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THE ANALYSIS OF COAL WITH PHENOL AS A SOLVENT

BY

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AND

H. F. HADLEY



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UNIVERSITY OF ILLINOIS
ENGINEERING EXPERIMENT STATION

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NOVEMBER, 1914

THE ANALYSIS OF COAL WITH PHENOL AS A SOLVENT
(WITH A STUDY OF THE RESULTING COMPOUNDS)

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THE ANALYSIS OF COAL WITH PHENOL AS A SOLVENT

I. INTRODUCTION.

1. *Present Methods of Coal Analysis.*—There are two processes in vogue at the present time for the chemical examination of coal; one is the ultimate, and the other is the proximate method of analysis. In the first the organic or combustible part of the coal is separated into its elemental constituents, carbon, hydrogen, oxygen, and nitrogen. The mineral or non-combustible portion is separately determined under two items as ash and moisture.

In the proximate method the organic material is separated into two divisions, one being that portion which under high temperature and out of contact with the air passes off in the gaseous form, and the other that part which remains behind as the non-volatile or coke-forming carbon.

Each procedure has doubtless come into use as the result of a specific demand. For example, the engineer needed the data from which he could calculate the total heat of the coal and, in arriving at a heat balance, he must also have at hand any negative factors chargeable to the fuel, such as the quantity and character of the gaseous products of combustion. These items, therefore, would call for the data furnished by the ultimate methods of analysis.

The proximate method was developed as a natural accompaniment of the gas and coke industries, since it furnished in either case an index of the yield which might be expected from a given coal. Formerly, also, the quantity of volatile matter was made to serve as an index of the grade or quality of a coal. Thus the data from proximate analyses have been put into the form of "fuel ratios" or the ratio of the non-volatile to the volatile part of the coal, such ratios supposedly serving as an indication of the general class or type to which the coal belonged.

It is evident from this brief statement that the results obtained from ordinary methods of analysis are of rather narrow application and that the data from neither the ultimate nor the proximate methods

convey much information which can be applied to the every-day problems connected with the handling and use of coal such as coking, storage, deterioration, oxidation, weathering, and spontaneous combustion.

It is to be noted in this connection that the ordinary methods of analysis give the percentage amounts of certain elements or compounds which result from destructive processes and do not even remotely take account of the actual constituents of the organic or active portions of the coal as they occur in unaltered form. But it is these unaltered constituents which have to do with such matters as coal storage and spontaneous combustion. Especially do they have to do with coking properties, with smoke formation, and the type of distillation products which they may be made to yield.

2. *Purpose of the Proposed Study.*—The purpose of this investigation has been to examine and enlarge upon certain processes of analysis which proceed in such a manner as to separate in unaltered form the fundamental substances of which coal is composed, and to study their characteristic properties in detail.

Efforts along this line have multiplied in recent years. Much suggestive information has come from the study of the types of vegetable matter which constituted the source of the initial coal substance. The microscopical studies of White,¹ of the United States Geological Survey, and Professor Jeffrey,² of Harvard, have developed the general proposition that the original organic constituents would fall into two general classes: those of the cellulose and those of the resinic type. A similar line of evidence has been developed by numerous investigators, who have made use of various solvents, which, in the main, have differentiated the compounds along the same general lines. The work of Frazer and Hoffman,³ Clark and Wheeler,⁴ and Professor Lewes⁵ may be cited in this connection.

In conformity, therefore, to this general proposition as to the coal constituents this investigation proceeded upon the general hypothesis that in coal two types of compounds are to be met with: (1) the degradation products of the original cellulose substances, and (2) the less altered and perhaps more characteristic compounds of the resinic type. The first line of investigation, therefore, from the chemical standpoint, would seem to consist of a study of various solvents which might lend themselves to such a separation into the two main subdi-

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1. Professional Paper 85-E, United States Geological Survey (1914).
 2. Proc. Am. Acad. of Arts and Sciences, 46, 273 (1910).
 3. Technical Paper 5, Bureau of Mines (1912).
 4. Journal of Chemical Society, 103, 1704 (1913).
 5. Progressive Age, 29, 1030 (1911).

visions of the coal constituent, and, second, a study of the general properties of each branch.

3. *Outline of the Investigation.*—(1). Various extractive agents were used to determine the extent or completeness of their solvent properties. Solvents were selected which would seem to have the greatest ability to remove those bodies which were of the resinic type. The substances tried are arranged in the order of their solvent properties in so far as that would seem to be indicated in preliminary studies directly concerned with the extent of the solvent action of each. The list is as follows: phenol, ortho-cresol, low boiling tar distillate, para-cresol, pyridine, phenol-toluene mixture, aniline, methyl aniline, acetone, toluene, benzene, carbon disulphide, and turpentine. While some of the above solvents might warrant further study, there are evident advantages over the other compounds in the use of phenol and it was made the subject of extended study.

(2). A general survey of the various types of coal was made to determine the relative amounts of phenol-soluble extract contained in each. The average amount of material extracted from Pocahontas coal was 1.8 per cent and the results varied in the case of Illinois coals from 22 per cent to 39 per cent. These results were calculated on an ash and moisture free basis.

(3). By effecting a separation as indicated under (1), it was possible to study separately the insoluble residue and the extracted material, to determine which had the greater avidity for oxygen, also to which division should be credited the coking constituents, and what products of decomposition, such as gas, illuminants, tar, etc., could be assigned to each.

4. *Summary of Results.*—1. Phenol at 100°C. will dissolve certain constituents of bituminous coals in their natural state. The two subdivisions, designated as *insoluble residue* and *extraction material*, together make substantially 100 per cent or the amount of the original substance.

2. The amount of extractive material is definite and susceptible of quantitative determination.

3. Studies upon these two type substances indicate that the extract is the vital constituent concerned in the coking of coal. It conforms to the principle already enumerated¹ in that it has a sufficiently definite melting point and a decomposition temperature which is above that of the melting point.

4. Each subdivision is capable of absorbing oxygen. The effect upon the extract is to modify its coking properties by lowering or

1. Bulletin No. 60, U. of Ill. Eng. Exp. Sta., p. 20.

greatly reducing its power to form a firm and coherent mass.

5. The oxygen taken up in either case is found to be chemically combined. The oxygen taken up by the fresh coal is similarly held. The insoluble portion has the greater avidity for oxygen. This relation to oxygen has a bearing upon other topics aside from that of coking, as the heating of coal in storage, spontaneous combustion, weathering, and deterioration.

II. EXPERIMENTAL.

A. APPARATUS AND METHOD OF PROCEDURE.

5. *Preliminary.*—In the preliminary part of the work, conducted to determine which solvent would be the best suited to the purpose, the adaptability of the solvent was determined by the amount of material which it would remove, and also, by the absence of chemical activity involved in its use. That is to say, the action must be that of a true solvent and must not develop chemical changes either in its own structure or that of the components of the coal. These features could be most readily indicated by noting (1) the amount of material dissolved, and (2) the sum of the extract and residue; this sum should correspond substantially to the weight of the original material, provided no chemical action had taken place. The refinements in analytical methods in the preliminary work were not developed beyond the point necessary to determine the applicability of the extractive agent to the work in hand. The solvents used are given in the order of their effectiveness, the most active being placed first: phenol, ortho-cresol, low boiling tar distillate, para-cresol, pyridine, phenol-toluene mixture, aniline, methyl aniline, acetone, toluene, benzene, carbon disulphide, and turpentine.

Para-cresol, ortho-cresol, and low boiling tar distillate decomposed during the extraction, thus apparently increasing the amount of extracted material. Ortho-cresol gave but little decomposition product and as a solvent it ranked next to phenol. Methyl aniline and aniline presumably underwent decomposition.¹ Pyridine and phenol-toluene mixture gave a certain amount of extract but it was not as large as that obtained by use of phenol alone. Furthermore, the increase in the amount of nitrogen which is found in both the extract and residue after treatment with pyridine must be taken as an index of the formation of substitution products not originally present in the coal substances.² Acetone, benzene, carbon disulphide and turpentine extracted only small amounts of material and were therefore discarded.

1. Lewes, *Progressive Age*, 29, 1033.

2. Lewes, *Progressive Age*, 29, 1033.

Clark and Wheeler, *Journal Chem. Soc.*, 103, 1709.

6. *Phenol and the Conditions which Influence the Amount and Character of Extract.*—As a result of the preliminary work, it was evident that phenol was the chemical best suited for use as an extractive agent. A study, therefore, of the ultimate requirements necessary in its use, was the next step to be undertaken. The fundamental conditions to be observed were found to be (a) time for the solvent act, (b) temperature, (c) presence or absence of air in any part of the process. The effect of these various conditions may be shown as follows:

(a) Time of Extraction.

In order to determine the amount of material extracted for various lengths of time, five grams of coal were treated for successive periods of five hours each, and the amount of extract determined. At the end of twenty hours the extraction was discontinued and the weight of the extract determined. The results of the extraction were as follows:

TABLE 1.

WEIGHT OF MATERIAL DISSOLVED FROM COAL USING PHENOL FOR FIVE HOUR PERIODS.

Weight of the extract after 1st five hours.....	1.188 grams
“ “ “ “ “ 2nd “ “110 “
“ “ “ “ “ 3rd “ “040 “
“ “ “ “ “ 4th “ “027 “

It will be seen from Table 1 that during the last five hours of extraction only .027 gram of material was obtained. To determine whether this last increment was true extract or decomposition products of the solvent, the phenol was distilled alone for ten hours as a blank without any coal in the extraction tube, and the phenol distilled off at the end of that time. The weight of the residue in the flask was .03 gram. This weight of decomposition product obtained after ten hours of distillation would represent an equivalent of .015 gram for five hours of distillation. When we consider that the amount of apparent extract during the last five hours was only .027 gram, if .015 gram of this were derived from the phenol, then only .012 gram would be the weight of the substance actually dissolved during that period. It was decided, therefore, to stop the extraction at the end of twenty hours and this length of time for the extraction process was used in all of the work.

(b) Temperature of the extraction.

In the earlier part of the work it was found that there was a

larger amount of material extracted from the coal if the solvent were applied hot, as at 110°C,¹ instead of at 20° to 25°. The use of the higher temperature was accomplished by condensing the phenol at the temperature of boiling toluene, i. e., 110°. By causing the condensate at this temperature to flow upon the coal, which in turn was held in the vapors of the phenol, the temperature of the solvent was thereby always kept above 110°. The weight of extract and residue from two coals, when extracted with phenol at the two different temperatures, is given in the following table:

TABLE 2.
WEIGHT OF EXTRACT AND RESIDUE FROM COAL EXTRACTED
AT DIFFERENT TEMPERATURES.

Coal	Divisions	Extracted at 25° C.	Extracted at 110° C.	Difference
A	Extract	1.564	1.691	+ 0.127 = + 2.56%
	Residue	3.543	3.451	- .092 = - 1.84%
B	Extract	1.326	1.458	+ 0.132 = + 2.64%
	Residue	3.788	3.674	- 0.114 = - 2.28%

It will be noticed that with both coals the weight of residue decreased and that the weight of extract increased when the extraction was carried out at the higher temperature. The discrepancies are to be referred to the decomposition resulting from the continuous boiling of the phenol. This topic is discussed more fully later. Although the additional amount of extract was not large, it was thought advisable to carry out the extraction at the higher temperature even though the apparatus which was necessary for doing it was slightly complicated.

(c) Presence of Air in the Extraction Apparatus.

In some of the preliminary experiments it was found that the weight of extract and residue combined was equal to more than the original weight of the coal. This would doubtless be due to the oxidation of some of the substances involved, i. e., extract, residue, or phenol. The extraction was, therefore, carried out in the absence of air, carbon dioxide being conducted through the apparatus. The following results show very plainly that there was an increase of weight in both the extract and residue when the distillation was carried out in the presence of air. The test was made on five grams of two different coals. These results are given in Table 3.

1. All temperature readings in this discussion are Centigrade.

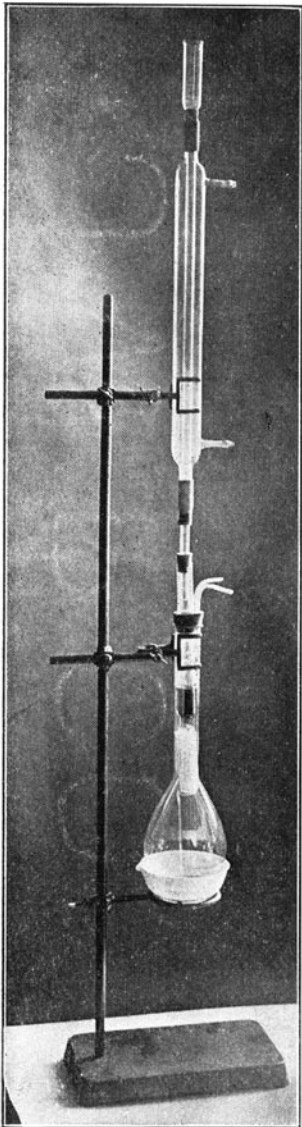


FIG. 1.
EXTRACTION APPARATUS
READY FOR OPERATING

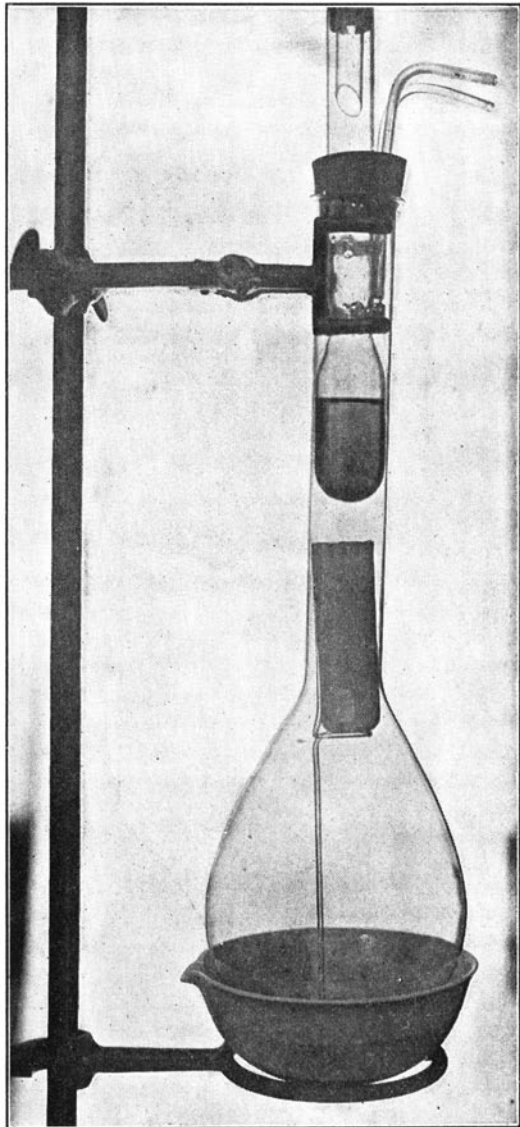


FIG. 2.
CONDENSER DEVICE FOR INCREASING THE TEMPERATURE OF THE SOLVENT

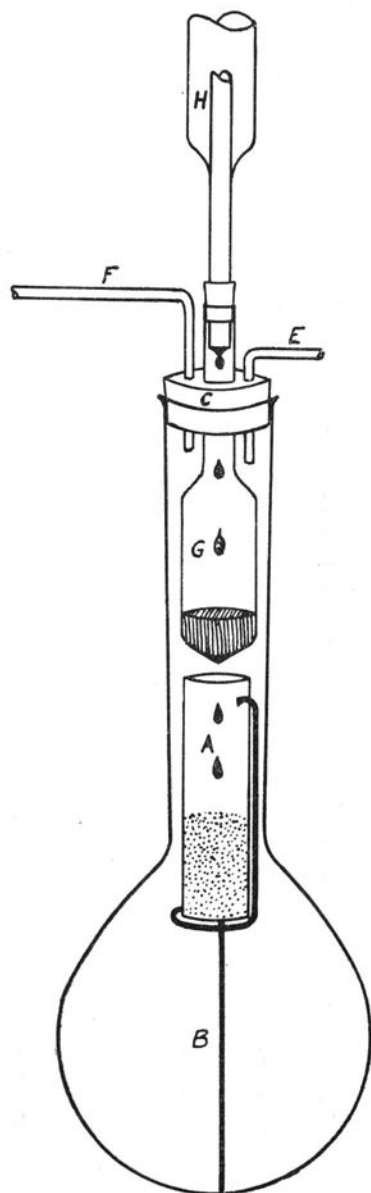


FIG. 3.
CONTINUOUS EXTRACTION
APPARATUS USING PHENOL AT 110°.

TABLE 3.
COAL EXTRACTED WITH PHENOL IN THE PRESENCE OF AIR AND
CARBON DIOXIDE.

Coal	Residual Substance	In CO ₂	In Air	Increase
A	Extract	1.564	1.614	.050 = 1.00 %
	Residue	3.543	3.691	.148 = 2.96 %
	Total	5.107	5.305	.198 = 3.96 %
B	Extract	1.326	1.389	.063 = 1.26 %
	Residue	3.788	3.921	.133 = 2.66 %
	Total	5.114	5.310	.196 = 3.92 %

7. *Apparatus.*—From the preliminary work the apparatus shown in Figs. 1, 2, and 3 was decided upon as being the best adapted for carrying out the process. Figs. 1, 2, and 3 show different views of the apparatus. An extraction cone (A), 25 by 80 cm., was supported in the neck of a 750 cc. Kjeldahl flask by means of a bent glass rod (B). A No. 7 rubber stopper (C) which contained three holes was placed in the neck of the flask. Two of the holes were for small glass tubes (E and F) through which carbon dioxide could be conducted. The one large hole in the center was for the upper end of a glass bulb (G) which acted as a condenser for the phenol. This condenser-bulb was almost as large as the neck of the flask and was drawn out at the upper end to fit a water condenser. It was necessary that this bulb should be large enough to hold about 25 cc. of toluene. The toluene itself was condensed by an ordinary 70 cm. water condenser. A Woods' metal bath was used for boiling the phenol. The whole flask was protected from draughts by asbestos paper.

8. *Process of Extraction.*—One important problem which presented itself in the extraction of coal with phenol was the matter of freeing the residue and the extract from the solvent. Obviously, the extract and the residue must be treated differently because of the nature of each. In spite of the fact that all precautions as to oxidation, decomposition, etc., were observed during the extraction, it was found that the sum of the weight of extract and residue was larger than the original weight of coal. This fact seemed to indicate that phenol was being retained in either the extract or the residue by a physical or chemical union. If the phenol was being held chemically, it could not be used as a solvent. Therefore, it was necessary to determine whether such was the case or not.

After the extraction, the residue was washed first with alcohol and then with ether in a Soxhlet extractor. The residue was subsequently dried in the air for one hour at 100°. It was found that this method of freeing the residue from phenol could not be used for two

reasons; even after the washing process and drying at 100° traces of phenol remained in the residue, and there was also the possibility of oxidation of the finely divided residue during the heating. In order to remove the adhering phenol the method would seem to be that of heating to a temperature above the boiling point of the solvent, i. e., to approximately 200°. Moreover by heating the residue which had been treated as above described to 200° traces of phenol were shown to be given off. The possibility of heating to this temperature raises the questions (1) of oxidation and (2) of decomposition of the material. The first point could easily be guarded by use of an inert atmosphere such as carbon dioxide. In the presence of such an atmosphere, heating to 200° could be carried on without decomposition as is shown by the following experiments.

TABLE 4.
SHOWING THE EFFECT OF HEATING COAL SUBSTANCE TO 200°
IN AN ATMOSPHERE OF CO₂.

Residue	Weight of Residue	Time of Heating	Weight after Heating	Difference
A	4.125	3 hours	4.121	— .004
B	4.120	2 "	4.123	+ .003
C	4.200	1 "	4.200	.000
D	3.994	2 "	3.981	— .013

As a result of these experiments the following procedure was adopted. After being washed with ether, the residue was dried for a short time in the air to allow the ether to evaporate and was then transferred to a flask. It was kept at 200° for one hour with a slow stream of dry carbon dioxide passing through the apparatus. At the end of one hour the flask was allowed to cool and the carbon dioxide displaced by dry air. The flask was then weighed. Because of the very hygroscopic nature of the material, it was necessary to protect it from all possible sources of moisture during the drying process.

To free the extract from phenol the first method used was to distil at reduced pressure. After practically all of the phenol had been driven off under this condition the temperature was raised to 200° while a current of carbon dioxide was conducted through the flask. It was thought that by simply bringing the mass to that temperature all of the phenol would be driven off. However, it was soon found that this was not the case. The heating of the extract was then continued for one hour in a stream of carbon dioxide, in a manner

exactly similar to that used with the residue. It was found that this method of treatment gave a lower weight for the extract. Although phenol was given off during the heating, it was not known what effect heating to 200° would have upon the composition of the material. To test the matter, the extract after being dried at 200° for one hour was again heated for a certain length of time under the same conditions. The results from some of the extracts as given in Table 5 show for the length of time indicated, that the decomposition, if there was any, was not large.

TABLE 5.

SHOWING THE EFFECT OF HEATING THE EXTRACT TO 200°
IN AN ATMOSPHERE OF CO₂.

Extract	Weight of Extract	Time of Heating	Weight after Heating	Difference
A	.895	1 hour	.893	— .002
B	1.027	1 "	.997	— .030
C	.932	2 "	.932	— .000

9. *Method of Procedure.*—As a result of the above experiments the following process was uniformly followed. Five grams of coal ground to 100 mesh, which had been dried at 105° for one hour, were extracted with 50 to 60 cc. of redistilled phenol. The extraction cones, previous to use, were dried and weighed with the exclusion of moisture. After extraction with phenol, the cone with the coal was placed in a Soxhlet extractor and washed for two hours with alcohol and then for two hours with ether. The ether was allowed to evaporate from the cone for a short time and the residue, when almost dry, was shaken out into a 100 cc. Erlenmeyer flask. The residue was then dried for one hour at 200° in a slow stream of carbon dioxide. The flask was allowed to cool and the carbon dioxide displaced by dry air. The flask was then weighed and the residue sealed in a small sample tube. The weight of the small amount of residue adhering to the cone was obtained by drying the cone at 105° and weighing. The phenol containing the extract was poured into a 200 cc. round-bottomed flask. The last trace of the extract was washed out of the Kjeldahl flask with pure phenol. The phenol was then distilled at reduced pressure (15 to 25 mm.) in the presence of carbon dioxide. The temperature of the Woods' metal bath was then raised to 200°, and carbon dioxide conducted through the flask to remove the last traces of phenol vapor. The flask was then transferred to an oven and maintained for one hour at 200°, while a slow current of carbon dioxide

was continued through the flask. After cooling, the carbon dioxide was displaced with dry air and the flask weighed. The extract was pulverized and preserved in sealed tubes.

After all the details of the apparatus had been worked out and the sources of error in the method corrected as far as possible, a number of coals were extracted. The method just outlined was followed in detail. The extractions were carried out to test the constancy of the results and to find the limits of accuracy by comparing the sum of extract and residue with the amount of coal taken. The results are shown in Table 6.

TABLE 6.
WEIGHT OF EXTRACT AND RESIDUE FROM FIVE GRAMS OF COAL.

Table No.	Sample	Weight of Extract	Weight of Residue	Total Weight
1	Williamson Co., Ill.....	1.120	4.006	5.126
2	Macoupin Co., Ill.....	1.458	3.674	5.132
3	Madison Co., Ill.....	1.691	3.451	5.142
4	Vermilion Co., Ill.....	1.826	3.236	5.062
5	Madison Co., Ill.....	1.446	3.642	5.088
6	Logan Co., Ill.....	1.887	3.219	5.106
7	Jackson Co., Ill.....	1.246	3.878	5.125
8	Sangamon Co., Ill.....	1.959	3.139	5.098
9	Montgomery Co., Ill.....	1.559	3.498	5.057
10	Williamson Co., Ill.....	1.327	3.749	5.076
11	Vermilion Co., Ill.....	1.937	3.104	5.041
12	Vermilion Co., Ill.....	1.689	3.372	5.061
13	Williamson Co., Ill.....	1.616	3.491	5.107
14	Pocahontas, W. Va.....	.090	4.923	5.013
15	Lignite, Wyo.630	4.559	5.189

It will be noted in the above table that the weight of extract combined with the weight of residue was in all cases above the weight of coal taken. This is to be accounted for in the following manner. Experiments already given (page 7) showed that when 50 cc. of phenol were distilled for five hours that the amount of decomposition product was .015 gram. For twenty hours the amount of decomposition product would be .06 gram provided the decomposition was regular and that the presence of coal did not in any way tend to increase the rate of decomposition. It must be admitted that the alteration of phenol during all of the extractions would not be the same for the reason that the rate of distillation could not be controlled accurately and as a result the decomposition would vary somewhat in amount. On examining the results above it will be seen that the sum of the weight of extract and residue was from .013 to .189 gram in excess of the original weight of the coal. These two results are extreme cases. Now granting that the amount of decomposition of phenol would

vary from one-half to two times the amount determined in the blank test, then the excess of weight from the process would be substantially accounted for by referring it to the phenol residue. Furthermore, oxidization or absorption of water by either the extract or residue would cause an increase in weight and would tend to augment the plus error observed.

It might be well to note in this connection that since the decomposition product from the phenol will appear in the extract only, a corrected weight of the extract would therefore be obtained by subtracting the weight of residue from the original weight of the coal. Thus the weight of the coal minus the insoluble residue is the corrected factor showing the degree of extraction.

B. APPLICATION OF THE METHOD TO DIFFERENT TYPES OF COALS.

While the preliminary work was done on two coals and though the methods were not as accurate as those finally devised, it could be easily seen that the two coals contained markedly different amounts of material soluble in phenol. One coal was from the central and the other from the southern part of the state. As a means of comparison of coals from different localities it was decided to make an extraction of coals from the same and from different districts. Samples of Illinois coals were, therefore, obtained from local dealers. The most recent shipments and large lumps were selected as best suited to the purpose. Samples of Pocahontas coal and lignite were also obtained. The name, location, volatile matter, fixed carbon, and ash of the coals are given in the following table.

TABLE 7.
SHOWING ANALYSIS OF COALS. DRY BASIS.

Table No.	Source of Sample	Ash per cent	Volatile Matter per cent	Fixed Carbon per cent
1	Williamson Co., Ill.....	10.41	36.86	52.73
2	Macoupin Co., Ill.....	13.54	40.12	46.34
3	Madison Co., Ill.....	11.65	43.06	45.29
4	Vermilion Co., Ill.....	8.68	44.36	46.96
5	Madison Co., Ill.....	11.73	37.95	50.32
6	Logan Co., Ill.....	8.74	41.99	49.27
7	Jackson Co., Ill.....	6.01	34.93	59.06
8	Sangamon Co., Ill.....	5.18	44.48	50.34
9	Montgomery Co., Ill.....	11.84	42.12	46.02
10	Williamson Co., Ill.....	7.88	36.78	55.34
11	Vermilion Co., Ill.....	3.30	47.70	49.00
12	Vermilion Co., Ill.....	6.74		
13	Williamson Co., Ill.....	7.85	37.85	54.30
14	W. Va. Pocahontas.....	13.38	16.80	69.82

The above coals were extracted with phenol. From the amounts of extract and residue as given in Table 6 the percentage amounts of each substance may be calculated, but on account of the variations in the ash it will be better to make such calculations on the ash free basis. It will be noted in Table 7 that the amounts of ash in these coals varied widely, from 3.30 to 13.54 per cent. Since the percentage of ash in the extract is seldom over 0.2 per cent it is quite evident that the phenol does not exert an appreciable solvent action on the mineral constituents in the coal.¹ Therefore, it is present simply as an inert material and serves to dilute the coal substance. If the amounts of residue and extract in one coal are to be compared with those same constituents in another coal, it can be done only after the residue and extract in each have been calculated to an ash free basis.

The following formula was used in this calculation:

$$\frac{\text{Per cent Residue as weighed} - \text{per cent ash in coal}}{100 - \text{per cent ash in coal}} = \text{Ash free Residue}$$

The corrected ash free extract was found as indicated on page 15 by subtracting the per cent of ash free residue from 100. As stated above the extract is not entirely free from ash, but as this was small in amount it was not considered in these calculations. The weight of residue and extract for Illinois coals were taken from Table 6 and calculated to an ash free basis. The results in the order of decreasing residue and increasing extract are presented in Table 8.

TABLE 8.
SHOWING PERCENTAGE OF INSOLUBLE RESIDUE AND EXTRACT
REFERRED TO THE ASH FREE SUBSTANCE.

Table No.	Description of Sample	Percentage of Ash free Residue	Percentage of Ash free Extract
1	Williamson Co., Ill.....	77.81	22.19
7	Jackson Co., Ill.....	76.14	23.86
10	Williamson Co., Ill.....	72.85	27.15
2	Macoupin Co., Ill.....	69.33	30.67
5	Madison Co., Ill.....	69.24	30.76
13	Williamson Co., Ill.....	67.26	32.74
9	Montgomery Co., Ill.....	65.93	34.07
12	Vermilion Co., Ill.....	65.09	34.91
3	Madison Co., Ill.....	64.94	35.06
4	Vermilion Co., Ill.....	61.37	38.63
6	Logan Co., Ill.....	60.97	39.03
8	Sangamon Co., Ill.....	60.76	29.24
11	Vermilion Co., Ill.....	60.80	39.20

1. Frazer and Hoffman, Bureau of Mines, Technical Paper 5, p. 8.

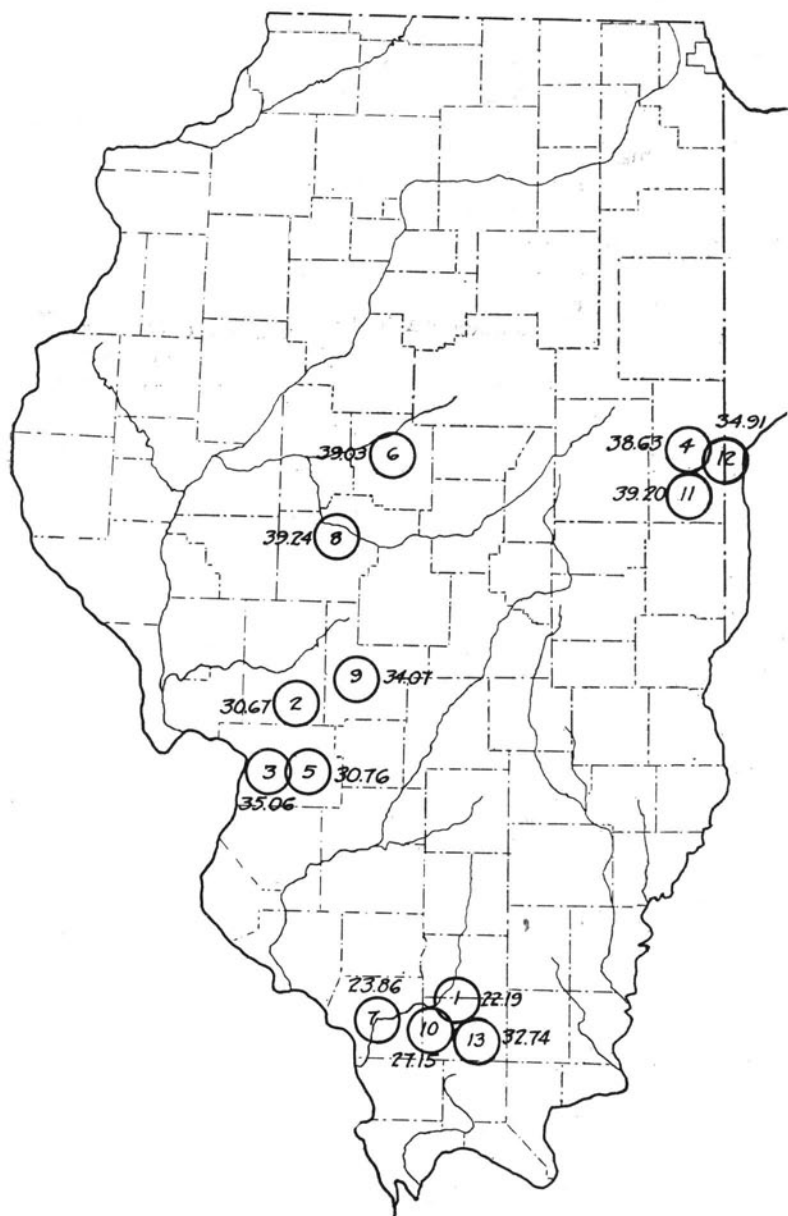


FIG. 4.
LOCATION OF THE ILLINOIS COALS.

For purposes of geographical comparison the location of the Illinois coals together with the percentage of ash free extract has been shown on a map (page 17). The coals may be divided into three classes; (1) those low in extract; (2) those medium in extract; and (3) those high in extract. The class low in extract, i. e., from 20-30 per cent, is located in the extreme southern end of the State and is confined as far as the tests extend to Jackson and Williamson counties. The class of medium extract, i. e., 30-35 per cent., is located in Madison, Macoupin, and Montgomery counties. The class high in extract, i. e., above 35 per cent, extends through the middle of the State. The coals which gave the high amount of extract were from Sangamon, Logan, and Vermilion counties.

From this preliminary survey of the coals it seems that this method of analysis may lead to a much better comparison of coals or a closer subdivision of varieties than can be shown by the ordinary methods of analysis. Of course, the study must be much wider in scope before a fixed and definite conclusion can be drawn as to its applicability in this connection.

C. GENERAL PROPERTIES OF EXTRACT AND RESIDUE.

10. *Relation of Extract and Residue to Coking Properties.*—The relation of extract and residue to the coking properties was studied by making volatile matter determinations according to a specified method.¹ The volatile matter determination was useful in two ways: (1) the percentage of volatile matter was indicated, and (2) the nature of the coke produced could be roughly determined by examining the material remaining in the crucible. The percentage of volatile matter will be given in another connection.

The "coke" produced from the volatile matter determination of the insoluble residue showed very little coherence. The so-called "coke" was in fact not a coke at all. Its tendency to hold together depended to a certain extent upon the coal from which the residue had been obtained, for in some cases the material fell to pieces on being dumped out of the crucible and in all cases it could be disintegrated between the fingers. It was much duller in appearance than ordinary coke. The residue, therefore, could properly be designated as non-caking and in consequence non-coking.

The extract and residue differed widely in character. When heated to 300° to 350°, the extract softened but it did not produce a sticky material at first. Later, as the temperature was raised, it became sticky. On cooling it again assumed the compact brittle state, which

1. Stanton and Fieldner, "Methods of Analyzing Coal and Coke", Technical Paper 8, Bureau of Mines (1912).

it originally possessed. The extract when subjected to the volatile matter test swelled to three or four times its original size and gave a large amount of volatile matter. The material remaining after the test possessed a bright, shiny appearance. It was very friable. However, it was a material entirely different from that produced from the residue.

If the residue and extract are mixed in the ratio in which they occurred in the coal and a volatile matter determination made, the material remaining is a coke about as strong in character and similar in appearance as that produced by the coal itself. From this it is evident that the coking constituent is the soluble extract and that its function is that of a cementing substance.

II. *Effect of Heating the Residue and Extract in the Air.*—It is well known that coal oxidizes readily in the air at 200°. It was desired to learn the behavior of the residue and extract when heated under the same conditions. The residue was weighed and heated in contact with air in an oven at 200° for successive one hour periods. The results are given in the following table.

TABLE 9.
SHOWING THE INCREASE IN WEIGHT OF RESIDUE WHEN
HEATED AT 200°.

	Weight	Increase
Weight of residue.....	0.6782	0.0000
after 1st one hour period.....	0.6868	0.0086
after 2nd one hour period.....	0.6903	0.0121
after 3rd one hour period.....	0.6926	0.0144
after 4th one hour period.....	0.6952	0.0170
after 5th one hour period.....	0.6960	0.0178
after 6th one hour period.....	0.6973	0.0191
after 7th one hour period.....	0.6982	0.0200

The extract was also heated to 200° in the air. The results for the extract are given in Table 10. The time of the first heating was 30 minutes and after that each successive period was one hour.

TABLE 10.
SHOWING THE INCREASE IN WEIGHT OF THE EXTRACT WHEN
HEATED TO 200°.

	Weight	Increase
Weight of extract.....	0.5718	0.0000
after heating 30 min.....	0.5720	0.0002
after 1st one hour period.....	0.5745	0.0027
after 2nd one hour period.....	0.5770	0.0052

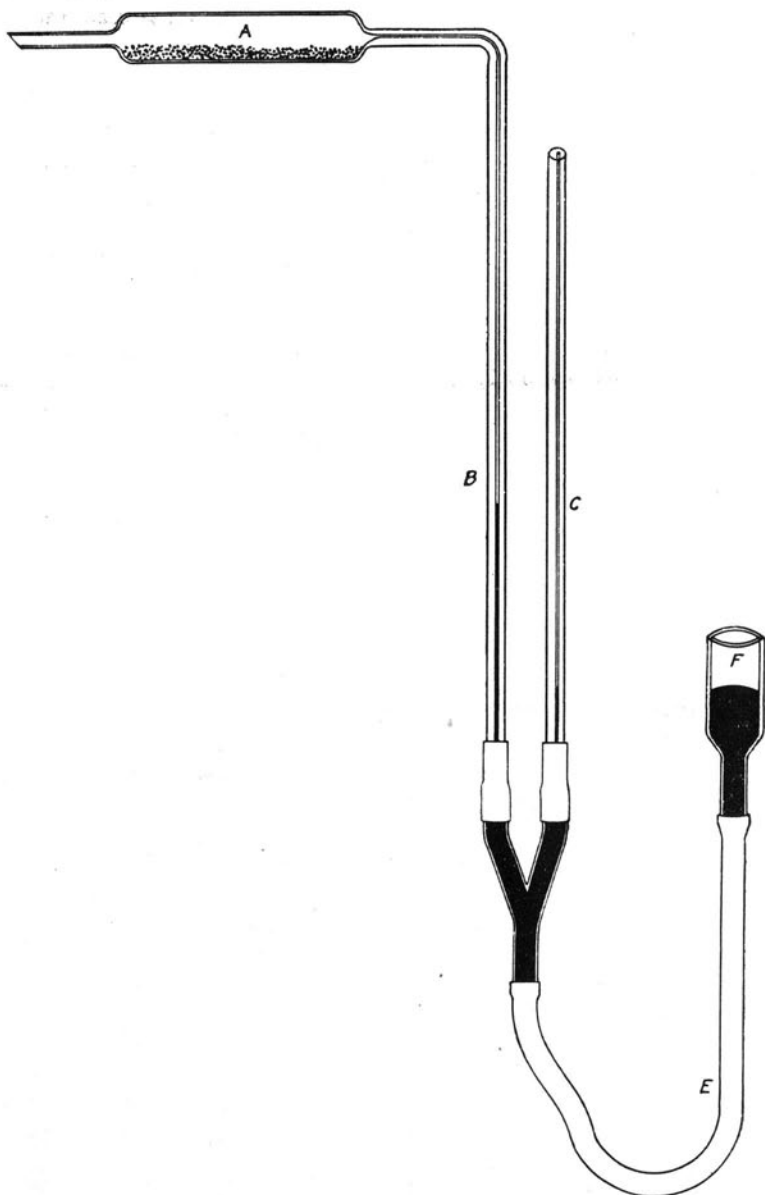


FIG. 5.
ABSORPTION OF OXYGEN BY COAL RESIDUES.

It was thus shown that both the residue and extract increased in weight when heated and that the rate of increase was faster in the case of the insoluble residue. These experiments are taken to mean that the increase in weight is an indication of oxidation of residue and extract.

12. *Avidity of Residue and Extract for Water and Oxygen at Ordinary Temperature.*—The hygroscopic nature of the residue was one of the properties to be noticed first. On exposure to air it increased in weight rapidly, and on drying at 105° for half an hour most of the water was driven off. However, it did not return to its original weight on drying, so that the excess in weight was probably caused by oxidation of the material.

The hygroscopic nature of the extract was also noticeable but it was not as marked as that of the residue. The extract increased in weight when exposed to the air, and the moisture could be driven out again by drying at 105° for thirty minutes.

Table II shows the increase in weight of extract and residue when exposed to the moisture of the air and the decrease in weight when they were dried at 105° for 30 minutes.

TABLE II.
EFFECT OF OXIDATION ON RESIDUE AND EXTRACT.

Substance	Weight	Weight after Exposure	Increase	Weight after Drying	Variation in Weight from Initial Material
Residue3029	.3129	.0100	.3037	+ .0008
Extract2467	.2485	.0018	.2462	— .0005

Oxidation of the residue and extract at ordinary temperature was also studied. Because of the hygroscopic nature of the residue and extract, it was difficult to determine whether oxidation was actually taking place or not. The rate of oxidation, if it occurred, would necessarily be slow at room temperature. Several experiments were performed for the purpose of determining whether or not the material did oxidize. Obviously, the experiment must be so carried out that moisture increases may be obviated in order to know definitely that the material had oxidized and had not taken up moisture.

The apparatus for showing the absorption of oxygen consisted of a barometer for measuring diminished pressure in a sealed space, which contained oxygen and the material to be tested. The plan of such an apparatus is shown in Fig. 5. The apparatus consists of

a 10 cc. pipette (A) fused to a capillary tube (B) 900 mm. in length. The capillary tube is connected at the bottom by a Y-tube to another capillary tube (C) which serves as a leveling tube and through which oxygen can be introduced into the apparatus. The Y-tube is connected by a rubber tube (E) to a leveling bulb (F).

In setting up the apparatus the procedure was as follows: One gram of extract (or residue) was introduced into the pipette and the end sealed off. Mercury was then placed in E and F, and a pinch clamp placed on E. The height of the mercury was such that it did not disconnect B and C. The apparatus was then evacuated repeatedly and filled five or six times with commercial oxygen which was dried by passing it through sulphuric acid. This procedure was followed to displace the air in the apparatus with oxygen. Finally a very slightly diminished pressure was maintained in the apparatus, the pinch cock on E opened, and the leveling tube raised. If this procedure were carried out properly, the mercury in B would stand a few mm. above the rubber connection and the mercury in both the capillary tube C and the leveling bulb F would stand at the same level. At certain intervals simultaneous temperature and barometric readings were taken and the true pressure calculated.

Table 12 shows the results of an experiment on the extract.

TABLE 12.

SHOWING THE AVIDITY OF THE EXTRACT FOR OXYGEN MEASURED IN DIMINISHED PRESSURE (mm.).

Time after Standing.	Diminished Pressure in mm.
0	0
23 hours	36
64 "	56
88 "	76
112 "	91
136 "	96
208 "	125

TABLE 13.

SHOWING THE AVIDITY OF THE RESIDUE FOR OXYGEN MEASURED IN DIMINISHED PRESSURE (mm.).

Time after Standing.	Diminished Pressure in mm.
0	0
26 hours	86
49 "	123
90 "	154
114 "	188
138 "	209
160 "	220
232 "	269



FIG. 7.
COKE FROM VERMILION COUNTY COAL.



FIG. 8.
INSOLUBLE RESIDUE FROM VERMILION
COUNTY COAL—AFTER DESTRUCTIVE
DISTILLATION.



FIG. 9.
PHENOL SOLUBLE EXTRACT FROM VERMILION
COUNTY COAL—AFTER DE-
STRUCTIVE DISTILLATION.



FIG. 10.
COKE FROM VERMILION COUNTY COAL.



FIG. 11.
INSOLUBLE RESIDUE FROM WILLIAMSON
COUNTY COAL—AFTER DESTRUC-
TIVE DISTILLATION.



FIG. 12.
PHENOL SOLUBLE EXTRACT FROM WIL-
LIAMSON COUNTY COAL—AFTER
DESTRUCTIVE DISTILLATION.

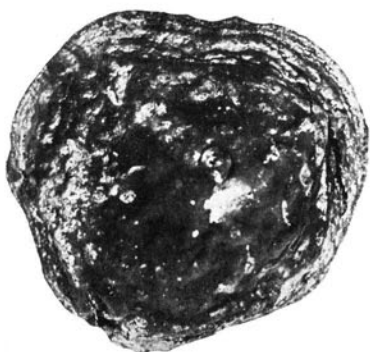


FIG. 13.
COKE FROM VERMILION COUNTY COAL
WITHOUT PREVIOUS HEATING

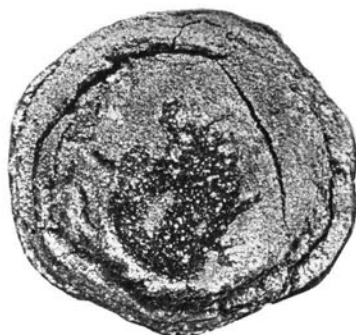


FIG. 14.
SAME AS IN FIG. 13.
PREVIOUSLY HEATED 26 HOURS AT 105°.

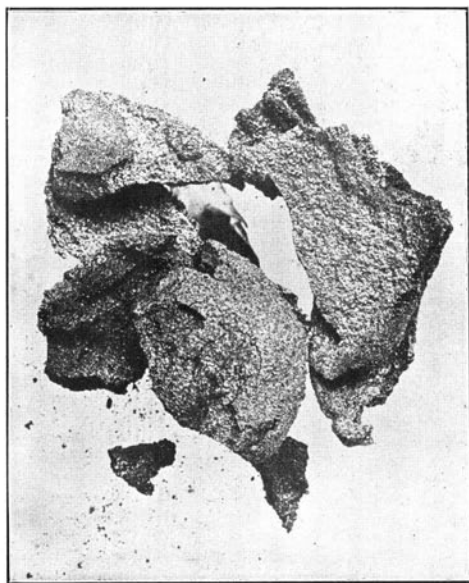


FIG. 15.
SAME AS IN FIG. 13.
PREVIOUSLY HEATED 50 HOURS AT 105°.

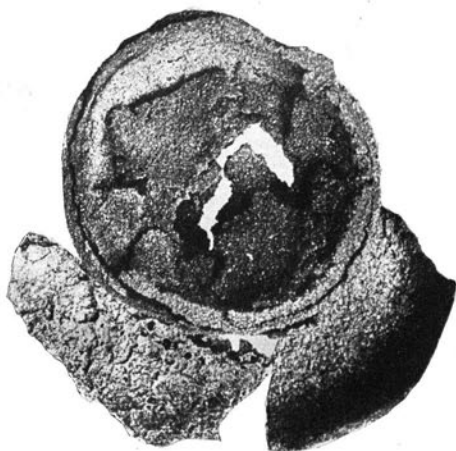


FIG. 16.
SAME AS IN FIG. 13.
PREVIOUSLY HEATED 120 HOURS AT 105°.



FIG. 17.
COKE FROM WILLIAMSON COUNTY COAL
WITHOUT PREVIOUS HEATING.



FIG. 18.
SAME AS IN FIG. 17.
PREVIOUSLY HEATED 26 HOURS AT 105°.



FIG. 19.
SAME AS IN FIG. 17.
PREVIOUSLY HEATED 50 HOURS AT
105°.

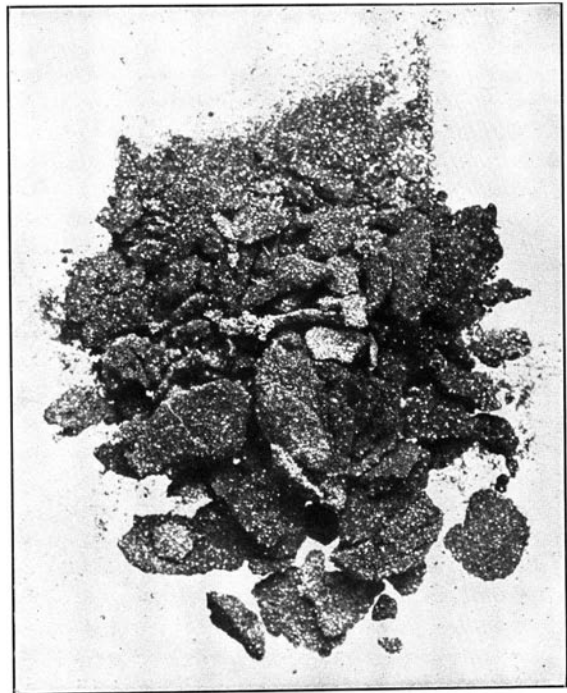


FIG. 20.
SAME AS IN FIG. 17.
PREVIOUSLY HEATED 120 HOURS AT 105°.

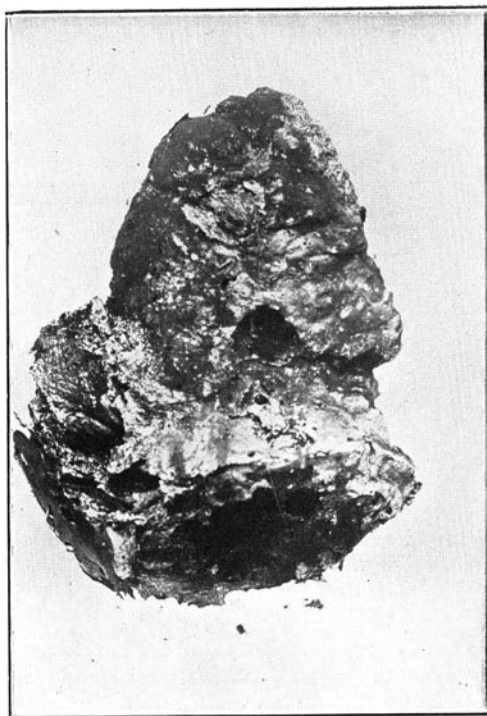


FIG. 21.
COKE FROM SOLUBLE EXTRACT, VERMILION
COUNTY COAL. NO PREVIOUS
HEATING OF EXTRACT.

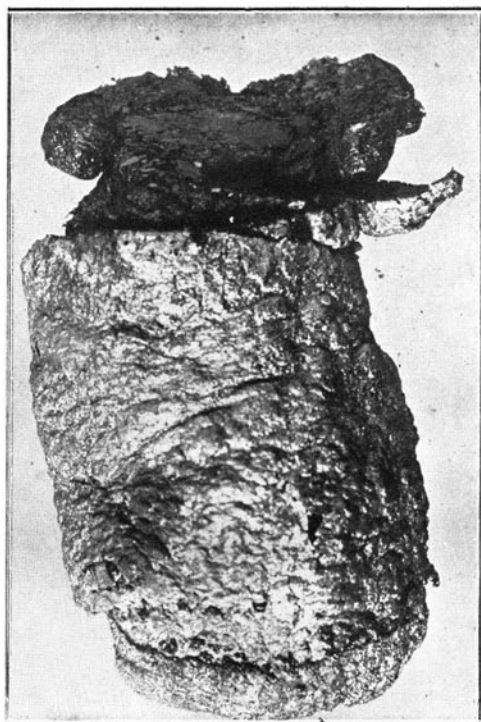


FIG. 22.
SAME AS IN FIG. 21.
PREVIOUSLY HEATED 50 HOURS AT 105°.

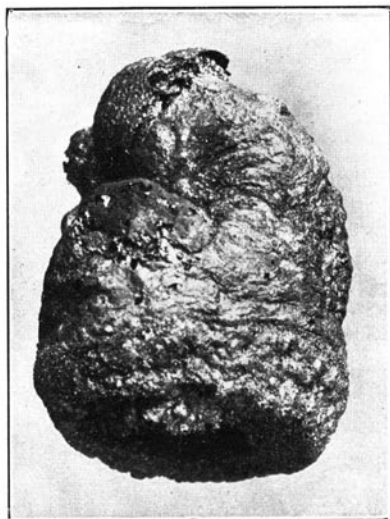


FIG. 23.
SAME AS IN FIG. 21.
PREVIOUSLY HEATED 122 HOURS AT 105°.



FIG. 24.
SAME AS IN FIG. 21.
PREVIOUSLY HEATED 216 HOURS AT 105°.

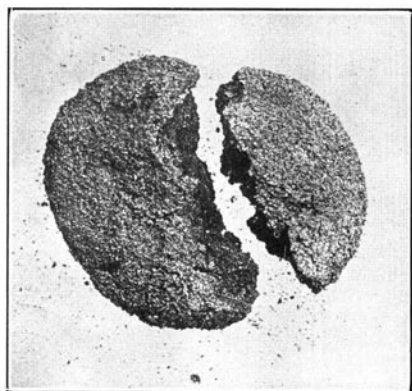


FIG. 25.
COKE FROM INSOLUBLE RESIDUE, VERMIL-
ION COUNTY COAL. NO PREVIOUS
HEATING OF RESIDUE.

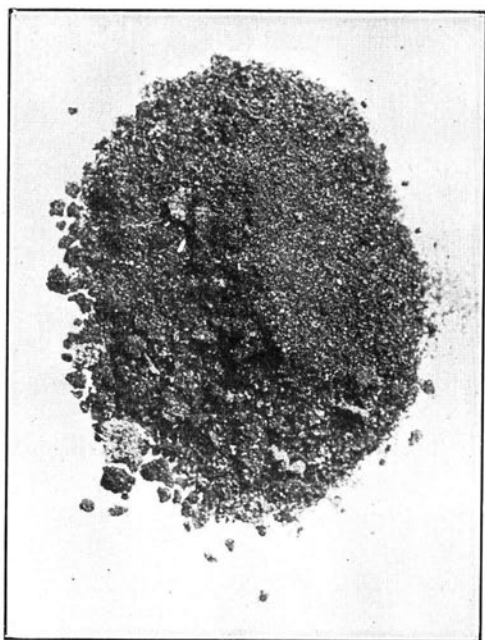


FIG. 26.
SAME AS IN FIG. 25.
PREVIOUSLY HEATED 50 HOURS AT 105°.

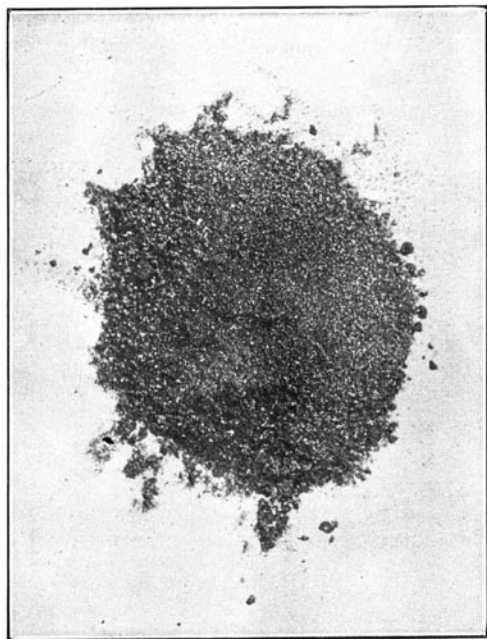


FIG. 27.
SAME AS IN FIG. 25.
PREVIOUSLY HEATED 122 HOURS AT 105°.

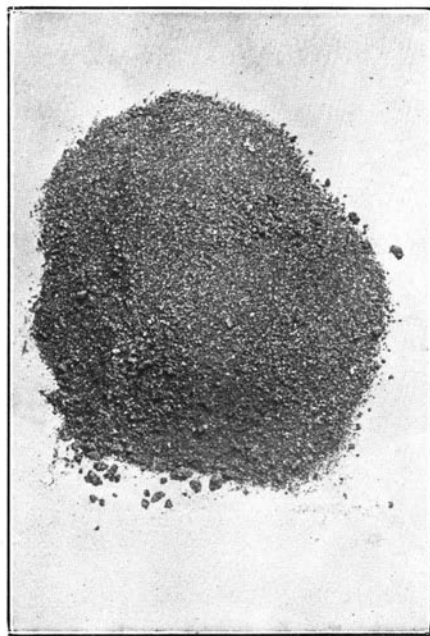


FIG. 28.
SAME AS IN FIG. 25.
PREVIOUSLY HEATED 216 HOURS AT 105°.

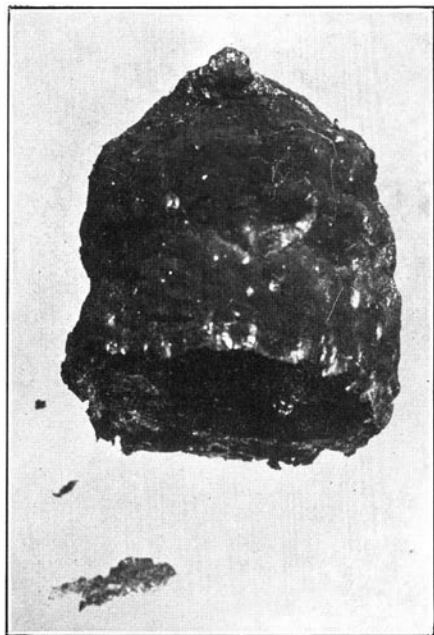


FIG. 29.
COKE FROM SOLUBLE EXTRACT, WILLIAMSON COUNTY COAL. NO PREVIOUS HEATING OF EXTRACT.

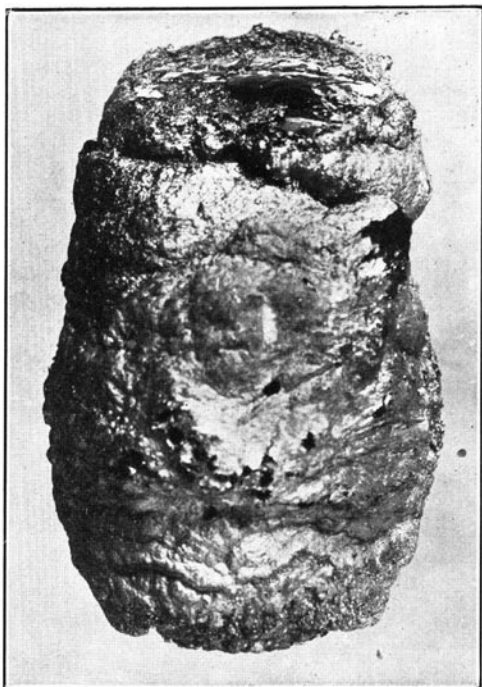


FIG. 30.
SAME AS IN FIG. 29.
PREVIOUSLY HEATED 90 HOURS AT 105°.

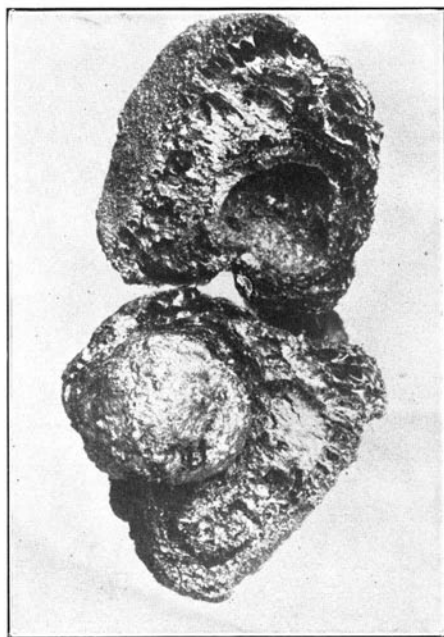


FIG. 31.
SAME AS IN FIG. 29.
PREVIOUSLY HEATED 242 HOURS AT 105°.

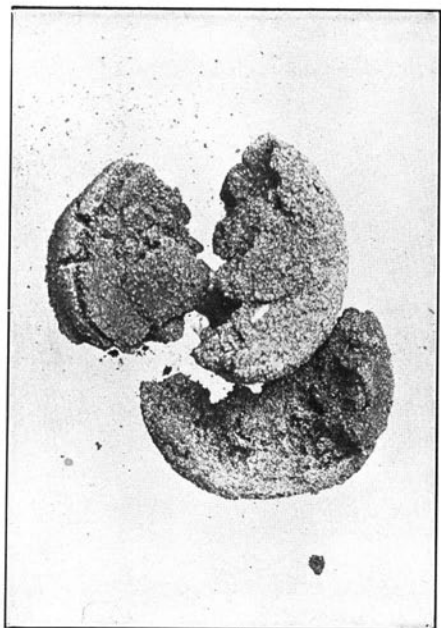


FIG. 32.
COKE FROM INSOLUBLE RESIDUE, WIL-
LIAMSON COUNTY COAL. NO PRE-
VIOUS HEATING OF RESIDUE.

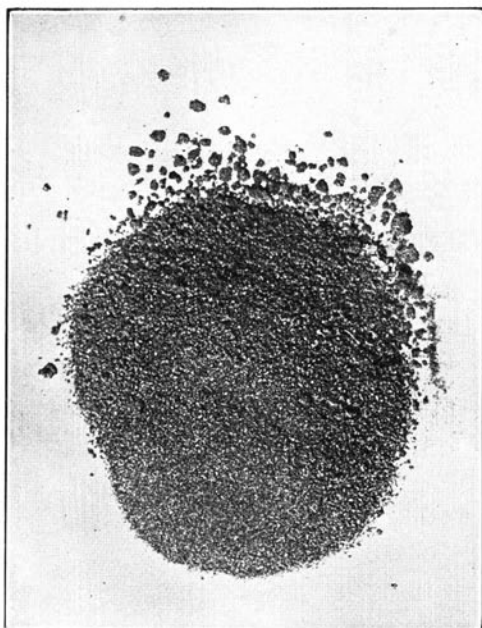


FIG. 33.
SAME AS IN FIG. 32.
PREVIOUSLY HEATED 90 HOURS AT 105°.

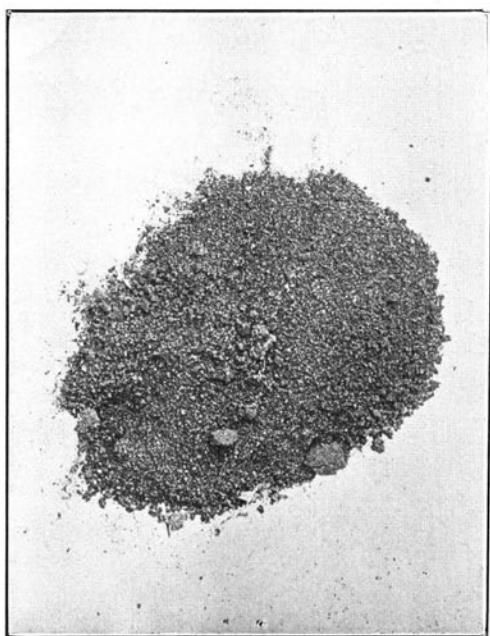


FIG. 34.
SAME AS IN FIG. 32.
PREVIOUSLY HEATED 242 HOURS AT 105°.

For purposes of comparison of the absorption rates of extract and residue, the above results were plotted. Plotting time against diminished pressure in millimeters, it is seen that the rate of absorption was faster in the case of the residue.

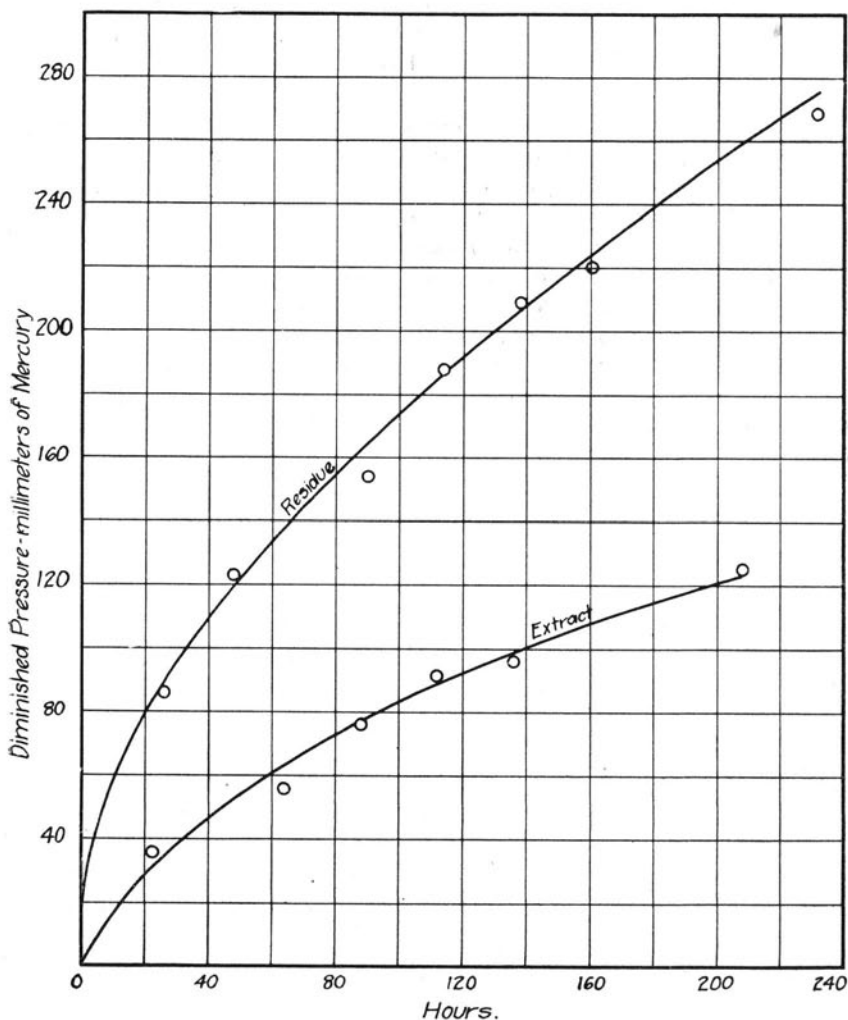


FIG. 6.

RATE OF ABSORPTION OF OXYGEN BY RESIDUE AND EXTRACT.

The results as given in Tables 12 and 13 show that oxygen was taken up by both the residue and extract. The amount of oxygen that

had been absorbed when the experiment was stopped was about 4-5 cc. in the case of the residue, and 2-2½ cc. in the case of the extract. The experiment does not show absolutely that oxidation had taken place, but from our knowledge of coal we would be led to suspect that such was the case. At any rate it can be said that the coal was either oxidized or that it was holding the oxygen in an occluded state. Further experiments given below show that the reaction was one of oxidation.

13. *Volatile Matter Determination of Coal, Residue, and Extract.*—Something of the relation of residue and extract to coking properties has already been discussed. Further data as to the relative amounts of volatile matter in the two divisions were desired.

The percentage of volatile matter and fixed carbon in the residue and extract from different coals was therefore determined and for purposes of comparison, volatile matter determinations were also made on the coals under examination. These results are shown in Table 14.

The fuel ratio or the ratio of fixed carbon to volatile matter is also given in Table 14. The use of this ratio is advantageous for the reason that we have in it a method of comparison which does not readily appear from the percentages of fixed carbon and volatile matter alone.

TABLE 14.

SHOWING THE PERCENTAGE OF FIXED CARBON AND VOLATILE MATTER IN COAL, RESIDUE, AND EXTRACT.

Table No.	Substance	Fixed Carbon per cent		Volatile Matter per cent		Fuel Ratio	
6	Lincoln Coal	49.27		41.99		1.173	
	Residue		47.15		39.63		1.190
	Extract		47.25		52.39		.902
7	New Kentucky Coal.....	59.06		34.93		1.691	
	Residue		57.90		34.51		1.678
	Extract		51.52		48.42		1.064
8	Springfield Coal	50.34		44.48		1.132	
	Residue		50.26		41.90		1.200
	Extract		47.61		51.98		.916
10	Carterville Coal	55.34		36.78		1.504	
	Residue		54.83		34.77		1.577
	Extract		51.79		48.10		1.077
9	Montgomery Co. Coal....	46.02		42.12		1.093	
	Residue		43.91		39.88		1.101
	Extract		48.82		50.45		.968
11	Catlin Coal	49.00		47.70		1.027	
	Residue		49.02		46.01		1.066
	Extract		47.35		52.30		.905
14	Pocahontas Coal	69.82		16.80		4.156	
	Residue		68.68		17.72		3.876

The fuel ratios of coal, residue, and extract are separately shown in Table 15. Here the results appear in such a form that the fuel

ratio of one coal can easily be compared with that of another. Likewise, the fuel ratio of the residue and extract can be compared.

The fuel ratios for the several extracts are uniformly lower than the ratios for the residues. It is to be remembered that a decrease in the ratio number denotes an increase in the relative amount of volatile constituent. The table is made to show in the last column the relation between the various samples, the fuel ratio for the residue being used in each case as the basis of reference. It is quite possible that when reduced to this basis some idea may be had as to the coking property of a coal. While more data would be required on which to base a conclusion, it may be said that the high percentage differences as shown in the table are consistent in representing the more pronounced coking property of the samples listed.

TABLE 15.
SHOWING THE FUEL RATIO OF COAL, RESIDUE, AND EXTRACT.

Table No.	Description	Coal $\frac{C}{vol}$	Residue $\frac{C}{vol}$	Extract $\frac{C}{vol}$	Percentage of decrease of ratio in extract over ratio for residue referred to residue as basis
7	New Kentucky	1.705	1.678	1.062	40.3
10	Carterville Nut	1.504	1.578	1.077	38.3
9	Montgomery Co.	1.093	1.101	.968	26.5
6	Lincoln	1.173	1.190	.902	32.2
11	Catlin	1.027	1.066	.904	13.7
8	Springfield	1.133	1.200	.916	24.1

14. *Ultimate Analysis of Coal, Residue, and Extract.*—A difference in the chemical constitution of the residue and extract would be expected, when we consider the marked differences in their physical properties. In order to find out if there was a difference in the chemical constitution of these two substances, ultimate analyses were made of extract, residue, and coal from a number of mines as follows:

TABLE 16.
VERMILION COUNTY COAL.
SHOWING THE ULTIMATE ANALYSIS OF COAL, RESIDUE, AND EXTRACT.

Substance	Carbon	Hydrogen	Nitrogen	Sulphur	Ash
Coal	72.40	5.35	1.27	2.72	8.68
	72.36	5.34	1.16		
Residue	69.16	4.92	1.29	3.72	13.03
	69.08	4.90			
Extract	81.14	5.88	1.24	1.33	.53
	80.97	6.04			

These results were averaged and calculated to an ash free basis.

TABLE 17.
VERMILION COUNTY COAL.
SHOWING THE ULTIMATE ANALYSIS OF COAL, RESIDUE, AND EXTRACT
ON AN ASH FREE BASIS.

Substance	Carbon	Hydrogen	Nitrogen	Sulphur	Oxygen
Coal	79.26	5.86	1.32	2.97	10.59
Residue	79.48	5.68	1.48	4.28	9.12
Extract	81.48	5.99	1.25	1.34	9.94

The results of the ultimate analyses of Williamson County coal, residue, and extract are given in Table 18.

TABLE 18.
SHOWING THE ULTIMATE ANALYSIS OF COAL, RESIDUE, AND EXTRACT.

Substance	Carbon	Hydrogen	Nitrogen	Sulphur	Ash
Coal	74.58	4.72	1.63	2.35	7.85
	74.42	4.89			
Residue	72.46	4.70	1.53	2.52	11.35
	72.17	4.73			
Extract	82.69	5.54	1.73	1.42	.56
	82.60	5.54			

These results were averaged and calculated to an ash free basis.

TABLE 19.
WILLIAMSON COUNTY COAL.
ULTIMATE CONSTITUENTS OF COAL, RESIDUE, AND EXTRACT
CALCULATED TO THE ASH FREE BASIS.

Substance	Carbon	Hydrogen	Nitrogen	Sulphur	Oxygen
Coal	80.85	5.21	1.77	2.55	9.62
Residue	81.57	5.32	1.72	2.84	8.55
Extract	83.11	5.57	1.74	1.43	8.15

On examining the results of the ultimate analysis, it will be seen that there was little difference in the proportion of the elements in the coal, residue, and extract. The percentage of carbon and hydrogen is slightly higher in the extract. However, because of the small variation no special significance can be attached to this fact.

The oxygen content was the chief point of interest to be consid-

ered in the analysis. Previous work with solvents such as pyridine¹ and benzene² indicated that the material dissolved from coal by these solvents contained a smaller proportion of oxygen. Professor Lewes³ states that "degradation products of the original vegetation are to be found in the bituminous coals, the residual body and humus forming the basis, which is luted together by the hydrocarbons and resins, and the characteristics of the various kinds of coal are dependent upon the proportion in which the four groups of the conglomerate are present". Assuming that the hydrocarbons and resins were the more soluble in phenol and would be dissolved by the phenol, it would be only natural to conclude that the extract would be low in the amount of oxygen it contained. Furthermore, if the bodies low in oxygen were dissolved, the residue would on that account contain a larger percentage of oxygen. When we consider that all the errors of the analysis are combined in the oxygen value, it would be impossible to state from the results of the above analyses that there was any difference in the percentage of oxygen in the extract from that of the residue.

It is to be noted that the percentage of sulphur in the residue is high. This is to be accounted for by the fact that the residue contained practically all of the ash that was present in the coal. That there was some sulphur in the extract was taken to indicate that a part of the sulphur in the coal was present in the organic form.

15. *Thermal Decomposition of Residue and Extract.*—The residue and extract were subjected to destructive distillation at 800° and the resulting gases analyzed. For purposes of comparison the coal was also treated in the same manner. The method for the destructive distillation of the material may be briefly described in the following manner.

One gram of the substance was placed in a hard glass test tube. Immediately above the substance was placed a short layer of asbestos which served the purpose of a tar scrubber and as a means of holding the extract in the lower part of the tube thus counteracting its tendency to foam. The tube was then connected by a rubber stopper, a glass stop cock, and a short piece of small rubber tubing to a 300 cc. gas holder which contained mercury. Previous to heating, the air was exhausted from the tube and from the connections by means of a filter pump. The test tube was then introduced into a gas furnace which had been previously heated to 800°.

The temperature was maintained between the temperature limits

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1. Clark and Wheeler, *J. of Chem. Soc.*, 103, 1709 (1913).
 2. Pictet and Ramseyer, *Arch. sci. phys. nat.*, 34, 234 (*Chem. Abstracts* 7, 1496) (1913).
 3. *Progressive Age*, 29, 1032 (1911).

of 780° and 810° throughout all of the experiments. The upper part of the test tube holding the rubber stopper was well insulated against the heat. As soon as the pressure in the tube was equal to the atmospheric pressure, the stop cock was opened and the gas collected. At the end of one hour the distillation was stopped. The gas was analyzed according to the usual procedure.

The average of duplicate determinations gave the following results for gases which were produced by the destructive distillation of Vermilion County coal, residue, and extract.

TABLE 20.

SHOWING THE COMPOSITION OF GASES PRODUCED BY DESTRUCTIVE DISTILLATION OF COAL, RESIDUE, AND EXTRACT.

Components of the Gas	Vermilion Co.	Residue	Extract
Carbon dioxide	18.1	15.1	12.9
*Ethylene	3.4	3.5	3.1
Oxygen5	.9	.1
Carbon monoxide	20.6	17.4	16.4
Hydrogen	35.6	39.2	47.8
Methane	17.4	18.1	13.4
**Ethane	1.6	1.5	2.8
Nitrogen	2.8	4.3	3.5

Coal, from Williamson County, residue and extract were also destructively distilled. The results of the analysis of the gas which was produced are given in Table 21.

TABLE 21.

SHOWING THE COMPOSITION OF GASES PRODUCED BY DESTRUCTIVE DISTILLATION OF WILLIAMSON COUNTY COAL.

Components of the Gas	Coal	Residue	Extract
Carbon dioxide	7.1	6.9	7.2
*Ethylene	3.1	2.9	2.6
Oxygen6	.8	.7
Carbon monoxide	12.8	16.5	11.8
Hydrogen	50.7	46.0	51.8
Methane	17.3	20.5	18.2
**Ethane	1.9	2.8	2.7
Nitrogen	6.5	3.6	5.0

* Including higher homologues and benzene.

** Including higher homologues calculated as ethane.

The work of Clark and Wheeler¹ on the extract and residue which was obtained by the extraction of coal with pyridine, showed a marked difference in the percentage composition of the gases which were produced by the destructive distillation of these substances. The methane and ethane in the gas from the extract were larger in amount than that obtained from the gas of the residue.

From their results it may be supposed that similar results might be obtained by the destructive distillation of the residue and extract obtained by extraction of coal with phenol. The above results as indicated in Tables 20 and 21 do not show an increase in the amount of methane in the gas from the extract but instead there was a decrease in this constituent and an increase in the hydrogen content.

It will be observed in Table 20 that the amount of carbon dioxide was high in all of the gases. This fact taken in conjunction with the fact that the Vermilion County coal had been in a finely ground condition for some time suggested that oxidation had taken place. This led to a consideration of that problem which will be treated under a subsequent heading.

The properties of the material which remained in the test tube after thermal decomposition, were very characteristic.

The coal gave a good sound coke of shiny appearance.

The residue gave a very poor coke. It could be easily broken and possessed a dull appearance.

The extract gave a very light and friable material. It had a very smooth surface after the glass was broken away. The surface was much brighter than that of the coke produced from the coal.

Photographs of the residues are shown in Figs. 7-12. Fig. 7 is of Vermilion County coal; Fig. 8, residue; and Fig. 9, extract. Fig. 10 is of the Williamson County coal; Fig. 11, residue; and Fig. 12, extract.

16. *The Effect of Oxidation on the Volatile Matter Determination.*—From some of the previous work it became evident that oxidation was playing an important role in producing changes in the residue and extract. It is a well-known fact that when coal is exposed it gradually loses its coking properties, and that this is due to the absorption of oxygen. It was desired to know if this was oxidation or simple absorption of oxygen. The first experiments were made on the coal itself. It was desired to see what effect heating at 105° would have on the percentage of volatile matter.

Coals from Vermilion County, and Williamson County were heated in air at 105° for different lengths of time and volatile matter determinations were made at successive stages of oxidation. The results on these two coals are given in Table 22.

1. J. Chem. Soc., 103, 1710 (1913).

TABLE 22.
SHOWING THE PERCENTAGE OF VOLATILE MATTER IN COAL AFTER
CONTINUED HEATING AT 105°.

Time after Heating. Hours	Vermilion County Coal		Williamson County Coal	
	Volatile Matter in coal	Difference	Volatile Matter in coal	Difference
0	44.36	0.00	37.85	0.00
26	42.39	-1.97	39.22	+0.37
50	41.78	-2.58	36.75	-1.10
120	40.51	-3.85	35.91	-1.94

These results show that the volatile matter decreased with progressive heating. The plus result which was obtained in the case of the Williamson County coal was in all probability a variable in the volatile matter determination. From the results of these two coals the decrease in volatile matter was more rapid in the case of the coal which had the higher percentage in the beginning. The material remaining in the crucible after the determinations indicated very plainly that the length of time of heating affected the coking properties as shown below.

The Vermilion County coal gave a very poor coke after 120 hours' heating. Figs. 13 to 16 show the material after the volatile matter determination. Fig. 13, coal without heating; Fig. 14, after heating 26 hours; Fig. 15, after heating 50 hours; Fig. 16, after heating 120 hours.

The Williamson County coal after heating 120 hours would not coke. Figs. 17 to 20 show the material after the volatile matter determination. Fig. 17, coal without heating; Fig. 18, after heating 26 hours; Fig. 19, after heating 50 hours; Fig. 20, after heating 120 hours.

The study of the effect of oxidation on the amount of volatile matter was extended to the residue and extract. The residue and extract from the Vermilion County coal were heated for various lengths of time and volatile matter determinations made. The results are given in Table 23.

TABLE 23.
SHOWING THE PERCENTAGE OF VOLATILE MATTER IN THE RESIDUE AND
EXTRACT AFTER HEATING AT 105°, VERMILION COUNTY.

Time of Heating. Hours	Volatile Matter in Residue	Difference	Volatile Matter in Extract	Difference
0	43.53	0.00	52.79	0.00
50	42.75	-0.78	52.22	-0.57
122	61.08	+17.55	47.68	-5.11
216	50.45	+7.92	48.23	-4.56

On examining these results it will be noted that the percentage of volatile matter in the extract decreased with the length of time of heating. It thus acted similarly to coal. On the other hand, the residue increased in the amount of volatile matter. This increase may be accounted for by the fact that during the volatile matter determination there was an excess of sparking. The material acted similarly to lignite coal when an attempt is made to coke a finely ground sample. There was evidence of considerable mechanical loss. The greater avidity of the residue for oxygen would also tend to increase the indicated amount of volatile matter by reason of the ultimate formation of CO_2 from the oxygen absorbed.

The material which remained after the volatile matter determination was of special interest. Figs. 21 to 24 show the extract after the volatile matter determination; Fig. 21, extract without previous heating; Fig. 22, after heating 50 hours; Fig. 23, after heating 122 hours; Fig. 24, after heating 216 hours. Heating the extract for 50 hours yields a more voluminous material by the volatile matter determination than is produced by the extract without heating. After heating 216 hours the extract had lost all tendency to swell but it still retained a slight coking tendency, i. e., the material did not fall to pieces.

Figs. 25 to 28 relate to the same source of material from Vermilion County, and show the material that remained after the volatile matter determination was made on the residue; Fig. 25 is the coke from residue without previous heating; Fig. 26, after heating 50 hours; Fig. 27, after heating 122 hours; Fig. 28, after heating 216 hours. If a volatile matter determination is made on the residue previous to heating a material is produced that will just adhere. After heating, the residue loses what little coking ability it originally possessed.

The residue and extract from Williamson County coal were also subjected to prolonged heating at 105° . The results are presented in Table 24.

TABLE 24.

SHOWING THE PERCENTAGE OF VOLATILE MATTER IN THE RESIDUE AND EXTRACT AFTER HEATING AT 105° , WILLIAMSON COUNTY COAL.

Time after Heating, Hours	Volatile Matter in Residue	Difference	Volatile Matter in Extract	Difference
0	37.44	0.00	47.21	0.00
90	56.40	+18.96	44.19	-3.02
242	67.33	+29.89	42.68	-4.53

A discussion of these results on Williamson County coal will not be taken up for they are identical in character with those obtained from the Vermilion County coal.

Figs. 29 to 31 show the material which remained after the volatile matter determination of the extract from Williamson County coal. Fig. 29, extract without heating; Fig. 30, after heating 90 hours; Fig. 31, after heating 242 hours.

Figs. 32 to 34 show the material which remained after the volatile matter determination was made on the residue from Williamson County coal; Fig. 32, residue without heating; Fig. 33, after heating 90 hours; Fig. 34, after heating 242 hours.

Reviewing this phase of the study it is seen that Figs. 7 to 34 inclusive, illustrate the effect of oxidation such as may occur upon simple exposure to the air at room temperature and at 105° , and for varying lengths of time. The temperature employed for the destructive distillation was approximately 800° . Before applying this temperature the samples were subjected to a vacuum for the purpose of removing the surrounding oxygen as well as that which might have been occluded by the sample. The effect upon the insoluble residues of long exposure to a temperature of 105° is hardly noticeable so far as modifying the coking property of that material is concerned, for the reason that this substance, even without exposure to oxidation, is practically devoid of any tendency to coke when highly heated. Quite the contrary is true in the case of the soluble extract. Its tendency to coke and so to ultimately produce coke is marked, but after long exposure to oxidizing conditions this property is practically lost. This feature also characterizes the coal, the unseparated extractive material being sufficiently pronounced in its behavior to govern the effect of oxidation on the unheated coal.

17. *The Effect of Oxidation on the Amount of Material Extracted from Coal by Phenol.*—The further effect of oxidation was noticed in a number of different ways. For example, the amount of material which could be extracted from coal depended to a certain degree on the extent of oxidation.

Williamson County coal was heated for five days at 105° in an atmosphere of oxygen. The amount of material extracted from this coal is shown in Table 25.

TABLE 25.
SHOWING THE EFFECT OF OXIDATION ON THE AMOUNT OF RESIDUE
AND EXTRACT IN COAL, WILLIAMSON COUNTY.

	Coal not previously heated	Coal heated before extracting	Change
Weight of residue.....	3.777	4.102	+.325
Weight of extract.....	1.484	1.094	-.390

A finely ground sample of Vermilion County coal was kept in a closed jar for four months. This coal was extracted with phenol and the amount of residue and extract determined. The coal was also heated at 105° for varying lengths of time in oxygen and subsequently extracted with phenol. The results of these experiments are shown in Table 26.

TABLE 26.
SHOWING THE EFFECT OF OXIDATION ON THE AMOUNT OF RESIDUE
AND EXTRACT IN COAL.

Condition of Coal	Weight of Residue	Increase in Weight of Residue	Weight of Extract	Loss in Weight of Extract
Fresh	3.236	0.000	1.826	0.000
Standing 4 mo.	3.494	+ .258	1.601	-.225
Heated 3 days.....	3.821	+ .585	1.325	-.501
Heated 6 days.....	3.860	+ .624	1.259	-.567
Heated 10 days.....	3.802	+ .566	1.242	-.584
Heated 15 days.....	3.795	+ .559	1.353	-.473

From the results in Tables 25 and 26 it is seen that the oxidation of coal increases the amount of residue insoluble in phenol, and that the decreased solubility becomes very marked when the coal is completely oxidized. Also it should be noted that in the sample of coal which was preserved in the sample bottle there was a decrease in the amount of soluble material. Thus the coal was being slowly oxidized although it was not directly exposed to the air. The fact that coal is so readily oxidized should be kept in mind in any work on coal and especially in work of the present nature.

18. *The Effect of Oxidation on the Composition of the Gases Produced by Destructive Distillation.*—During the work on the destructive distillation of coal as noted on page 28 one gas was produced which was high in the amount of carbon dioxide. This result, when considered in connection with the fact that the sample of coal was not

fresh, and in the light of the preceding experiment, would indicate that oxidation had taken place. It was desired to see what effect oxidation of the extract and residue would produce in the composition of the gases when destructively distilled. In order to have a sample of coal which was not already partially oxidized, a fresh sample of Vermilion County coal was secured. The residue and extract from this coal were destructively distilled and the gases analyzed. The residue and extract were also heated at 105° for 24 hours and then destructively distilled. The results from the analyses are as follows:

TABLE 27.
SHOWING THE COMPOSITION OF GASES PRODUCED BY DESTRUCTIVE DISTILLATION OF RESIDUE AND EXTRACT, VERMILION COUNTY COAL.

Gas	From Residue not oxidized	From Residue oxidized by heating	From Extract not oxidized	From Extract oxidized by heating
Carbon dioxide	5.2	8.7	6.8	10.0
*Ethylene	4.8	4.6	5.1	4.2
Oxygen3	.3	.2	.4
Carbon monoxide	15.1	15.2	11.9	19.0
Hydrogen	44.1	43.0	40.5	44.8
Methane	26.2	24.0	24.0	17.4
**Ethane	3.6	3.3	7.3	3.2
Nitrogen7	.9	4.2	1.0

The residue and extract from the old sample of Vermilion County coal were heated for 48 hours at 105° in the air. They were then destructively distilled and the gases analyzed. For purposes of comparison some of the residue and extract which had not been heated were destructively distilled and the gases analyzed. The results are presented in Table 28.

TABLE 28.
SHOWING THE COMPOSITION OF GASES PRODUCED BY DESTRUCTIVE DISTILLATION OF RESIDUE AND EXTRACT, VERMILION COUNTY COAL.

Gas	From Residue oxidized by standing only	From Residue oxidized by heating	From Extract oxidized by standing only	From Extract oxidized by heating
Carbon dioxide	8.3	14.4	10.1	12.4
*Ethylene	5.2	3.5	4.0	3.2
Oxygen9	.1	.4	.3
Carbon monoxide	14.2	18.5	21.0	14.6
Hydrogen	39.8	40.5	40.8	50.0
Methane	23.1	20.4	16.3	15.6
**Ethane	3.4	1.4	2.8	2.5
Nitrogen	5.1	1.2	4.6	1.4

* Including higher homologues and benzene.

** Including higher homologues calculated as ethane.

Extracts from the Vermilion County coal and also from Williamson County were heated for a much longer time and then destructively distilled. The extract from the Vermilion County coal was heated for 216 hours at 105° and that from Williamson County for 264 hours. The results are given in Table 29.

TABLE 29.
SHOWING THE COMPOSITION OF GASES PRODUCED BY THE DESTRUCTIVE
DISTILLATION OF EXTRACT.

Gas	From Vermilion County Extract after being heated for 216 hours.	From Williamson County. Extract after being heated for 264 hours.
Carbon dioxide	9.1	7.9
Ethylene	3.7	3.8
Oxygen3	.4
Carbon monoxide	17.9	16.1
Hydrogen	44.2	47.5
Methane	19.9	20.9
Ethane	3.1	2.1
Nitrogen	1.8	1.3

On examining the data in Tables 27 and 28 it is to be noted that the amount of carbon dioxide in the gas is higher after the material has been heated. Also, the percentage of carbon monoxide increased in every case except one. The fact that the percentage of methane and ethane in the gas from the material that had been heated was reduced in every case, should also be noted. The above results seem to indicate that the oxygen taken up during the heating at 110° was not held in an absorbed condition but that it had entered into chemical combination with the material. Furthermore the apparatus was exhausted to 15-20 mm. before the destructive distillation was started, and when the temperature was raised, any oxygen which was merely occluded would be given off as oxygen before a temperature was reached at which active oxidation with the formation of CO₂ would take place. As a result of these experiments it may be stated that upon exposure to oxidizing conditions oxygen enters into chemical combination with both residue and extract.

The results in Table 29 seem to show that the oxidation may reach a point of saturation so far as the ability of the molecular structure is concerned and that beyond this point, if the oxidizing conditions are continued, a dissipation of the oxygen compounds may occur, presumably being discharged as H₂O. However, further proof of this reaction after such long continued periods of heating should be made.

III. SUMMARY AND CONCLUSIONS.

(1) A study of solvents for the purpose of subdividing coal constituents without decomposition was carried out. Phenol was adopted as the most active solvent for the purpose of this investigation.

(2) An apparatus was devised for carrying out the extraction so that the temperature of the solvent would be above 110° .

(3) The application of the method to different types of coal showed that coal varied widely in the amount of material dissolved by phenol. The amount of soluble material seemed to differentiate sharply between subdivisions of coal types. On the ash and moisture free basis the high volatile coals of Vermilion County give 35-40 per cent of soluble material; the coals from Madison and Montgomery Counties give 30-35 per cent and the low volatile coals of Williamson County show 20-30 per cent of soluble material.

(4) Extraction of coal leaves a residue which will not coke: The coking constituent of the coal is in the extract.

(5) Residue and extract oxidize at room temperatures and more readily at 100° , the rate of oxidation being more rapid in the residue.

(6) Residue and extract possess an avidity for water as well as oxygen at ordinary temperatures. The residue shows the greater avidity in both cases.

(7) Volatile matter determinations show that the extract contains more volatile matter than the residue.

(8) The ultimate analysis of the coal, residue, and extract shows that the percentage composition of carbon, hydrogen, nitrogen, and oxygen is substantially the same.

(9) Destructive distillation of coal residue and extract gave gases of practically the same composition.

(10) Oxidation of coal, residue, or extract produces a lowering in the percentage of volatile matter.

(11) Oxidation decreases the amount of material which may be extracted from coal by phenol and the coking properties are decreased in proportion to the extent of the oxidation.

(12) Oxidation of coal, residue, and extract is shown by an increase in the percentage of carbon dioxide in the gases produced by thermal decomposition. It is concluded, therefore, that the oxygen absorbed is chemically held.

IV. HISTORICAL.

Professor Bedson¹ refers to work which was done by Dr. Smythe on coal at Göttingen in 1851. This work was published in a report

1. J. Soc. Chem. Ind., 27, 149 (1908).

printed for the Commissioners of the 1851 Exhibition. A brown coal from Brühl, near Cologne, was used in the work. The following solvents were used in the extraction in the order named: benzene, chloroform, alcohol, ethyl ether, petroleum ether, and acetone. Benzene extracted 3 per cent of the coal, chloroform 1.8 per cent, and alcohol 2.4 per cent. The other extracts were very small. Attempts were made to obtain pure substances from these extracts by the ordinary methods of purification used in organic chemistry.

Reinsch¹ in 1885 supposed that coal was composed of two substances, which could be distinguished by their action towards alkaline solution. With an alkaline solution he was able to isolate substances which were very characteristic in not being attacked by mineral acids.

Baker² in 1901 examined the solvent action of pyridine on certain classes of coals. The extraction was carried out for 50 hours in a Soxhlet extractor. Anthracite coal from South Wales gave very little material soluble in pyridine. A bituminous coal from Durham was soluble in pyridine to the extent of 20.4 per cent. The pyridine extracts were dark brown in color and showed fluorescence in some cases. An ultimate analysis did not show a concordant change in the proportions of elements present in the coal, residue, and extract.

Andersen and Henderson³ in 1902 extracted Bengal and Japan coal with pyridine. They selected samples from Boiaker (Bengal), Paronai and Yubari (Japan) and also, for comparison two Scotch coals: Linrigg Lower Dumgray and Bannockburn Main. They did not give the amounts which were dissolved. The pyridine extract, after the removal of the solvent, possessed a black lustrous appearance similar to bitumen. The extract from all of the coals was similar in character. Ultimate analyses of the extract were given and the results showed that there was little difference in the chemical constitution of the coals of Bengal and Japan, from those of Scotland. They stated that pyridine was the best solvent that had been used. The percentage of carbon, hydrogen, and nitrogen in the extract was about the same as that in the original coal. The coking properties of a poor coal could be entirely removed but that it was only partially removed in a strong coking coal.

Professor Bedson⁴ in 1908 worked upon the pyridine extract from certain gas coals. The coals varied from 22 to 33 per cent of pyridine extract. Also, four Cannel coals were extracted giving from 7 to 29

1. *Dingl. Poly. J.*, 256, 224. (*Chem. Soc. A*, 48, 876; 1885).

2. *Trans. North Engl. Inst. Mining and Mech. Eng.*, 50, [2], 23 (1901). (*J. Soc. Chem. Ind.*, 20, 789; 1901).

3. *J. Soc. Chem. Ind.*, 21, 242 (1902).

4. *J. Soc. Chem. Ind.*, 27, 147 (1908).

per cent of extract. Bedson called attention to the fact that in the first six coals the percentage of volatile matter is not far removed from the percentage of pyridine extract. After commenting upon this fact he decided that it would be dangerous to base any definite conclusions upon this fact alone. Although those coals were "gas" coals and similar in character, frequently substances identical in chemical constitution, give marked differences in physical properties. So that in coal work any problem may not be considered from the purely statistical standpoint alone.

Through Bedson, Blair¹ became interested in the proximate constituents of coal. Blair extracted coal from the Busley Seam of the Botley Colliery. After freeing the extract from pyridine, it was extracted with solvents in the following order: petroleum ether, ethyl ether, absolute alcohol and chloroform. Only a scanty description of these compounds was given. A complete account of the results was reserved for another communication.

Professor Lewes² in 1912, in a series of lectures on coal carbonization has reviewed some of the work upon the extraction of coal with pyridine. He himself had done some work but the results with the exception of one analysis were not given. He states that in all probability the coking property of a poor coking coal can be entirely removed by pyridine and that such will not be the case with a strong coking coal, for the reason that in the latter case some of the resinic bodies resist the solvent action of pyridine. Lewes in his criticism of pyridine as a solvent, points out the following facts as objections to it. He has worked on some coals that gave a higher percentage of volatile matter after extraction than they did before. He gives some results of Andersen and Henderson and claims that they show the same property. However, the results which he compared were not apparently considered by the authors as comparable. Professor Lewes states that the increase in volatile matter in the residue over the original volatile matter in the coal can only be taken to mean that pyridine has formed an additional product in some way. However, Clark and Wheeler, as well as Wahl, in work to be mentioned later, found that the residue did not increase as to the percentage of volatile matter. It was noted that in some cases when an ultimate analysis was made of the extract, the amount of nitrogen was greater than it was in the coal, in spite of the fact that the extract had been carefully washed free from pyridine. Furthermore, the two most successful solvents, according to Lewes, were aniline and pyridine,—both organic bases. The same effect is produced

1. J. Soc. Chem. Ind., 27, 150 (1908).

2. Progressive Age, 29, 1030 (1911).

upon coal by these solvents as with NaOH, i. e., a non-coking residue remains. Sodium hydroxide is known to saponify resinic substances, and it is probable that pyridine and aniline form a compound with some of the resin, which is soluble in an excess of the solvent. Of course, this would be shown in the high nitrogen content of the extract.

Lewes gives the following conclusions: "These considerations seem to make it clear that the resin constituents condition the coking of coal during destructive distillation, and that they are of at least two kinds—the one easily oxidizable, soluble in pyridine and saponifiable by alkalis, and which on weathering is oxidized into a humus body with the evolution of water and carbon dioxide, and is responsible for the heating of coal in storage; the other class non-oxidizable, not saponified by alkalis, and forming with pyridine a compound insoluble in excess of the reagent, and this class may be the hydrocarbons from decomposed resins, as the residue in which they are present yields rich liquid hydrocarbons, as tar and pitch, but not rich in gas".

A. Wahl¹ in 1912, extracted a number of coals with pyridine. Volatile matter determinations were made before and after the extraction. The percentage of volatile matter in the coal was from 13 to 55 per cent and after extraction, the volatile matter in the residue was slightly lower, in some cases as much as 5 per cent. The amount of material extracted was from 6 to 26 per cent. The coke which was produced from the residue was harder and more compact than that of the coal. The material dissolved by the pyridine was an amorphous brown solid, which produced a voluminous coke. The ultimate analyses of coal and pyridine extract were given in some cases, but these showed scarcely any difference in composition.

In 1912, Frazer and Hoffman² published a paper on "The Constituents of Coal Soluble in Phenol". They tried the effect of a number of reagents and organic solvents on coal and found that pyridine, aniline and phenol removed the largest amount of soluble material. The published report was concerned with those constituents of coal soluble in phenol. Illinois coal from Franklin County was used in the extraction. The amount extracted was 10.87 per cent, calculated on a moisture and ash free basis. After freeing the extract from phenol by distillation an analysis gave ash 2.16 per cent. In the light of the present investigation this amount of ash in the extract seems to be abnormally high. By the use of sodium hydroxide and organic solvents such as ether, acetic acid, methyl alcohol, acetone, benzene and petroleum ether, they were able to separate the extract into certain

1. Comtes rend., 154, 1094 (1912).
2. Technical Paper 5, Bureau of Mines.

portions, which when distilled in vacuo, gave varying physical properties. Analyses of these substances were made. The authors concluded that in the absence of evidence to the contrary coal substance soluble in phenol was present as such in the coal itself. They also believed that some of the substances isolated were approximately pure compounds, but this could not be proven absolutely on account of the small amount of material which they had at their disposal. They intend to follow the investigation using pyridine as a solvent.

Pictet and Ramseyer,¹ in 1913, extracted large quantities of washed coal from Montrambert (Loire) with benzene. From 248 kg. coal, almost 244 grams of almost black, quite fluid oil were obtained. They distilled this at various temperatures and pressures and worked on the different fractions. From their studies they think that coal contains among other constituents polymerized hydroaromatic hydrocarbons.

In 1913, Clark and Wheeler² in a paper on the "Volatile Constituents of Coal" described some of their work on the extraction of coal with phenol. They contend that coal is composed of two different types of bodies, which possess different degrees of ease of decomposition. They called the two constituents, the "hydrogen-yielding" and the "paraffin-yielding", respectively. However, they did not mean to say that above a certain temperature, one type of body decomposes and that below that temperature it will not decompose. From their experiments on coal they concluded that because of an increase in the amount of hydrogen evolved between 750° and 800°, that there was a hydrogen-yielding constituent in coal. They did not mean to designate by a hydrogen-yielding constituent one which gave all hydrogen, or by a paraffin-yielding constituent one that gave all paraffin, but one which gave a predominance of hydrogen or paraffin. It was also shown by them that the temperature of the destructive distillation influenced to a large extent the kind of gas given off. Clark and Wheeler extracted coal with pyridine and then the pyridine extract was itself extracted with chloroform. It was noted from the ultimate analysis that the increase in the amount of nitrogen in the residue and extract was 2.75 per cent, showing that nitrogen was being added from some source. Data were given for the analysis of gases which were produced by the destructive distillation of the pyridine extract, pyridine residue, chloroform extract and chloroform residue, and for dehydrated cellulose. The gas which was produced from the pyridine extract contained a larger amount of methane than the gas from the residue. The results

1. Arch. sci. phys. nat., 34, 234 (Chem. Abs. 7, 1496; 1913).

2. J. Chem. Soc., 103, 1704 (1913).

in the case of the present investigation did not show this difference. From the results obtained they did not hesitate to identify the pyridine residue as a degradation product of cellulose but they were doubtful if all of the pyridine extract was resinous in character. However, by extracting the pyridine soluble material with chloroform, they thought they were able to get out material consisting almost entirely of resinous matter. Detailed methods of extraction and of destructive distillation were given.

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- Bulletin No. 1.* Tests of Reinforced Concrete Beams, by Arthur N. Talbot. 1904. *None available.*
- Circular No. 1.* High Speed Tool Steels, by L. P. Breckenridge. 1905. *None available.*
- Bulletin No. 2.* Tests of High-Speed Tool Steels on Cast Iron, by L. P. Breckenridge and Henry B. Dirks. 1905. *None available.*
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- Circular No. 3.* Fuel Tests with Illinois Coal (Compiled from tests made by the Technologic Branch of the U. S. G. S., at the St. Louis, Mo., Fuel Testing Plant, 1904-1907), by L. P. Breckenridge and Paul Diserens. 1909. *Thirty cents.*
- Bulletin No. 3.* The Engineering Experiment Station of the University of Illinois, by L. P. Breckenridge. 1906. *None available.*
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