that the loss resulting from drying above  $107^{\circ}$  is moisture. There were no indications that anything but water had been absorbed by the calcium chloride tube.

*Drying in Desiccators.*—As stated in the report of the Committee on Coal Analysis<sup>1</sup> moisture may be determined in coal when the amount is less than two per cent., by drying in desiccators over sulphuric acid. When coal contains more than two per cent. the method is not satisfactory. In order to test the method on lignites, a number of determinations were made. These verify the statement of the Committee. Samples of coal containing a known per cent. of moisture were dried for five days in a desiccator over sulphuric acid.

The following table will show results obtained :

Loss after 1 day.....	28.76	28.22	10.01
“ “ 2 “ .....	32.69	29.01	10.21
“ “ 3 “ .....	33.12	29.42	10.46
“ “ 4 “ .....	33.29	29.66	10.60
“ “ 5 “ .....	33.37	30.23	10 65
Total moisture in sample .....	34.81	31.65	11.60
Difference.....	1.44	1.32	.99

It is quite evident that drying in a desiccator over sulphuric acid is not sufficient to remove all the moisture from lignite.

*Drying in Vacuum Desiccator.*—Determinations of moisture were made in vacuum desiccators over sulphuric acid and under a diminished pressure of 10 mm. After practically constant results were obtained, samples were dried in an air bath at  $107^{\circ}$  and  $125^{\circ}$ , respectively.

The following table gives the loss in moisture :

	No. 1	No. 2	No. 3	No. 4
In vacuo after 1 day .....	10.60	10.07	11.09	30.64
2 .....	11.01	10.34	11.51	30.84
3 .....	11.10	10.28	11.38	30.79
4 .....	11.10	10.31	11.51	30.84
7 .....	11.29	10.52	11.51	30.79
Maximum .....	11.29	10.52	11.51	30.84
These samples were now placed in an air bath and dried.				
Dried at $107^{\circ}$ .....	10.77	9.81	11.10	30.59
125°.....	12.10	11.10	12.39	31.41
Difference between $107^{\circ}$ and 125° C.....	1.33	1.26	1.29	0.82
Difference between vacuum and 125° C.....	0.81	0.58	0.88	0.57

In these determinations the same weighing bottles and the same samples were dried *in vacuo* and in air. All the vacuum dried samples gained materially on drying in an air bath at  $107^{\circ}$  and lost by drying at  $125^{\circ}$ . The difference between the vacuum dried coal and that dried at  $125^{\circ}$  is between 0.5 and 0.8 per cent., while the difference between the

<sup>1</sup> This Journal, 21, 1121.

coal at  $106^{\circ}$  and  $125^{\circ}$  is about one per cent., about the same as that obtained by drying the coal directly in an air-bath at  $107^{\circ}$  and  $125^{\circ}\text{C}$ .

In order to determine whether or not the loss and gain in the above tables were due to moisture or to oxidation, the following experiments showing loss by weight and extending over a period of eighteen months were carried out :

		No. 1	No. 2	No. 3	No. 4
Dried in vacuo over $\text{H}_2\text{SO}_4$	2 days	8.13	9.4	10.34	29.01
" " " " "	10 "	8.74	10.07	11.21	29.59
" " " " "	30 "	8.86	10.10	11.32	29.82
Dried in Hydrogen at $107^{\circ}$	2 hrs.	8.65	9.77	11.05	29.56
" " Air " "	" "	7.91	9.08	10.25	28.26
" again in Hydrogen at $107^{\circ}$	" "	8.67	9.71	11.11	29.22
" 1 year in desiccator over $\text{CaCl}_2$		7.01	8.05	9.25	27.28
" in vacuo over $\text{H}_2\text{SO}_4$	6 days	8.70	9.62	11.23	29.24
" in Hydrogen in Toluene bath	1 hr.	8.15	9.20	10.70	28.68
" " " " " "	5 "	8.43	9.41	10.90	29.02
" " vacuo over $\text{H}_2\text{SO}_4$	12 "	8.64	9.62	11.09	29.23
" " " " "	10 days	8.78	9.83	11.43	29.45

The maximum loss seems to have been reached after drying 30 days *in vacuo* over sulphuric acid. Drying two days longer failed to show further loss. It was expected from past experience that further loss would take place by drying at  $107^{\circ}$  in an atmosphere of hydrogen. On the contrary there was a very slight gain after drying two hours at  $107^{\circ}$ . Great care was taken in drying the hydrogen and the only explanation which seems plausible is the short exposure to the air while transferring to the hydrogen bath and while the air was being driven out of the drying oven. The increase in weight by drying the air at  $107^{\circ}$  is of special interest. In each sample the gain in weight was very marked, the samples from the extreme north gaining about one per cent., while those from the Dakota field gained about one and one-half per cent. From the peculiar hygroscopic nature of dried lignite, this gain in weight was attributed largely if not wholly, to absorption of moisture, and hence if dried again under exactly the same conditions the loss of weight ought to be the same as the previous loss. This is practically true of the first three samples. There was a slight loss in sample No. 4 by drying the second time in hydrogen.

These samples then stood one year in a desiccator over calcium chloride. The desiccator stood in a dark place and was not opened during the year. The gain in each case was very marked, being about two per cent. in each sample. Drying *in vacuo* over sulphuric acid for six hours failed to bring the loss quite back to that previously obtained by drying under the same conditions. However, after repeated drying in a hydrogen toluene bath and again drying *in vacuo* over sulphuric acid, the loss was practically the same as that obtained in the beginning of the ex-

periments in two of the samples. In the other two there was a slight increase in weight.

From these and numerous other experiments it would appear that no oxidation takes place in the drying of the lignite coal, but that the great variation observed in drying the lignites is due to hygroscopicity.

*Hygroscopicity.*—As already stated, lignite loses moisture when exposed to the air. When the sample is freshly mined the loss is so rapid as to make weighing extremely difficult in an open vessel. If exposed to the air for a long time, a point is finally reached at which the moisture may increase or decrease slightly, depending upon atmospheric conditions. If the lignite is pulverized, the loss of moisture goes on rapidly until an equilibrium is reached. The time required is about 5 days, although most of the moisture is lost during the first 2 days. Finely pulverized lignite exposed in open capsules will reach an equilibrium frequently in 24 hours. Four samples were placed in the weighing bottles, already mentioned, and weighed every day until the weights were constant.

The following table gives the results obtained :

Sample	No. 1 Beaver Hill	No. 2 Klondike	No. 3 Kenmare	No. 4 Wilton
Total moisture in sample.....	11.41	10.54	11.38	31.56
Per cent. of moisture lost by drying in air				
1 day.....	1.00	0.91	0.85	8.62
2 " .....	2.62	2.23	2.48	19.59
3 " .....	2.72	2.27	2.44	21.59
4 " .....	3.15	2.63	2.94	23.10
5 " .....	3.51	2.98	3.32	23.82
6 " .....	3.51	2.94	3.36	23.97
7 " .....	3.60	2.81	3.34	24.22
8 " .....	3.30	2.64	3.34	24.07
9 " .....	3.38	2.60	3.30	24.00
10 " .....	3.28	2.62	3.32	24.06
Maximum loss.....	3.89	3.22	4.01	24.34
Moisture in Coal after equilibrium is reached	7.52	7.32	7.50	7.22

These experiments were continued for 100 days, weighing every two days. During that time the loss never became greater than the above maximum, and never dropped more than six tenths of one per cent. below this maximum.

The above equilibria are obtained by subtracting the maximum loss of moisture from the total moisture present in the coal.

After this point of equilibrium is reached, further exposure to the air may change the equilibrium slightly, depending, however, upon the relative humidity of the air. In all these determinations the temperature and the relative humidity of the air were taken before each weighing. After the equilibrium had once been established, we were able to predict an increase or loss by weight from the temperature and relative humidity of the air. In most of these determinations the temperature was kept as



