STATE OF ILLINOIS
HENRY HORNER, *Governor*
DEPARTMENT OF REGISTRATION AND EDUCATION
JOHN J. HALLIHAN, *Director*
DIVISION OF THE
STATE GEOLOGICAL SURVEY
M. M. LEIGHTON, *Chief*
URBANA

BULLETIN No. 64

Coke From Illinois Coals

GILBERT THIESSEN
ASSOCIATE CHEMIST IN CHARGE OF FUEL CHEMISTRY

WITH THE COLLABORATION OF
WALTER H. VOSKUIL, MINERAL ECONOMIST, AND PAUL E. GROTTS,
ASSISTANT IN GEOCHEMISTRY

CONTRIBUTION OF
THE SECTION OF GEOCHEMISTRY
FRANK H. REED, *Chief Chemist*

PRINTED BY AUTHORITY OF THE STATE OF ILLINOIS

URBANA, ILLINOIS
1937

STATE OF ILLINOIS
Hon. Henry Horner, *Governor*

DEPARTMENT OF REGISTRATION AND EDUCATION
Hon. John J. Hallihan, *Director*
Springfield

BOARD OF
NATURAL RESOURCES AND CONSERVATION
Hon. John J. Hallihan, *Chairman*

Edson S. Bastin, Ph.D., *Geology*
William A. Noyes, Ph.D., LL.D., Chem.D., D.Sc., *Chemistry*
John W. Alvord, C.E., *Engineering*
William Trelease, D.Sc., LL.D., *Biology*
Henry C. Cowles, Ph.D., D.Sc., *Forestry*
Arthur Cutts Willard, D.Engr., LL.D., *President of the University of Illinois*

STATE GEOLOGICAL SURVEY DIVISION
Urbana
M. M. Leighton, Ph.D., *Chief*
Enid Townley, M.S., *Assistant to the Chief*

GEOLOGICAL RESOURCES

Coal
G. H. Cady, Ph.D., *Senior Geologist*
L. C. McCabe, Ph.D.
James M. Schopf, Ph.D.
Earle F. Taylor, M.S.
Charles C. Boley, B.S.

Non-Fuels
J. E. Lamar, B.S.
H. B. Willman, Ph.D.

Oil and Gas
A. H. Bell, Ph.D.
G. V. Cohee, Ph.D.
Frederick Squires, B.S.
James L. Carlton, B.S.

Areal and Engineering Geology
George E. Ekblaw, Ph.D.
Victor N. Fischer, M.S.

Subsurface Geology
L. E. Workman, M.S.
J. Norman Payne, M.A.
E. A. Atherton, Ph.D.
Donald G. Sutton, M.S.

Stratigraphy and Paleontology
J. Marvin Weller, Ph.D.
W. A. Newton, M.S.

Petrography
Ralph E. Grim, Ph.D.
Richards A. Rowland, Geol. E.

Physics
R. J. Piersol, Ph.D.
M. C. Watson, Ph.D.
Donald O. Holland, M.S.

GEOCHEMISTRY

Frank H. Reed, Ph.D., *Chief Chemist*
W. F. Bradley, Ph.D.
G. C. Finger, M.S.
Mary C. Neill, M.S.

Fuels
G. R. Yohe, Ph.D.
P. E. Grotts, B.S.

Non-Fuels
J. S. Machin, Ph.D.
F. V. Tooley, M.S.

Analytical
O. W. Rees, Ph.D.
Norman H. Nachtrieb, B.S.
George W. Land, B. Ed.
P. W. Henline, B.S.

MINERAL ECONOMICS

W. H. Voskuil, Ph.D. *Mineral Economist*
Grace N. Oliver, A.B.

EDUCATIONAL EXTENSION

Don L. Carroll, B.S.

PUBLICATIONS AND RECORDS

George E. Ekblaw, Ph.D.
Dorothy Rose, B.S.
Alma R. Sweeny, A.B.
Meredith M. Calkins

Consultants: *Ceramics*, Cullen Warner Parmelee, M.S., D.Sc., University of Illinois; *Pleistocene Invertebrate Paleontology*, Frank Collins Baker, B.S., University of Illinois.

Topographic Mapping in cooperation with the United States Geological Survey.

2
(32725)
Sept. 1, 1937

Contents

PART I—INTRODUCTION

PART II—THE COKE MARKET AND COMPETITIVE FUELS IN THE ILLINOIS COAL MARKET AREA

PART III—TECHNOLOGY OF COKE MANUFACTURE

PART IV—EXPERIMENTAL INVESTIGATIONS OF THE PRODUCTION OF COKE FROM ILLINOIS COALS

PART V—CONCLUSIONS AND RECOMMENDATIONS

APPENDICES

Illustrations

FIGURE PAGE

APPENDIX

Tables

APPENDIX A

Coke from Illinois Coals

GILBERT THIESSEN

WITH THE COLLABORATION OF
W. H. VOSKUIL AND PAUL E. GROTTS

Part 1 — Introduction

CHAPTER I

PURPOSES OF THE REPORT

THE manufacture of by-product and beehive coke has furnished a market for almost fifteen per cent of the bituminous coal mined in the last decade in the United States. This quantity approaches that used for locomotive fuel and is considerably larger than that used by public electric utilities[1] (Table 1 and fig. 1). Illinois coals, however, have found a very small outlet in this major market provided by the coke manufacturing industry. The Illinois State Geological Survey in its program of work designed to assist the mineral industries of Illinois undertook certain studies of Illinois coals in an effort to determine whether and in what way these coals could better participate in the coking coal market. This bulletin reports the results of certain of those investigations.

The purposes for which this report has been prepared are:

(1) To consider the possible market for coke in the Illinois coal market area and the competitive position of coke from Illinois coal in that market.

(2) To review critically the present state of the art of coking Illinois coals, and to show the position occupied by Illinois coals in the various schemes which have been set up to evaluate coals for coke-making purposes.

(3) To present the results of experimental investigations designed to furnish fundamental data concerning the coking properties of Illinois coals, the effects of impurities, the influence of test conditions, and the results of blending coals.

(4) To review the operation of a commercial installation for coking Illinois coals.

(5) To present a summary concerning the coke which may be made from Illinois coals, its qualities and possible markets.

---

[1] U. S. Bureau of Mines, Minerals Yearbook 1936.

TABLE 1.—QUANTITIES OF BITUMINOUS COAL CONSUMED IN THE UNITED STATES FOR BEEHIVE AND BY-PRODUCT COKE MANUFACTURE, BY THE PUBLIC ELECTRIC UTILITIES, AND TOTAL CONSUMPTION, 1917-1936 [a]

Year	Beehive coke		By-product coke		Public electric utilities		Total consumption thousands of tons
	Thousands of tons	Per cent of total consumption	Thousands of tons	Per cent of total consumption	Thousands of tons	Per cent of total consumption	
1917	52,247	9.9	31,506	6.0	33,500	6.3	529,409
1918	48,160	9.1	36,868	6.9	34,500	6.5	530,593
1919	29,730	6.2	35,857	74.4	35,100	7.3	481,658
1920	31,986	6.3	44,205	8.7	37,124	7.3	508,595
1921	8,475	2.2	28,713	7.3	31,585	8.1	391,849
1922	13,286	3.1	41,053	9.6	34,179	8.0	426,915
1923	30,084	5.8	54,276	10.5	38,966	7.5	518,993
1924	15,914	3.3	49,061	10.1	37,556	7.8	484,004
1925	17,423	3.5	57,110	11.4	40,222	8.1	499,193
1926	19,225	3.6	63,647	12.0	41,311	7.8	532,581
1927	11,208	2.2	63,240	12.7	41,888	8.4	499,801
1928	7,018	1.4	70,166	14.1	41,350	8.3	498,828
1929	10,028	1.9	76,759	14.8	44,937	8.6	519,555
1930	4,284	.9	65,521	14.4	42,898	9.4	454,990
1931	1,767	.5	46,846	12.6	38,735	10.4	371,869
1932	1,030	.3	30,887	10.1	30,290	9.9	306,917
1933	1,461	.5	38,681	12.0	30,575	9.5	321,748
1934	1,635	.5	44,343	12.8	33,555	9.7	347,043
1935	1,469	.4	49,046	13.9	34,807	9.7	360,292
1936	2,893	.7	63,976	15.3	41,973	10.1	417,203

[a] Data from U. S. Bureau of Mines, Mineral Resources 1930, Part II, page 676, Table 61; Minerals Yearbook 1936, page 550, Table 4; and Bituminous Coal Tables 1935-36.

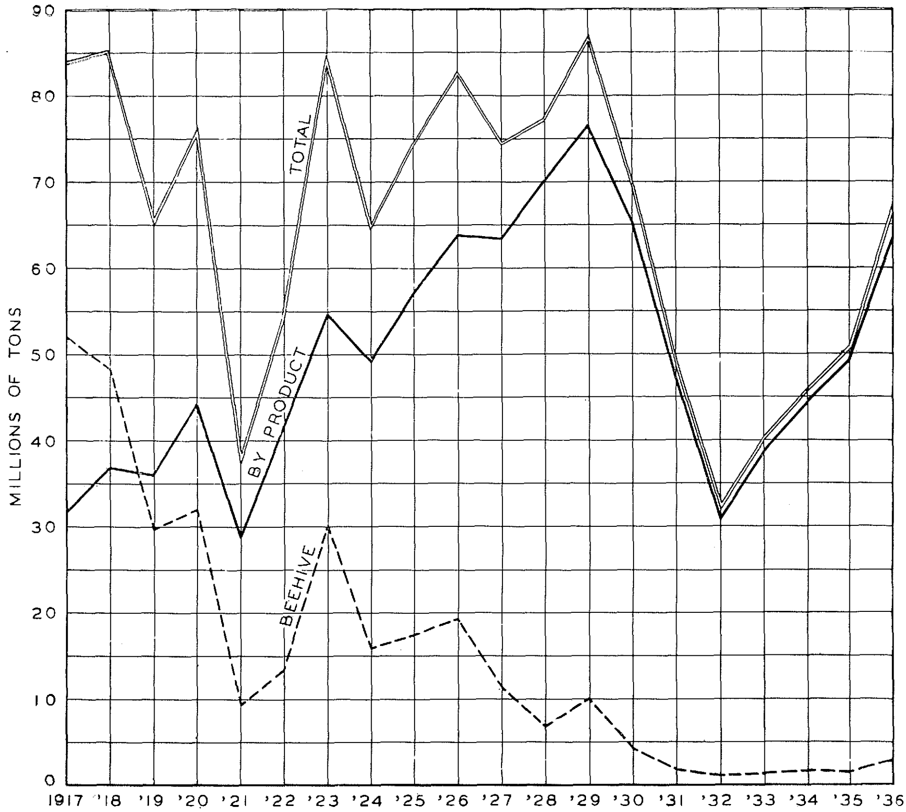


Fig. 1.—Bituminous Coal Consumed for Beehive and By-product Coke Manufacture in the United States, 1917-1936.

SCOPE OF THE REPORT

This report presents a discussion of the economic position of the coke industry in Illinois, the place occupied by Illinois coal in the various schemes for classifying coal for coking purposes, a brief review of the art of coke manufacture, a history of coke manufacture in Illinois, and a statistical review of the industry, as well as the results of a variety of investigations undertaken in the laboratories of the Illinois Geological Survey.

This report is not to be considered as a complete monograph on the manufacture of coke nor as containing all that is desirable or necessary to know about the coking of Illinois coals. Some of the material has been previously published as individual articles.[2]

---

[2] Thiessen, G., Behavior of sulfur during coal carbonization: Ind. and Eng. Chem. vol. 27, pp. 473-478, April, 1935.

Thiessen, G., Coke from Illinois coal, temperature conditions in sole-flue ovens: Ind. and Eng. Chem. vol. 29, pp. 506-513, May, 1937.

ACKNOWLEDGMENTS

The author is under great obligation to his associates on the staff of the Illinois State Geological Survey and to many others who have assisted in or made possible much of the work which this bulletin reports, and he wishes to take this opportunity to express his appreciation.

To Dr. M. M. Leighton, Chief of the Illinois State Geological Survey, the author expresses his sincere appreciation for the active interest and support received during the entire progress of this undertaking. This work was one of the early projects for which he sought the establishment of the Survey's new laboratories.

This bulletin is a report of work carried on in the Geochemical Section of the State Geological Survey, of which Dr. Frank H. Reed is Chief Chemist and to whom the author is especially indebted for guidance and suggestions during the course of the work and during the preparation of the manuscript. Much assistance and many helpful suggestions during the course of the work have come from other associates on the Survey staff, especially Dr. G. H. Cady, Dr. O. W. Rees, Dr. Walter Voskuil, Dr. L. C. McCabe, and during the earlier stages of the work from Dr. C. F. Fryling. The samples used in the tests were collected by members of the Coal Division of the Geological Resources Section of the Survey.

Much credit is due my assistant, Mr. P. E. Grotts, for his part in carrying out much of the experimental work and in preparing many of the illustrations. Messrs. J. W. Teter, A. L. Ryan, B. Bilder, and J. E. Pentecost assisted in this work during three months in 1934 under a C.W.A. project. Most of the analytical work was performed under the direction of Dr. O. W. Rees by J. W. Robinson, Jr., Carl Westerberg, C. E. Imhoff, L. D. McVicker, G. C. Finger, and W. F. Bradley. Mr. L. D. Vaughan assisted with the photographic work and in many other ways. Mr. A. W. Gotstein was of much assistance in the design and construction of equipment.

The author wishes to express his thanks to Mr. E. T. Johnston, Chief Chemist of the Central Laboratories of the American Steel and Wire Company at Joliet, Illinois, for aid furnished in standardizing our procedure in the use of the United States Steel Corporation dry distillation test; to Dr. A. C. Fieldner, to Mr. J. D. Davis, and Mr. W. A. Selvig of the United States Bureau of Mines for their assistance in connection with the use of many of the test procedures; and to Mr. M. D. Curran, president of the Radiant Fuel Corporation for permission to make and publish the tests on the Knowles oven installation at West Frankfort, Illinois, and to Mr. George Curran, plant superintendent, Mr. F. E. Dodge, chemical engineer, and Mr. C. E. Case for their assistance during the tests.

Many others have assisted with suggestions, criticisms, supplying samples and information for which the author expresses his thanks.

Part II — The Coke Market and Competitive Fuels in the Illinois Coal Market Area

CHAPTER II—THE ECONOMIC BASIS OF A COKING INDUSTRY IN ILLINOIS

By Walter H. Voskuil

GEOGRAPHIC LIMITS OF THE ILLINOIS COAL MARKET AREA

The Illinois coal market area is defined as the territory included in the states of Illinois, Missouri, Iowa, Minnesota, and Wisconsin, the eastern cities of Kansas and Nebraska, and a small section of the Dakotas. The boundaries of this so defined "Illinois coal market area" are determined by competition from other coal fields and other forms of fuel. Within the area so described 90 per cent of Illinois coal is marketed. In the southwest part of the area fuel oil and natural gas dominate the market almost to the exclusion of coal. The westward movement of Illinois coal in Kansas, Nebraska, and the Dakotas is met by an eastward flow of coals from Colorado, Wyoming, and Montana. In the lake shore counties of Minnesota and Wisconsin the market is dominated by Appalachian coals, cheaply carried over the Great Lakes and reaching the ports of Lake Michigan and the head of Lake Superior. Illinois coals, however, are marketed to a considerable extent in the southern and western sections of these two lake states. Only small quantities of Illinois coals are shipped east of the Illinois line. This territory is occupied almost entirely by the neighboring coal fields of Indiana and the Appalachian coal fields of Ohio, West Virginia, eastern Kentucky, and Pennsylvania.

Within this market area itself there are also consumed Appalachian coals, and also fuel oil and natural gas, but bituminous coal is by far the most important source of fuel for industry, railroads, and domestic heating. For further information on this point, the reader is referred to Bulletin No. 63 of this Survey.

NATURE OF THE FUEL MARKET

Fuels consumed in the Illinois coal market area find an outlet in the manufacturing industries, smelting, railroad fuel, public utilities, mining and quarrying, domestic heating, and miscellaneous smaller uses. The miscellaneous uses include the heating of large non-factory buildings, such as hotels, apartments, stores, offices, theatres, garages, and also a number of non-domestic uses, such as waterworks, threshing engines, construction work, power laundries, grist mills, and small factories. Altogether the consumption of fuel in this area in a year of high productivity is approximately 102,000,000 tons of coal and its equivalent in liquid and gaseous fuels.

The consumption of coke in this area in 1929 was approximately 7,000,000 tons of which 5,000,000 tons were consumed in the manufacturing industries and 2,000,000 tons were used as domestic fuel. Public utilities, mining, and quarrying use practically no coke. In the manufacturing industries, the principal outlet for coke, possibly 80 per cent is used in the blast furnace industry and the remainder is distributed among industries such as commercial baking plants, cast iron pipe manufacture, chemical manufactures, foundry and machine shop products, gas manufacture, smelting and refining of nonferrous metals, and in such metal products industries as engine manufacture, stoves and ranges, steam fittings and plumbers supplies, and motor vehicles. Clay products, lime manufacture, meat packing, and paint and varnish making also use coke to some extent as a fuel.

IMMEDIATE OUTLOOK FOR A COKE INDUSTRY BASED UPON ILLINOIS COAL

As has been pointed out, the two largest outlets for coke in the Illinois coal market area are for metallurgical purposes and for domestic heating.

The metallurgical market.—Coke for metallurgical use is obtained mainly from eastern coke ovens and from coking coal imported from Kentucky, Pennsylvania, and West Virginia. In 1935, for example, a total of 2,500,000 tons were shipped into the state for coke-making purposes. There is, at present no Illinois coal used in the manufacture of metallurgical coke, although in times past it has been employed to some extent.

The domestic market.—The principal hope for an outlet of coke made from Illinois coal lies in an expansion of the domestic market rather than the metallurgical. The annual domestic fuel requirements in the Illinois coal market area are approximately the equivalent of 35,000,000 tons of coal divided among the various fuels as follows:

	Tons of coal or its equivalent
Pennsylvania anthracite (1936)	1,000,000
Fuel briquets (1935)	575,000
By-product coke (1929)[a]	2,078,000
Oil heating (1935) (19,006,000 bbls.)	4,751,200
Natural gas (1935) (67,770,000,000 cu. ft.)	2,591,000
Bituminous coal, estimated	24,000,000
Total	35,095,000

[a] The latest year for which detailed coke distribution data are given.

The use of coke for domestic heating increased consistently until it reached a peak in 1933 and declined in 1934 and 1935. The Bureau of Mines offers the following explanations for the recent decline:[1]

(1) Greater industrial activity and a greater use of coke by industrial users with the result that less coke reaches the domestic market

(2) Increasing installations of domestic and commercial stokers with cheaper coal replacing some coke

(3) Increasing competition of natural gas

(4) Declining prices of anthracite

(5) Increasing prices of raw coal resulting in higher prices of coke

(6) Growing competition from cheap trucked coal, both anthracite and bituminous.

In the Illinois coal market area, coke occupies a position between the higher priced fuels and the prepared sizes of bituminous coal. For those consumers who wish to obtain the advantages of cleanliness, smokelessness, and convenience, coke must compete to some extent with natural gas, anthracite, heating oil, and fuel briquets. For those consumers who are interested mainly in a low priced fuel, coke will find difficulty in competing with prepared sizes of bituminous coal. The market outlet probably must be sought among those who desire smokelessness but will not go to the extent of using gas, heating oil, or anthracite.

Shipments of anthracite are confined principally to Chicago and the lake shore counties of Wisconsin, and in 1936 aggregated 1,000,000 tons. The importance of anthracite as a domestic fuel in this area appears to be declining. The increased use of natural gas, fuel oil, and coke are tending to displace anthracite.

Fuel oil.—Since 1926, the year for which data are first available, the consumption of fuel oil in the Illinois coal market area has shown an almost constant upward trend. If the consumption of fuel oil is broken down into uses for domestic heating and for all other purposes, an interesting trend is observable. Fuel oil for manufacturing, bunkers, smelting and refining, and transportation has shown no increase since 1926. On the other hand, the use of fuel oil for heating purposes has shown a rapid upward trend and will probably continue

---

[1] Young, W. H., Bennit, H. L., and Plein, L. N., Coke and by-products: U. S. Bureau of Mines Minerals Yearbook, pp. 581-624, 1936.

to increase in the immediate future, as indicated by the continued increased rate of sales of oil burners. Consumption of heating oils in the Illinois coal market area in 1935 was approximately 19,000,000 barrels or an equivalent of about 4,750,000 tons of coal.

There are, however, certain factors that must be considered in evaluating fuel oil as a domestic fuel in the future. The consumption of fuel oil in this area in 1935 was practically equal to the quantity of furnace oils of the heating grade obtained in the process of refining in the Illinois-Indiana refining area. If the rate of oil burner installation continues it is not unlikely that the demand upon the oil refining industry for heating oils will become so heavy that price rises will result which will tend to discourage further increases or the oil industry will be compelled to meet this demand by increasing runs to stills over the present high level. A substantial rise in fuel oil consumption will necessitate readjustments in the refining processes in which the percentage of gasoline recovered is decreased in winter months and the percentage of fuel oil recovered is decreased in the summer months to correspond with the seasonal demands for these two products of oil refining.

Natural gas.—The supply of natural gas available to cities in the Illinois coal market area by pipe lines from Texas and Kansas to Chicago, to cities in central Illinois and to cities in Iowa, eastern Nebraska, and southern Missouri, appears to be sufficiently large to last for several decades. Consumption of natural gas in the Illinois coal market area is now equivalent to approximately 2,600,000 tons of coal and will probably increase somewhat in the future. The limiting factor in the use of natural gas is not a shortage of supply but price. Another factor limiting the use of natural gas is the fact that its distribution is limited practically to cities. The high cost of distribution will prevent its use in small communities except near newly discovered sources.

Briquets.—At the present time the manufacture of briquets in the Illinois coal market area is governed largely by the amount of screenings which result from the transportation of coal over the Great Lakes with resultant degradation and breakage. In order to dispose of these fine sizes, briquetting plants are in operation in St. Paul, Minnesota; Omaha, Nebraska; Superior, Sheboygan, Ashland and Milwaukee, Wisconsin. There are also some briquetting plants in North Dakota using lignite in an attempt to improve this fuel. Consumption of briquets is at present less than a million tons in this area.

The total consumption of anthracite, natural gas, fuel oil, and briquets is approximately equivalent to 8,000,000 tons of coal. An estimated 70 per cent of the domestic fuel consumption consists of bituminous coal from various sources. Coal used in the domestic fuel market is obtained from Illinois and from several eastern fields, the principal contributing states being West Virginia and eastern Kentucky. There are certain factors which favor the importation of large quantities of eastern coal for use in the domestic fuel market, such as higher heating value per ton, cleanliness, and lower ash content, for which the domestic consumer is willing to pay a higher price. The possibility of coke from Illinois coal in replacing these fuels depends upon the ability of a coking industry to supply a fuel which gives the advantages of present solid fuels at a lower price than is now paid for eastern coals or to supply a fuel of improved characteristics over such bituminous coal, for which the householder is willing to pay an added price.

FACTORS FAVORABLE TO THE USE OF ILLINOIS COAL FOR MAKING COKE

Experience in the coking of Illinois coals indicates that is is economically and technically difficult to produce metallurgical coke, but that a coke which is suitable and desirable for domestic heating and for other nonmetallurgical uses is feasible.

Experimental investigation indicates that coking-coal resources of Illinois are confined principally to central and southern Illinois. The two largest domestic fuel markets in the Illinois coal market area and near to these producing fields are St. Louis and Chicago. At present about 85 per cent of the coal shipped into the St. Louis district is obtained from the southern Illinois fields. This market area consumes probably about the equivalent of 1,800,000 to 2,000,000 tons of fuel for domestic heating.

The Chicago market consumes approximately an equivalent of 5 to 6 million tons for domestic heating. This area appears to be less favorable for a market for coke from southern Illinois coal because of a higher freight rate than to the St. Louis market and because of greater competition from fuels in the higher price brackets. Natural gas for domestic use is available by pipeline from the Panhandle of Texas, heating oils in large quantities are available from nearby refineries, and eastern coals are available by lake transportation.

At present, coal is available in southern Illinois at a cost of $1.60 to $1.75 per ton at the mine and screenings can sometimes be obtained at a lower price. The freight rate on coal, or coke, from the producing fields to the two principal markets and to the smaller cities in the Illinois coal market area give to the southern Illinois coal district an advantage in delivered cost of fuel over coke or high rank coal from the more distant eastern coal fields. The most difficult problem of developing a coke industry is that of establishing in the minds of the domestic consumer the advantages of coke as a domestic fuel. Coke has burning characteristics that are different from either anthracite or bituminous coal. To introduce coke into a market area, it is necessary to carry on a campaign of educational advertising.

Because coke possesses certain properties, it requires special handling as a domestic fuel. It is distinctly more bulky than bituminous coal or anthracite. Some of the difficulties encountered in marketing coke for domestic heating have resulted from attempts to use the larger sizes in furnaces that were too small.

The chemical characteristics of coke usually do not concern the domestic uses unless the coke employed has unusually high ash or high sulfur content. Sulfur produces corrosive products of combustion which are highly objectionable. In producing coke care should be taken with regard both to the ash and sulfur content.

DISPOSAL OF BY-PRODUCTS

In the development of a coking industry, the problem of the disposal of coke-oven gas, tar, light oil, and ammonia must be considered. The principal outlet for gas other than that used as fuel by the plant is in public utility distribution, and as fuel in industry. In southern Illinois, gas could be disposed of in public utility distribution systems for domestic cooking and for such local industries as commercial bakeries, laundries, restaurants, etc. A coke oven located in the St. Louis area would meet competition from natural gas.

The marketing of gas from a coke oven plant presents a peculiar problem. It must be used within a few miles from the plant at places which can be reached by gas mains or pumping lines. Moreover, it is very bulky and, therefore, cannot be stored for more than a few hours. In these respects gas differs from other products of the coke oven which not only can be stored but also can be shipped considerable distances before they are used.

The disposal of tar, light oils, and ammonia presents no particular problems in this area since they can be shipped over greater distances than the gas.

DEVELOPMENT OF NEW MARKETS FOR COKE AND ITS BY-PRODUCTS

Although coke for domestic heating offers the largest immediate outlet for coke it is conceivable that in the future other markets may be developed. Particular reference should be made to the production of motor fuel in the event of a declining crude oil supply.

For the production of liquid fuels, two processes are now in progress of development, the Bergius process for the liquefaction of coal by hydrogenation of a mixture of pulverized coal and tar under high pressures and temperatures with the aid of a catalyst, and the Fischer-Tropsch process for the gasification of coke and the synthesis of liquid hydrocarbons from gas and hydrogen. The latter process seems to be gaining favor and may provide an outlet for coke to meet future motor fuel requirements.

CHAPTER III—THE PRODUCTION, DISPOSAL, AND USES OF COKE

BY GILBERT THIESSEN AND PAUL E. GROTTS

PRODUCTION AND DISTRIBUTION BY USES OF COKE IN THE UNITED STATES

The production and the quantitative and relative distribution by uses of by-product and beehive coke in the United States since 1918 are shown in Tables 2 and 3 and graphically in figure 2. The continued rise of the domestic coke market and its importance as an outlet for coke during periods of depression are obvious.

PRODUCTION OF COKE IN THE ILLINOIS COAL MARKET AREA

Much coke has been and is being produced in the Illinois coal market area.[1] Most of this coke is or has been made for metallurgical purposes at plants located close to Great Lakes navigation, or as a by-product in the manufacture of city gas from coals originating outside of the Eastern Interior basin of Illinois, Indiana, and western Kentucky. At the lake steel plants a combination of price and quality factors practically excludes Illinois coals at present. The amount of gas-works coke produced is much smaller than the amount produced primarily for metallurgical purposes, but a greater proportion of it goes into the domestic market. Table 4 gives the number, type, and location of the coke ovens in operation in the Illinois coal market area in 1925, 1930, and 1935. The number and location of the ovens are diagrammatically shown in figures 3, 4, and 5. The origin of the coal coked in these ovens in 1935 as closely as can be estimated from Bureau of Mines statistics[2] is shown in Table 5.

---

[1] Voskuil, W. H., The competitive position of Illinois coal in the Illinois coal market area: Illinois State Geol. Survey Bull. 63, pp. 10-11, 1936.

[2] Young, W. H., Bennit, H. L., and Plein, L. N., Coke and by-products: U. S. Bureau of Mines Minerals Yearbook, pp. 581-624, 1936.

TABLE 2.—BY-PRODUCT AND BEEHIVE COKE PRODUCED AND SOLD OR USED BY THE PRODUCER IN THE UNITED STATES, 1918-1936
(Million net tons, exclusive of screenings and breeze)

Year	Coke produced	Used by producer in blast furnace etc.	Coke sold					References[d]
			Furnace[a]	Foundry	Other industrial[b]	Domestic[b]	Total[c]	
1918	56.48	22.64	27.36	3.86	2.75		33.97	M.R. Pt. II, 1930, pp. 496, 532.
1919	44.18	20.79	19.25	2.83	3.03		25.11	
1920	51.35	26.05	19.24	3.52	2.55		25.32	
1921	25.29	13.97	6.55	1.75	1.74		10.04	
1922	37.12	22.18	9.04	2.77	4.05		15.85	
1923	56.98	29.64	18.14	3.60	2.28	2.73	26.75	
1924	44.27	25.60	10.87	2.92	1.82	2.95	18.55	
1925	51.27	30.26	11.75	2.99	2.65	4.48	21.86	
1926	56.87	32.91	12.11	3.33	2.63	5.44	23.51	
1927	51.09	31.59	8.73	2.75	2.59	4.81	18.88	
1928	52.81	34.31	7.64	2.71	1.76	6.33	18.45	
1929	59.88	37.97	8.81	2.89	2.33	7.51	21.55	
1930	47.97	29.64	4.89	2.13	2.03	8.03	17.07	
1931	33.48	18.31	2.30	1.36	1.84	8.50	14.00	M.R. Pt. II, 1931, pp. 394-6.
1932	21.79	9.60	.93	1.05	1.30	9.63	12.91	M.Y. 1932-33, Appendix, pp. 271-3.
1933	27.59	13.44	1.38	1.00	1.84	10.49	14.71	M.Y. 1935, pp. 673-4.
1934	31.82	15.31	2.19	1.26	1.89	10.52	15.86	M.Y. 1935, pp. 673-4.
1935	35.14	20.11	2.47	1.48	2.24	9.43	15.62	M.Y. 1936, pp. 607-8.
1936	46.28	28.05	4.27	1.92	2.03	10.02	18.25	M.Y. 1937, preliminary statistics.

---

[a] Includes coke sold to affiliated corporations and merchant sales for furnace use.
[b] Domestic coke and other industrial coke were not separately reported prior to 1923.
[c] Difference between "coke produced" and sum of coke "used by producer" and "total sold" is accounted for by changes in stocks.
[d] "M. R." refers to United States Bureau of Mines Minerals Resources of the United States for the year indicated. "M. Y." refers to the United States Bureau of Mines Minerals Yearbook for the year indicated.

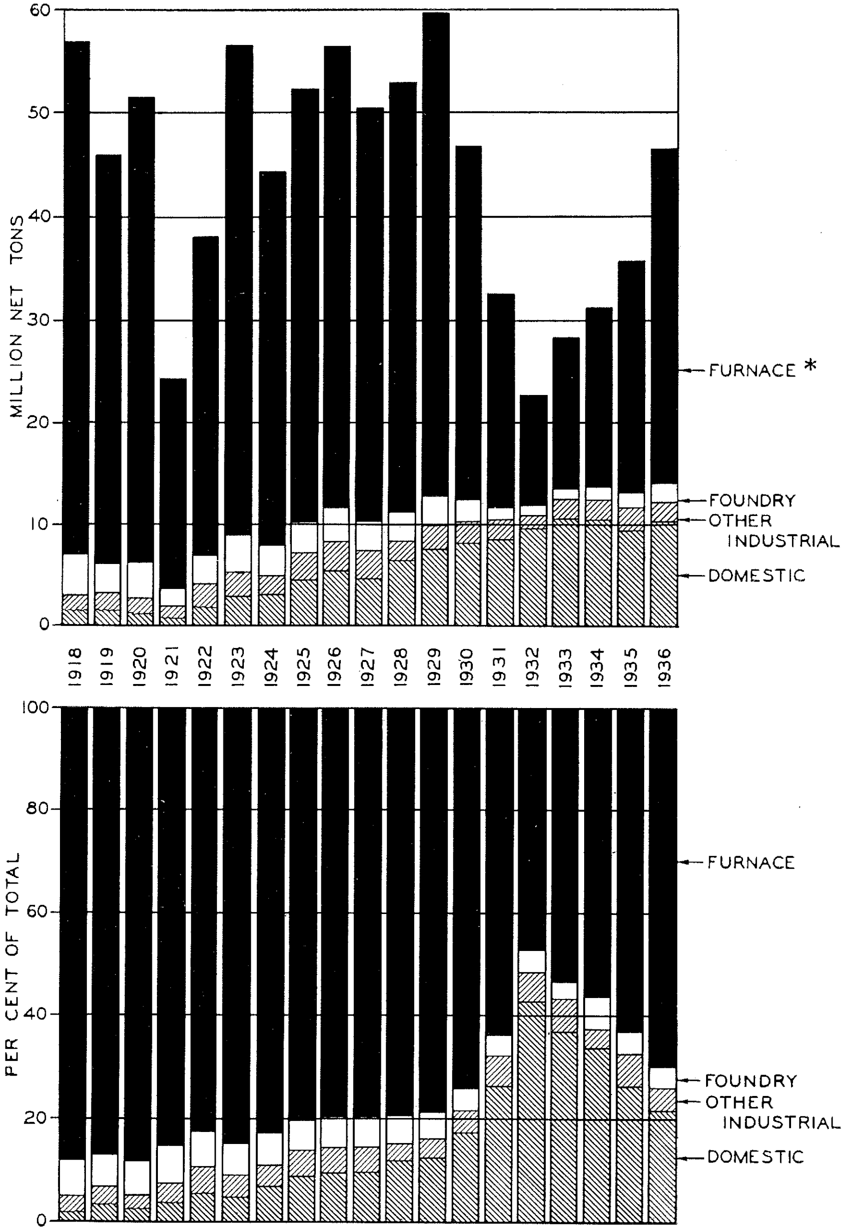


Fig. 2.—Distribution of By-product and Beehive Coke Sold for Furnace, Foundry, other Industrial, and Domestic Use, 1918-1936.

*Includes coke used by producer in blast furnace.

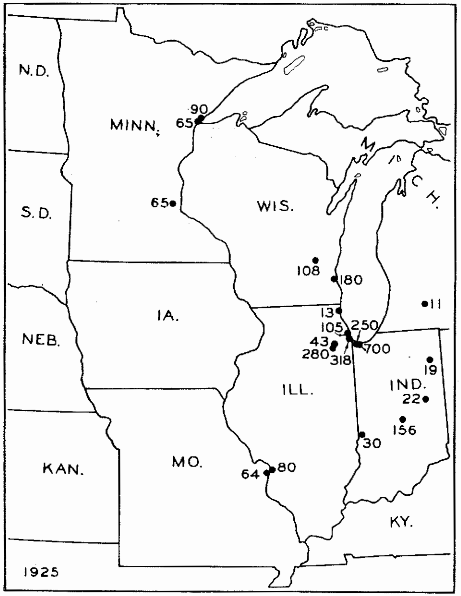


Fig. 3

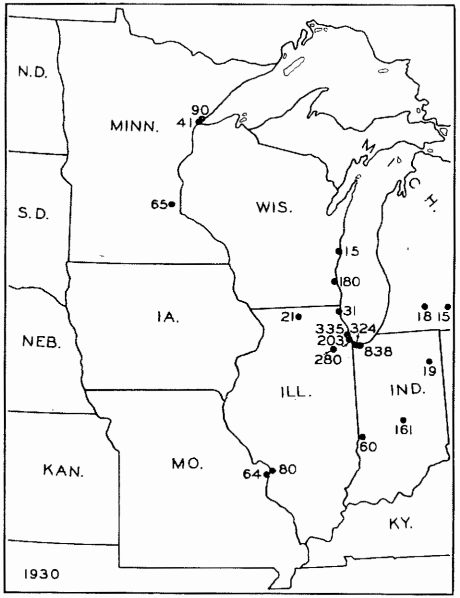


Fig. 4

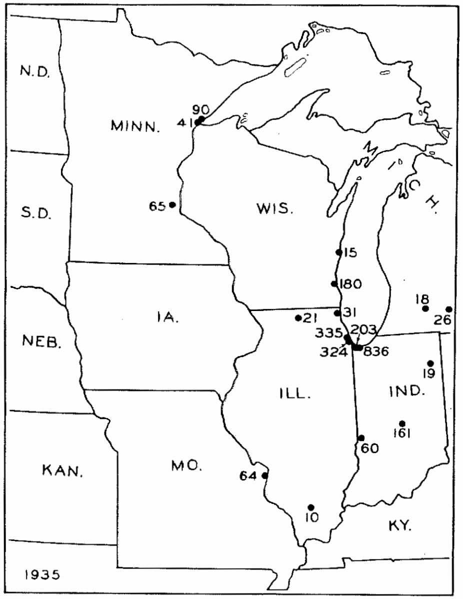


Fig. 5

Figs. 3, 4, 5.—Number and Location of By-product Coke Ovens in the Illinois Coal Market Area in 1925, 1930 and 1935.

TABLE 3.—PERCENTAGE DISTRIBUTION OF BY-PRODUCT AND BEEHIVE COKE SOLD OR USED BY THE PRODUCER IN THE UNITED STATES, 1918-1936

Year	Used by producer in blast furnace, etc.	Sold			
		Furnace[a]	Foundry	Other industrial[b]	Domestic[b]
1918........	39.99	48.33	6.82	4.86	
1919........	45.29	41.94	6.17	6.60	
1920........	50.71	37.45	6.85	4.96	
1921........	58.18	27.28	7.29	7.25	
1922........	58.32	23.77	7.28	10.65	
1923........	52.56	32.17	6.38	4.04	4.84
1924........	57.98	24.62	6.61	4.12	6.68
1925........	58.06	22.54	5.74	5.08	8.60
1926........	58.33	21.46	5.90	4.66	9.64
1927........	62.59	17.30	5.45	5.13	9.53
1928........	65.03	14.48	5.14	3.34	12.00
1929........	63.79	14.80	4.86	3.91	12.62
1930........	63.46	10.47	4.56	4.35	17.19
1931........	56.67	7.12	4.21	5.69	26.31
1932........	42.65	4.13	4.66	5.78	42.78
1933........	47.74	4.90	3.55	6.54	37.26
1934........	49.12	7.03	4.04	6.06	33.75
1935........	56.28	6.91	4.14	6.27	26.39
1936........	60.58	9.22	4.15	4.38	21.64

[a] Includes coke sold to affiliated corporations and merchant sales for furnace use.
[b] Domestic coke and other industrial coke were not separately reported prior to 1923.

TABLE 4.—SUMMARY OF OVENS IN OPERATION IN

City	Company	Date installed	1925
	Illinois		
Chicago	Chicago By-Product Coke Co. (M)	1921–22	100 Koppers 5 Koppers-Becker
Chicago	Interlake Iron Corporation[b] (M)	1925	110 Koppers
Chicago	Interlake Iron Corporation[b] (M)	1905–15	120 Semet-Solvay
South Chicago	Wisconsin Steel Co. (F)[c]	1919	88 Wilputte
South Chicago	Wisconsin Steel Co. (F)[c]	1929	
South Chicago	Youngstown Sheet and Tube Co. (F)	1928	
Lockport	Coal Products Mfg. Co. (M)	1912	35 Koppers
Lockport	Coal Products Mfg. Co. (M)	1914	18 Wilputte
Joliet	Illinois Steel Co. (F)	1908–09	280 Koppers
Rockford	Rockford Gas, Light and Coke Co. (M)	1929	
Granite City	St. Louis Coke and Iron Co. (F)	1921	80 Roberts
Waukegan	North Shore Gas Co. (M)	1912	13 Semet-Solvay
Waukegan	North Shore Coke and Chemical Co. (M)	1928	
West Frankfort	Radiant Fuel Corporation (M)	1933–34	
	Total		849
	Plants		7
	Indiana		
Fort Wayne	Northern Indiana Gas & Electric Co. (M)	1925	19 Koppers
Gary	Illinois Steel Co. (F)		560 Koppers
Gary	Illinois Steel Co. (F)	1911–31	140 Wilputte
Gary	Illinois Steel Co. (F)		
Indiana Harbor	Inland Steel Co. (F)	1913–29	130 Koppers
Indiana Harbor	Youngstown Sheet & Tube Co. (F)	1919	120 Semet-Solvay
Indianapolis	Citizens Gas Co. (M)	1909–25	75 United Otto
Indianapolis	Citizens Gas Co. (M)	1914	41 Semet-Solvay
Indianapolis	Citizens Gas Co. (M)	1918–29	40 Wilputte
Muncie	Central Indiana Gas Co. (M)	1912	22 Klonne
Terre Haute	Indiana Coke and Gas Co. (M)	1919	30 Koppers
Terre Haute	Indiana Consumers Gas & By-Product Co.	1919–26	
	Total		1177
	Plants		7
	Minnesota		
Duluth	Minnesota Steel Co. (F)	1915–16	90 Koppers
W. Duluth	Zenith Furnace Co. (F)	1904–16	65 United Otto
St. Paul	Minnesota By-Product Coke Co. (M)	1918	65 Koppers
W. Duluth	Interlake Iron Corp. (F)	1928	
	Total		220
	Plants		3
	Wisconsin		
Mayville	Youngstown Sheet & Tube Co. (F)	1914–17	108 Otto Hoffman
Milwaukee	Milwaukee Coke & Gas Co. (M)	1901–06	80 Semet-Solvay
Milwaukee	Milwaukee Coke & Gas Co (M)	1921–22	100 Koppers
Sheboygan	Wisconsin Public Service Co. (M)	1929	
	Total		288
	Plants		2
	Missouri		
St. Louis	Laclede Gas Light Co. (M)	1915	56 Koppers
St. Louis	Laclede Gas Light Co. (M)	1921	8 Piette
	Total		64
	Plants		1

(Footnotes on p. 30.)

ILLINOIS COAL MARKET AREA IN 1925, 1930, 1935[a]

1930	1935	Source of additional data
	Illinois	
100 Koppers	100 Koppers	
5 Koppers-Becker	5 Koppers-Becker	Proc. A.G.A. 15: 773 (1933)
110 Koppers	110 Koppers	
120 Semet-Solvay	120 Semet-Solvay	
88 Wilputte	88 Wilputte	
45 Koppers-Becker	45 Koppers-Becker	Proc. A.G.A. 12: 1097 (1930)
70 Koppers-Becker	70 Koppers-Becker	Proc. A.G.A. 12: 510 (1930)
...	...	
...	...	
280 Koppers	...	Private communication[d]
21 Koppers	21 Koppers	Proc. A.G.A. 11: 1030 (1929)
80 Roberts	...	Private communication
...	...	
31 Koppers-Becker	31 Koppers-Becker	Proc. A.G.A. 15: 778 (1933)
...	10 Knowles	16 additional ovens completed at the end of 1935
950	600	
8	7	
	Indiana	
19 Koppers	19 Koppers	
560 Koppers	420 Koppers	Proc. A.G.A. 12: 1098, 1105 (1930);
140 Wilputte	140 Wilputte	13: 943 (1931); also private communication
138 Becker	276 Becker	
204 Koppers	204 Koppers	Proc. A.G.A. 12: 1097 (1930)
120 Semet-Solvay	120 Semet-Solvay	
...	...	
41 Semet-Solvay	41 Semet-Solvay	
120 Wilputte	120 Wilputte	
...	...	
...	...	
60 Koppers	60 Koppers	
1402	1400	
6	6	
	Minnesota	
90 Koppers	90 Koppers	Proc. A.G.A. 11: 1026 (1929
...	...	
65 Koppers	65 Koppers	
41 Koppers	41 Koppers	
196	196	
3	3	
	Wisconsin	
...	...	
80 Semet-Solvay	80 Semet-Solvay	
100 Koppers	100 Koppers	
15 Koppers	15 Koppers	Proc. A.G.A. 11: 1027 (1929); 12: 1096 (1930)
195	195	
2	2	
	Missouri	
56 Koppers	56 Koppers	
8 Piette	8 Piette	
64	64	
1	1	

TABLE 4—

City	Company	Date in-stalled	1925
	Michigan		
Battle Creek...	Battle Creek Gas Co. (M)....................	1924	11 Koppers.........
Jackson........	Consumers Power Co. (M)....................	1925–30	....
	Total.............	....	11
	Plants............	....	1

---

[a] Based on statistics in U. S. Bureau of Mines Mineral Resources Reports and Minerals Yearbooks and additional data.

[b] Listed under the name "By Products Coke Corporation" at S. Chicago in 1925. Included with S. Chicago ovens on 1925 map.

[c] Listed under name "International Harvester Co." in 1925.

[d] Several batteries of these ovens have again been put into use in 1936.

[e] Only western half of State considered.

(F) "Furnace plant" or plants associated with iron furnaces and steel works.

(M) "Non Furnace Plants" or other plants including merchant plants, plants associated with industries other than iron and steel and plants supplying gas under contract for city use.

CONCLUDED

1930	1935	Source of additional data
	Michigan	
18 Koppers.	18 Koppers	
15 Koppers.	26 Koppers.	Proc. A.G.A. 12: 1098 (1930); 13: 943 (1931)
33	44	
2	2	

TABLE 5.—QUANTITY AND SOURCE OF COAL USED IN THE MANUFACTURE OF BY-PRODUCT COKE IN THE ILLINOIS COAL MARKET AREA IN 1925, 1930, AND 1935 [a]
(In tons)

Source of coal	State in which coal was used				
	Illinois	Indiana	Minnesota	Wisconsin[b]	Missouri[b]
			1925		
Illinois	325,095	....	....		
Indiana	53	....	....		
Kentucky	1,925,000	1,923,486	204,810		
Pennsylvania	35,939	156,834	246,708		
Virginia	41,451	....	....		
West Virginia	1,971,753	4,786,851	340,474		
	4,299,293	6,867,171	791,992	1,156,500	257,000
			1930		
Illinois	565,362	....	....		
Kentucky	1,312,004	2,569,228	247,507		
Pennsylvania	503,591	382,166	395,903		
West Virginia	2,727,597	3,948,759	294,328		
	5,108,554	6,900,153	937,738	954,300	313,500
			1935		
Kentucky	625,194	2,181,970	189,029		
Pennsylvania	466,967	150,637	256,144		
Virginia	....	226,484	....		
West Virginia	1,382,938	2,677,234	184,577		
	2,475,099	5,236,325	629,750	941,600	309,100

[a] Data from U. S. Bureau of Mines Mineral Resources and Minerals Yearbook.
[b] Estimated from concealed figures on basis of number of ovens.

The distribution by uses of the coke produced in Illinois, Indiana, and Minnesota in the years 1925, 1930, and 1935 are presented in Table 6. The figures for coke produced in other states in the Illinois coal market area are not available. The distribution of coke produced in Illinois for each year since 1918 is presented in Table 7 and figure 6. In figure 6 the difference between total production and total sales is accounted for largely by the coke consumed by the producer and by changes in stocks. As is the case for the United States as a whole, the domestic market has been an important outlet for coke in Illinois, especially in the years 1932-35 when the amount of coke used by the producer fell to very low figures. The statistics presented here do not include the coal coked at the installation of 26 Knowles type sole-flue ovens at West Frankfort, Illinois, the only plant at which coke was made for commercial sale from Illinois coal in 1936. This plant and its operation is described later in this report for the reason that it is a new type of oven and is now operating with Illinois coal.

Up to the summer of 1935 two batteries of 40 ovens each of Roberts type ovens were operated by the St. Louis Coke and Chemical Co. at Granite City. These ovens at times operated on Illinois coal alone, at other times on mixtures containing substantial proportions of Illinois coals, and at other times on out-of-state coal alone. The abandonment of this installation leaves the Radiant Fuel Corporation's plant as the only plant making coke entirely from Illinois coal.

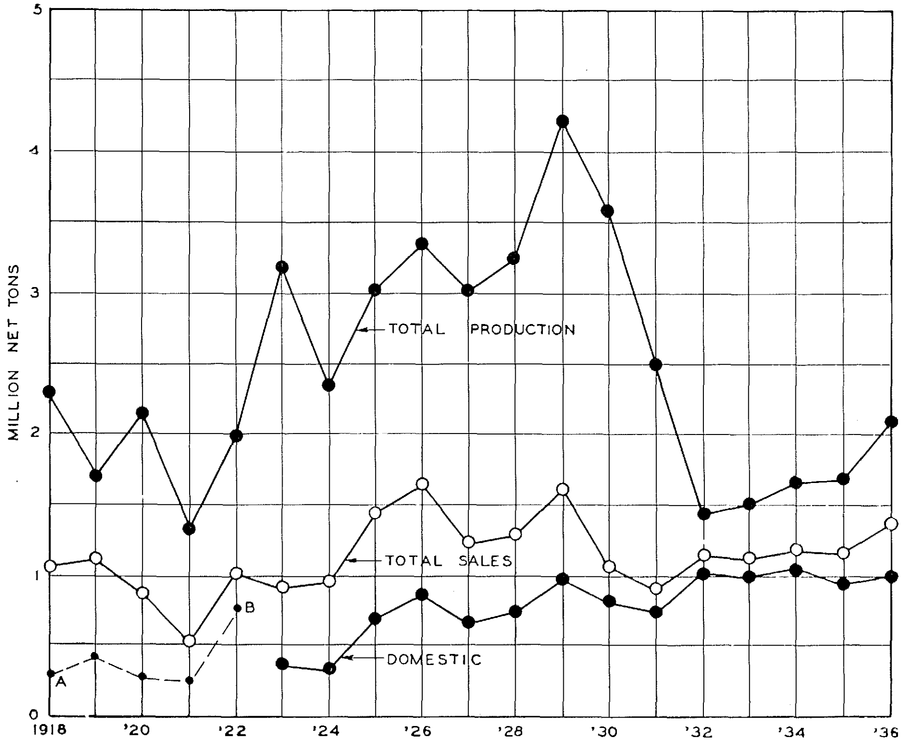


Fig. 6.—Coke Produced, Domestic and Total Sales for Illinois, 1918-1936. A-B represents domestic and miscellaneous industrial coke.

TABLE 6.—DISPOSAL OF BY-PRODUCT COKE PRODUCED BY STATES IN THE ILLINOIS COAL MARKET AREA IN 1925, 1930, AND 1935
(Exclusive of screenings and breeze)

	Total coke produced	Coke used by producer and other affiliated works	Coke sold				
			Furnace	Foundry	Other industrial	Domestic	Total
			Illinois				
1925[a] net tons	3,011,497	1,828,991	227,882	209,403	318,962	689,402	1,445,649
per cent	100	60.7	7.6	6.9	10.6	22.9	48.0
1930[b] net tons	3,576,577	2,110,318	(d)	(d)	79,756	813,271	1,058,771
per cent	100	59.0	....	....	2.2	22.7	29.6
1935[c] net tons	1,668,523	612,704	....	150,912	58,316	945,084	1,154,312
per cent	100	36.7	....	9.0	3.5	56.6	69.1
			Indiana				
1925[a] net tons	5,141,940	4,539,369	3,101	232,457	17,298	370,651	623,507
per cent	100	88.3	0.1	4.5	0.3	7.2	12.1
1930[b] net tons	4,984,620	4,321,384	....	164,197	34,187	362,238	560,622
per cent	100	86.7	....	3.3	0.7	7.3	11.3
1935[c] net tons	3,768,480	3,270,640	6,997	84,770	23,721	373,677	489,165
per cent	100	86.8	0.2	2.3	0.6	9.9	13.0
			Minnesota				
1925[a] net tons	518,355	288,520	....	68,085	2,977	341,414	412,476
per cent	100	55.6	....	13.1	0.6	65.9	79.6
1930[b] net tons	641,205	256,716	(d)	(d)	(d)	(d)	365,151
per cent	100	40.0	....	....	....	....	57.0
1935[c] net tons	430,082	14,347	....	187	43,318	372,708	416,213
per cent	100	3.2	....	0.0	10.1	86.7	96.8

(a) Mineral Resources, 1925, Pt. II, p. 570-71.
(b) Mineral Resources, 1930, Pt. II, p. 527.
(c) Minerals Yearbook, 1936, p. 607.
(d) Not reported separately.

TABLE 7.—DISTRIBUTION BY USES OF BY-PRODUCT COKE PRODUCED AND SOLD OR USED BY THE PRODUCER IN ILLINOIS, IN 1918-1936
(In net tons, exclusive of screenings and breeze)

Year	Coke produced	Used by producer in blast furnace, etc.	Coke sold					References
			Furnace[a]	Foundry	Other industrial[b]	Domestic[b]	Total	
1918.......	2,285,610	1,236,571	525,538	247,520	298,674		1,071,732	M.R. 1920 Pt. II, pp. 403-4
1919.......	1,703,903	780,860	396,410	298,720	424,355		1,119,485	
1920.......	2,136,793	1,259,168	387,790	216,537	267,755		872,082	
1921.......	1,322,178	663,074	200,755	78,686	250,224		529,665	M.R. 1922, Pt. II, p. 731
1922.......	1,982,906	1,071,098	106,571	156,576	753,724		1,016,871	
1923.......	3,187,168	1,907,479	67,371	180,620	323,481	351,422	922,894	M.R. 1924, Pt. II, pp. 622-5
1924.......	2,355,474	1,327,820	136,897	163,208	333,513	329,789	963,407	
1925.......	3,011,497	1,828,991	227,882	209,403	318,962	689,402	1,445,649	M.R. 1925, Pt. II, p. 571
1926.......	3,336,962	1,717,860	302,578	240,068	235,804	853,866	1,632,316	M.R. 1926, Pt. II, p. 625
1927.......	3,013,940	1,653,043	([c])	([c])	348,175	654,463	1,236,140	M.R. 1927, Pt. II, p. 629
1928.......	3,240,964	1,888,276	274,278	([c])	([c])	739,408	1,285,638	M.R. 1928, Pt. II, p. 746
1929.......	4,204,116	2,564,257	([c])	([c])	247,016	981,418	1,611,445	M.R. 1929, Pt. II, p. 611
1930.......	3,576,577	2,110,318	([c])	([c])	79,756	813,271	1,058,771	M.R. 1930, Pt. II, p. 527
1931.......	2,478,984	1,464,319	([c])	([c])	76,091	743,234	911,301	M.R. 1931, Pt. II, p. 394
1932.......	1,428,334	513,257	([c])	([c])	47,117	1,004,512	1,135,857	M.Y. 1932-33, Appendix, p. 271
1933.......	1.501,020	291,884	([c])	67,794	53,308	998,310	1,119,412	M.Y. 1934, p. 610
1934.......	1,649,907	425,371	391	94,678	57,545	1,025,128	1,177,742	M.Y. 1935, p. 673
1935.......	1,668,523	612,704	([c])	150,912	58,316	945,084	1,154,312	M.Y. 1936, p. 607
1936.......	2,082,516	846,925	56,938	238,090	63,451	999,890	1,358,369	M.Y. 1937 Preliminary Statistics

[a] Includes coke sold to affiliated corporations and merchant sales for furnace use.
[b] Domestic coke and other industrial coke were not separately reported prior to 1923.
[c] No data.

CHAPTER IV—THE USE OF COKE AS A DOMESTIC FUEL

By Gilbert Thiessen

The domestic fuel market for coke from Illinois coal is especially important for these coals because as coke they enter the market as a high quality, economical, smokeless fuel in a region quite far removed from sources of anthracite and smokeless coal. Although coke from Illinois coals may in time play an important part in the iron and steel industry, certain of its characteristics make its present competition difficult with coke from coals of the eastern fields.

Coke is definitely a smokeless fuel. The seriousness of atmospheric pollution, especially by coal smoke, is recognized. It has been estimated that the cost of smoke pollution in the larger cities in the soft coal fuel districts amounts to from 10 to 20 dollars or even more per capita per year.[1] The general public is gradually being educated to the damaging effects of atmospheric pollution and already demands that something be done to minimize it in our larger cities in the soft coal districts. Any such abatment action will place a premium on smokeless fuels. Smoke abatement movements and ordinances will be more popular if a smokeless fuel is available which permits the average householder to heat his home as economically and conveniently as with bituminous coal. Gas, oil, and stoker fired coal also provide domestic heat without smoke but results of various tests have indicated that none of these provide domestic heat as cheaply as does coke.

United States Bureau of Mines tests have shown that coke is more efficiently utilized in domestic heating furnaces than is bituminous coal.[2] These tests in six different steam and hot water boilers showed that coke was burned as efficiently as anthracite and that the efficiencies obtained in burning Pittsburgh coal were approximately 10 per cent lower and Illinois coal 20 per cent lower than those obtained in burning coke or anthracite.

Fuel tests with domestic heating boilers by the University of Illinois Engineering Experiment Station have given similar results with respect to efficiencies of use.[3] Averaged values for results of similar tests have been selected and presented as examples by Kreisinger and Fieldner.[4] These values are presented again in Table 8 for easy reference and comparison.

---

[1] Kreisinger, H., and Fieldner, A. C., Why and how coke should be used for domestic heating: U. S. Bureau of Mines Tech. Paper No. 242, p. 6, 1919.

[2] Kreisinger, H., Blizard, J., Jarrett, H. W., and McKitterick, J. J., Comparative tests of by-product coke and other fuels for house-heating boilers: U. S. Bureau of Mines Tech. Paper No. 315, 1923.

[3] Snodgrass, J. M., Fuel tests with house-heating boilers: Univ. of Illinois Eng. Exp. Sta. Bull. 31, 1909.

[4] Kreisinger, H., and Fieldner, A. C., Why and how coke should be used for domestic heating: U. S. Bureau of Mines Tech. Paper No. 242, p. 17, 1919.

TABLE 8.—RESULTS OF TESTS OF VARIOUS FUELS IN HOUSE-HEATING BOILERS
Tests Made at the Engineering Experiment Station of the University of Illinois.[a]

Fuel	Efficiency of boiler and furnace per cent	Fuel fired at each firing pounds	Average interval between firing hours	Rated capacity developed per cent
By-product coke	61.63	75	3.71	64.46
Gas house coke	56.22	75	3.21	65.48
Anthracite	51.93	75	2.47	66.00
Illinois coal, Williamson Co.	48.00	75	2.92	64.04
Pocahontas coal	46.51	75	3.30	63.88

[a] Data from Snodgrass, J. M., Fuel tests with house-heating boilers: Univ. of Illinois Eng. Exp. Sta. Bull. 31, p. 82-83, Table 12, 1919. (Referred to erroneously by Kreisinger and Fieldner as U. of I. Eng. Exp. Station Bull. 19.)

Recent tests in Columbus, Ohio, showed that the over-all cost of heating homes under conditions prevailing there at the time the tests were carried out was lowest with coke.[5] The results are summarized in Table 9.

TABLE 9.—RELATIVE TOTAL COSTS OF HEATING TYPICAL COLUMBUS, OHIO, HOUSE WITH VARIOUS FUELS

Fuel	Unit fuel price	Relative cost per cent
Bituminous coal, hand fired	$6.75/T	100
Semi-bituminous coal, hand fired	$7.00/T	97
Coke, hand fired	$8.35/T	95
Bituminous coal, stoker fired	$5.00/T	119
Natural gas	$0.55/M.C.F.	151
Oil	$0.065/Gal.	177

The average efficiencies with which these fuels were used in the furnaces tested in actual use in house heating are given in Table 10.

In addition to being less smoky and more economical, coke has the advantage of being cleaner than coal. The walls in houses heated with coke remain cleaner than do those in which bituminous coal is burned. It is also possible to maintain a more even temperature if the coke fire is correctly regulated. Less frequent firing is required and in general operation of the heating plant is less troublesome. Another important but more intangible benefit is the resulting more efficient use of coal, effecting thereby some conservation of the nation's fuel resources.

---

[5] Sherman, R. A., and Cross, R. C., Efficiencies and costs of various fuels in domestic heating: Bituminous Coal Research, Inc. Technical Report No. 3, 1936.

TABLE 10.—AVERAGE EFFICIENCIES OF USE OF VARIOUS FUELS IN HOUSE-HEATING FURNACES, COLUMBUS, OHIO

Fuel	Furnace or boiler	Test efficiency per cent	Average efficiency per cent	Assumed seasonal efficiency per cent
Hand fired bituminous coal..........	Warm air	54	..	..
	Steam	46	..	..
	Warm air	41	47	45
Hand fired semi-bituminous coal......	Warm air	54	..	..
	Warm air	52	53	50
Hand fired coke....................	Warm air	76	..	..
	Warm air	75	75.5	70
Stoker fired bituminous coal.........	Steam	69	..	..
	Hot water	56	..	..
	Warm air	55	60	55
Natural gas........................	Warm air[a]	69	..	70
	Steam[a]	76	..	75
	Warm air[b]	70	72	70
Oil................................	Warm air	65	..	..
	Steam	51	58	60

[a] Designed for gas.
[b] Converted to gas.

MANAGEMENT OF A COKE FIRE

Combustion conditions in a fuel bed of coke differ from those in a bed of high-volatile-content coal and a different firing procedure must be adopted. Many of the complaints against coke as a domestic fuel and much of the difficulty in attempts to introduce it as such arise from improper firing conditions which are usually aggravated by equipment in poor condition. The use of coke under improper conditions usually results in a greater amount of trouble than is experienced when coal is incorrectly fired. Coke is more sensitive to the conditions under which it is fired than is coal. Directions for burning coke in the domestic plant have been presented in two publications by the United States Bureau of Mines.[6] The directions are similar, and those given by Nicholls and Landry are quoted here for convenient reference.

---

[6] Nicholls, P., and Landry, B. A., Coke as a domestic fuel: U. S. Bureau of Mines, Report of Inv. 2980, 1929.

Kreisinger, H., and Fieldner, A. C., Why and how coke should be used for domestic heating: U. S. Bureau of Mines Tech. Paper 242, 1919.

"1. Before starting to use coke, inspect the furnace and see that it is in good repair and that the dampers will close; see that the ash pit is tight and has no leaks which will admit air in spite of its damper being closed.

"2. Clean the flues and smoke pipe.

"3. Use the size of coke recommended for the heater. There is no standard scale for coke sizes, and producers differ in their methods of grading, both on size and on the number of sizes carried; consequently, recommendations have varied.

"4. Carry a deep fuel bed, and fire as large a charge as the furnace will hold; do not carry a shallow bed but fill the fire pot to its capacity, even in mild weather.

"5. Do not poke the fire; be content with levelling it and remove the ashes by shaking the grates, leaving a layer of ash to protect the grates and to increase the resistance of the fuel bed.

"6. Open the dampers the minimum distance necessary to obtain the desired heat.

"Such instructions can only be very general and they leave a lot for the user to learn from experience if he is to get the most satisfactory results; his problem with coke is, however, no worse than with other fuels, except that the adjustment of the dampers may have to be finer. Success with coke does, however, call for more self-restraint than with other fuels; it is easier to obtain a fierce fire, and trouble is less likely to result if one exercises patience and gives the house time to warm up without making use of the full possibilities of the extra hot fire obtainable with coke."[7]

It is very desirable that the heating system be provided with automatic control devices. A room thermostat operating the dampers will greatly improve operating conditions. Further improvement is obtained by the addition of a high temperature limit control in the stack pipe to prevent the development of excessive temperature in the fuel bed, loss of heat in hot stack gases, overheating of the house, and the formation of clinkers. Reliable devices for such control are on the market.

---

[7] Nicholls, P., and Landry, B. A., Op. cit., p. 5.

Part III—Technology of Coke Manufacture

By Gilbert Thiessen

CHAPTER V—REASONS FOR THE MANUFACTURE OF COKE

Coal is coked to produce a fuel having certain desirable characteristics for use in metallurgical and industrial processes, to provide a smokeless domestic and industrial solid fuel, to produce gas and other distillation products, and to utilize smaller sizes of coal. The production of metallurgical coke was the main incentive for the development of the coke industry. In more recent years the demand for a smokeless domestic fuel, that is cheaper than oil, gas, and anthracite has furnished a steadily growing market for coke which has approached in magnitude the industrial and metallurgical market.

Bituminous coal is not very suitable for blast furnace and many other metallurgical and industrial uses because of the volatile matter it contains or because it becomes soft and semi-fluid when heated. Anthracite is not a competitor either because of price or because of its low reactivity, high ash content, or other characteristics. Although other methods for the reduction of iron ore have been proposed and some have been adopted, the modern iron and steel industry relies on coke and the by-products made in coke manufacture for the operation of its fundamental units, the blast furnace, the open hearth, and the cupola.

The rising price of anthracite has caused a considerable shift to the use of coke in the northeastern part of the United States where the use of smokeless fuel is compulsory in many localities. This change was not made without considerable effort on the part of coke producers who undertook elaborate educational programs. In other parts of the United States coke has come into use as smokeless fuel which is cheaper, if less convenient, than oil and gas.

The manufacture of illuminating gas was the reason for the first establishment of the coal carbonization industry. The carbonization of coal has continued to be the main source of manufactured gas. Formerly, practice was directed toward complete gasification of coal, usually in steps, with little attention paid to the quality of coke produced during the process, since this coke was not marketed. The growing domestic market for coke has caused a change in the manufactured gas industry. Recent practice is directed toward the production of gas and high quality coke in coke ovens, the ovens being heated either by combustion of a part of the coal-gas produced in the ovens or by combustion of water-gas made from a part of the coke breeze. The use of by-product coke ovens in the manufactured gas industry is especially noticeable on the eastern seaboard where the coke is sold in competition with anthracite to customers who have learned to appreciate it, or who are compelled by ordinances, to use smokeless fuel.

Because the coking process utilizes very small sized coal it offers a means of converting certain sizes of coal into a more valuable and more easily marketable fuel. However, because of the usual tendency of the mineral matter to concentrate in the naturally formed smaller sizes of coal, it is usually necessary that such coal be cleaned before being coked, if the ash content is to be kept within reasonable limits . The importance of small coal in providing a source of cheap fuel for coking depends upon conditions determining the local marketability of such coal.

CHAPTER VI—METHODS OF PRODUCING COKE

All processes for producing coke from coal are similar in that they provide means for heating coal in the absence of air to a temperature at least high enough to pass the coal through its plastic state and to set the plastic mass to cellular porous coke.

The first method of coking coal was to build specially arranged piles in which flues were built out of blocks of the coal itself. Heat for the coking process was furnished by combustion of part of the coal by air brought in through the flues. These piles were covered with earth or clay to conserve heat and coke.

This very primitive method of coke manufacture developed into the beehive process in which the coal is coked in a layer on the floor of a beehive-shaped oven by the heat provided from combustion of the volatile matter and part of the coke. The coking process is initiated by heat stored in the oven walls from the previous charge. Very desirable metallurgical coke has been and is being made in beehive ovens from suitable coals. At one time there was a strong prejudice among blast furnace superintendents in favor of beehive oven coke over by-product coke.

Coke for metallurgical purposes has been produced by the beehive oven or similar methods since the middle of the eighteenth century. For many years likewise, independently of the metallurgical industry, coal has been carbonized to obtain volatile products, such as illuminating gas, tar, and ammonia, with domestic coke often produced as a by-product. The manufactured gas industry carbonized coal in special types of retorts or ovens. The coke so obtained was frequently of poor quality. Development work on large chamber ovens in which good metallurgical coke could be produced, at the same time permitting recovery of by-products, dates from about 1850. The change to by-product coke ovens in the United States reached its full stride in the period 1915-1920, under the influence of the war-time demand for benzene and toluene. Today by-product coke ovens supply the basic demand for coke, while beehive ovens, because of their greater flexibility, supply the requirements in excess of production by operating by-product ovens.

The modern by-product coke oven in the slot form which has become almost universally adopted consists of a rectangular refractory brick chamber, about 40 feet long, 10 feet high and 18 inches wide. Heat is supplied by the combustion of gas in flues built into the side walls of the ovens. The ends are closed by doors which are removable to permit the coke to be pushed out. Coal is fed into the ovens through charging holes built into the tops of the ovens. The gas offtakes are located at the ends of the ovens and also are built into the top. These ovens are constructed in groups, or batteries, side by side, separated by sets of heating flues common to the adjoining ovens. Recuperators or regenerators for conserving heat by preheating the incoming air and fuel gas by means of the heat contained in the combustion gases coming from the heating flues are usually located beneath the ovens. A cut-away block model of one of the widely adopted constructions is shown in figure 7, and a view of an installation of such ovens is shown in figure 8. Rectangular coke ovens have been constructed in a wide, low form with the heating flues beneath the floor or sole of the oven on which the coal is coked. Such ovens, termed "sole-flue" ovens have been built in a variety of designs one of which is shown in cross section in figure 62 and an installation of such ovens in figure 61 (pp. 180, 182).

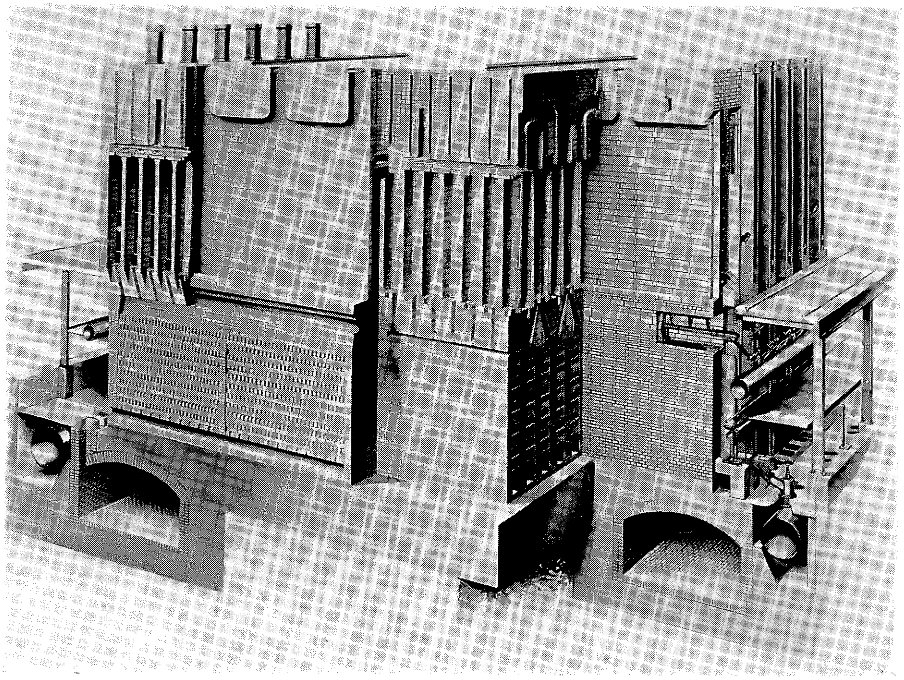


Fig. 7.—Cut-Away Block Model of Modern By-product Coke Oven.
Courtesy of Koppers Co.

Figure 9 shows the relative sizes and shapes of the more common types of chambers in which coal is carbonized at high temperatures to produce a coke substantially free from volatile matter. Commercial coke is now produced mainly in slot-type by-product ovens, beehive ovens, and (in Illinois) sole-flue ovens. Heat is supplied to the coal in the Knowles ovens mainly through the floor, in the beehive ovens largely from above, in the slot-type ovens from the sides, and in the gas retorts from the entire periphery. Gas offtakes are from the tops of the ovens or charges in all cases. The temperatures attained in the charges are in the range of 750°-1200°C. (1382°-2192°F.) in almost all cases. The temperatures attained at the completion of coking in beehive and slot-type by-product coke ovens in which most of the coke produced today is made is about 1000°C. (1832°F.)

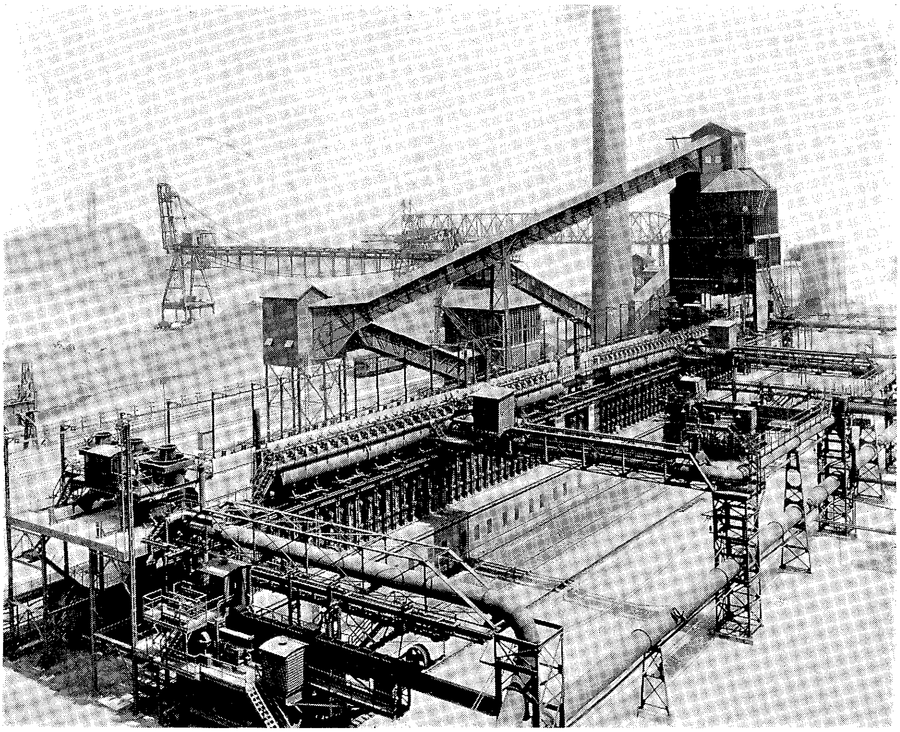
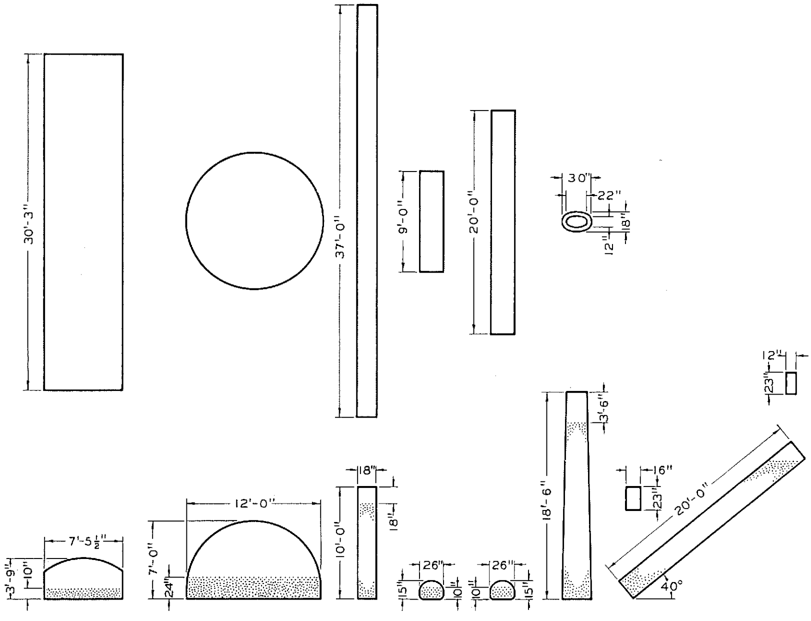


Fig. 8.—An Installation of By-product Coke Ovens at South Chicago.
Courtesy of Koppers Co.

HIGH- VERSUS LOW-TEMPERATURE CARBONIZATION

The high temperatures (about 1000°C. or 1832°F.) employed in by-product ovens subject the distillation products which first arise at temperatures up to about 550°C. (1022°F.) to rather severe decomposition or "cracking" and also decrease the ease with which the resulting coke may be ignited and kept lit at low rates of burning. The cracking which the tarry products first liberated from the coal undergo as they pass into zones of higher temperature on their way out of the oven results in a lower tar yield, a higher gas yield, and the formation of the compounds such as benzene, naphthalene, and phenols characteristic of high-temperature coke oven tars. This thermal alteration of the so-called primary decomposition products of coal has been considered objectionable for various reasons and much attention has been given to the problem of coking coal at temperatures only slightly higher than the temperature required to form solid coke from plastic coal. Reasons for this effort have been the desire to produce (1) more easily ignitable and combustible cokes which could be used as smokeless domestic fuel with greater convenience than could high-temperature coke, and (2) larger quantities of liquid products from which motor fuels could be



OVEN TYPE	KNOWLES BY-PRODUCT	BEEHIVE	SLOT TYPE BY-PROD	HORIZONTAL		VERTICAL	INCLINED
				STOP-END	THROUGH		
				GAS RETORTS			
COAL CHARGE	5 TONS	6 TONS	12 TONS	450 LBS.	1700 LBS.	1700 LBS.	1 TON

Fig. 9.—Relative Sizes of Coal Carbonizing Equipment.

prepared. The second reason has been given its greatest emphasis in those countries which have an insufficient domestic supply of petroleum. This has been especially true at times when a world petroleum shortage has seemed imminent and previous to the development of other processes for producing liquid fuel from coal.

The advantages for domestic use of a coke which is more easily combustible are well known. One of the main difficulties encountered in low-temperature carbonization systems is the low capacity due to the low temperature gradient permissible if the coke and tar are not to be overheated. Considerable attention is now being given to so-called medium-temperature carbonization processes which produce coke more easily combustible than high-temperature coke, yet which permit higher heat gradients to be used than can be used in low-temperature carbonization processes. Failure of true low-temperature coal tars to live up to their estimated commercial value and usefulness has been another point in favor of the medium-temperature processes which can produce a tar more like high-temperature coal tar. In spite of the large amount of money spent on the development of so-called "low-temperature carbonization" systems (estimated at 40 million dollars in the United States alone) very few plants are operating successfully today from either a technical or financial standpoint. Most of these which are operating have some form of subsidy, either direct or indirect, to assist their financial position.

CHAPTER VII—HISTORICAL SURVEY OF THE PRODUCTION OF COKE FROM ILLINOIS COALS

INTRODUCTION

Efforts at the production of coke from Illinois coals followed the general progress of the coke industry in the United States. Before 1900 attention was directed almost entirely towards the use of the beehive or similar ovens. With the introduction of the by-product coke oven in this country on a large scale after about 1907 and especially during the war time boom from 1915 to 1920, attention was directed towards the use of these ovens or to the development of such ovens for making metallurgical coke from Illinois coals. More recently effort has been directed towards the production of domestic fuel with recognition that the high ash and high sulfur content of many Illinois coals makes the production of metallurgical coke from them an uncertain proposition.

The development of ways of making domestic coke have followed several lines. At one time it was thought that low-temperature carbonization had great possibilities. The production of coke in relatively small, low capital cost units utilizing local screenings and supplying gas to nearby users is now receiving attention. A brief summary of the early work directed towards the production of coke from Illinois coals was prepared by Ovitz in 1917[1] under a cooperative agreement between the United States Bureau of Mines, the Illinois State Geological Survey, and the Department of Mining Engineering of the University of Illinois. This bulletin was published before any appreciable work had been done on the carbonization of Illinois coal in by-product coke ovens. Much of the early history of the manufacture of coke from Illinois coal is obscure.

USE OF BEEHIVE TYPE OVENS FOR COKING ILLINOIS COALS

Illinois coal is not well suited for coking in beehive coke ovens. Nevertheless a considerable quantity of beehive coke has been produced from Illinois coal, mainly in the period 1880-1900 (fig. 10). By 1910 beehive coke oven operation in Illinois had practically ceased. The largest operations were: (1) at Mount Carbon, near Murphysboro, Jackson County, where, according to Ovitz, from three to four hundred thousand tons of coke were produced; (2) at Equality, Gallatin County, where there was an installation of 24 ovens, 20 feet long,

---

[1] Ovitz, F. K., Coking of Illinois Coals: U. S. Bureau of Mines Bull. 138, 1917.

5 feet high, and 20 inches wide, perhaps not beehive ovens; and (3) at South Chicago where there was an installation of 24 beehive ovens fitted with flues in the floor and an arrangement for introducing hot or cold air over the charge to increase the oven temperature and improve control with a view to coking Illinois coals.[2]

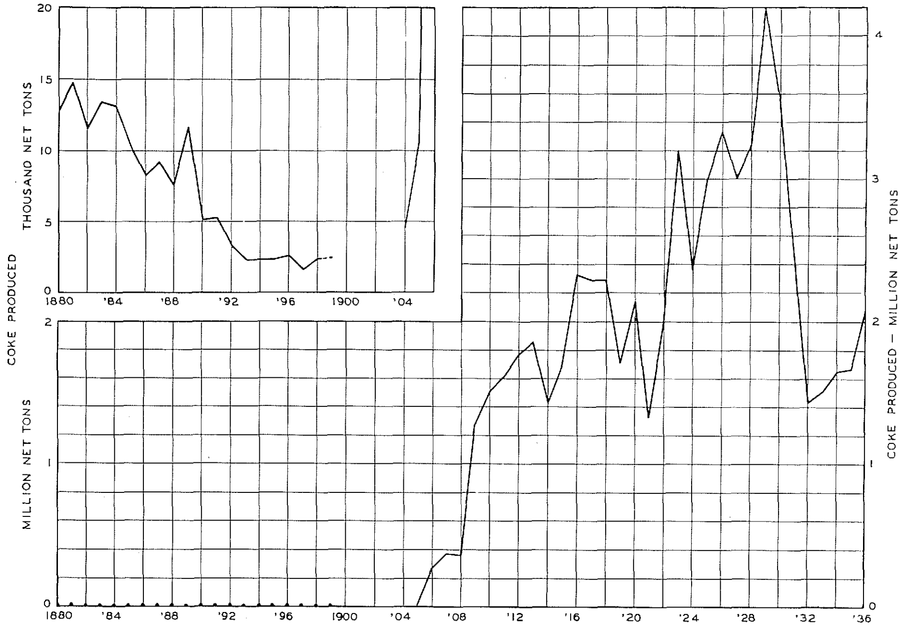


Fig. 10.—Coke Produced in Illinois, 1880 to 1936.

The most exhaustive experimental attempts to produce beehive coke from Illinois coals were made at the fuel testing plant of the United States Geological Survey, located at the Louisiana Purchase Exposition from 1904 to 1907, and at Denver, Colorado, from 1907 to the summer of 1909.[3] Two standard sized beehive ovens were used in these tests. Briefly, these tests showed that a fair metallurgical coke could be made from some Illinois coals, that washing the coal in many cases improved the quality of the coke or made possible its successful

---

[2] Moss, R. S., Improved Heminway Process: Mines and Minerals, vol. 21, No. 6, pp. 412-14, April 1901.

[3] Parker, E. A., Holmes, J. A., and Campbell, M. A., Report on the operations of the coal-testing plant of the United States Geological Survey at the Louisiana Purchase Exposition, St. Louis, Mo., 1904: Part III Producer-Gas, Coking, Briquetting and Washing Tests. U. S. Geol. Survey, Prof. Paper 48, 1906.

Holmes, J. A., Preliminary report on the operations of the fuel testing plant of the United States Geological Survey at St. Louis, Mo., 1905: U. S. Geol. Survey Bull. 290, 1906.

Holmes, J. A., Report of the United States fuel-testing plant at St. Louis, Mo., January 1, 1906 to June 30, 1907: U. S. Geol. Survey Bull. 332, 1908.

Belden, A. W., Delamater, G. R., and Groves, J. W., Washing and coking tests of coal at the fuel-testing plant, Denver, Colorado. July 1, 1907 to June 30, 1908: U. S. Geol. Survey Bull. 368, 1909.

Belden, A. W., Delamater, G. R., Groves, J. W., and Way, K. M., Washing and coking tests of coal at the fuel-testing plant, Denver, Colorado, July 1, 1908 to June 30, 1909: U. S. Bureau of Mines Bull. 5, 1910.

production, and that the sulfur contents of the cokes were in many cases higher than was was desirable for foundry or blast furnace cokes. At that time there was little or no demand for coke as a domestic fuel. The coke was, therefore, judged as to quality by its suitability for use in a cast-iron foundry cupola. Much of the coke classed as undesirable for metallurgical use by these tests because of its sulfur content or fingery structure would probably be acceptable as domestic coke today. Inasmuch as Illinois coals in most cases rapidly lose much of their coking power on weathering and since the length of time elapsed between the mining and the testing of the coals is not given, it is possible that many of the coals which did not yield good cokes in the test would have done so had they been fresh and coked under more favorable conditions.

Fig. 11.—General View of Abandoned Beehive Coke Ovens near Sparta, Randolph County, Illinois.

In addition, there have been several semi-commercial attempts by private organizations to coke Illinois coal in beehive type ovens. In most of these cases only a few ovens were built and they were soon abandoned. An installation of 50 beehive ovens was located on the Mobile and Ohio Railroad, southwest of Sparta in Randolph County. Only a few were ever put into operation and these were later abandoned. Their present state is shown in figures 11 and 12. It is said that satisfactory coke could not be made in them from coal obtained at an adjoining mine and that the battery of ovens was built before adequate tests had been made.

USE OF BY-PRODUCT OVENS FOR COKING ILLINOIS COAL

The close proximity of the Illinois coal fields to several of the large markets for metallurgical and industrial coke has naturally resulted in attempts to use Illinois coals for by-product coke manufacture. This was especially true during the World War when supplies of eastern coals and transportation facilities westward were curtailed. The following paragraphs contain a brief review of these attempts and summarize the conclusions drawn from them.

Fig. 12.—Abandoned Ovens at Sparta: view of Oven Door and Arch with Part of Stone Facing Removed.

The United States Bureau of Standards made a study of the possibility of coking Illinois coals in an installation of 24 Roberts type recuperative ovens at Canal Dover, Ohio, at various times during 1917-1918.[4] Trouble was encountered with the operation of the recuperators during the tests but a satisfactory metallurgical coke was produced from No. 6 Illinois coal and used in a blast furnace. The conclusions to which the Bureau of Standards came were as follows:

"At least the majority of the coals from Franklin County, Illinois, and some from Pike County, Indiana, located in the midcontinent field can be coked in by-product coke ovens, and a fairly satisfactory grade of metallurgical coke can be produced. Likewise by-products of reasonable quantity and good quality can be obtained from these coals.

---

[4] U. S. Bureau of Standards Miscellaneous Publications 46. War Work of the Bureau of standards, pp. 73-82, Coke-oven investigations, 1921.

"The Bureau believes that at least some of the coal from the midcontinent field should be used in the coke-oven plants in the central section of the country. Even though the coke, on the whole, proved not to be uniformly as satisfactory as that from the so-called coking coals, the advantage due to the elimination from the coke-oven plants in the eastern section will go far in overcoming any lack of quality of the coke. There can be no doubt that where domestic coke is wanted these coals can fulfill all requirements. Such uses will materially aid in the proper development of the fuel resources of the country."

The Bureau of Standards in cooperation with the Bureau of Mines also made a large scale test using about 7600 tons of Illinois coal from Orient No. 1 mine in Franklin County in an installation of Koppers ovens at St. Paul, Minn.[5] The conclusions from these tests were that it was "clearly demonstrated that some of the Illinois coals can be coked in the 'chamber type' oven without radical change in operating methods for the production of coke which can be successfully used in a blast furnace", and that it appeared that "the temperature at which Illinois coal should be handled for the production of the best coke is somewhat lower than the best operating temperatures for eastern coals, and moreover the speed of coking of the Illinois coal is somewhat less . . . The coke produced was very irregular in size, had a longitudinal fracture, was fingery, brittle, and shattered easily. The cell structure was very small and irregular. The coke was lighter than the average by-product coke, weighing only 23 pounds per cubic foot." The coke was tested by use in a blast furnace and found to be satisfactory. At the time the tests were carried out there was a shortage of coke, for which reason the suitability of the coke as a domestic fuel was not considered.

It is said that at this time also, Illinois coals were being coked experimentally on a large scale at Gary, Indiana, by the United States Steel Corporation and at other places. As an outgrowth of the development work on the Roberts type ovens at Canal Dover, Ohio, 80 ovens of an improved Roberts form in two batteries of 40 ovens each were placed in operation at Granite City, Illinois, in January 1921 for making metallurgical coke from southern Illinois coals,[6] and were operated until June 15, 1935 when they were abandoned. These ovens were operated for several months with Illinois coal alone from Franklin and Williamson counties. The more usual operation was with mixtures of about 85 per cent Illinois coal and 15 per cent West Virginia Pocahontas coal. The Illinois coal came from Franklin and Williamson counties in the following proportions:

Year	1928–1930 inclusive (per cent)	1931 (per cent)
Franklin Co.	65	80
Williamson Co.	35	20

---

[5] McBride, R. S., and Selvig, W. A., Coking of Illinois coal in Koppers type oven: U. S. Bureau of Standards Tech. Paper 137, 1919.

[6] Ditto, M. W., Design and operation of Roberts coke oven: Trans. Amer. Inst. Min. Met. Eng. vol. 69, pp. 483-512, 1923.

The Illinois coal had to be reasonably fresh to give good results. If the coal were stored more than two weeks difficulties arose due to the charge sticking in the ovens. Considerable quantities of this coke were sold for domestic fuel purposes in the St. Louis area during the last few years of the plant's operation.

On the basis of the results obtained during the operation of two experimental ovens at St. Louis, a battery of 10 Knowles sole-flue type ovens was erected at West Frankfort, Franklin County, in 1934.[7] This installation was increased to 26 ovens by the addition of two batteries of 8 ovens each in 1936. This plant is in regular operation, coking screenings from Illinois No. 6 coal produced at nearby mines. The coke is sold for domestic and industrial purposes. Because this plant successfully cokes Illinois coal and because its operation has not been described elsewhere in detail, a more detailed discussion of the operating conditions of these ovens is presented in this report (pp. 179-203).

APPLICATION OF LOW- AND MEDIUM-TEMPERATURE CARBONIZATION PROCESSES TO ILLINOIS COALS

The Parr process.—The pioneer in work on low-temperature carbonization of Illinois coals was the late Professor S. W. Parr, who was interested in the production of smokeless fuel from them. He has written as follows:[8]

> "First and historically this work was begun at the time of the great anthracite strike in 1902, but our idea from the start was to work on fundamental factors involving especially the rôle of oxygen, both inherent in the chemical composition of coal and that absorbed from the time of breaking out at the mine. We worked almost entirely on laboratory studies and with laboratory apparatus, but we moved from tests involving the use of a few ounces of coal up to a few pounds—twenty pounds, then thirty-three pounds, then six hundred, and finally, in ton lots. At the end of about ten years of experimentation I became thoroughly convinced that the low temperature range, say from 700° to 900°F. (360°-480°C.) was not at all suited to our economic conditions and environment. From that time on we centered our efforts upon a mid-temperature range of from 1,400° to 1,500°F. (760°-815°C.). Our final apparatus might well be considered a semi-industrial size of plant. . .
>
> "I may say, moreover, that this outfit operated continuously for three hundred and sixty-five days, twenty-four hours per day, without a single shutdown or delay other than for making minor repairs or adjustments, and that while we operated upon car-lots from as far west as Iowa and as far east as Pocahontas and Pittsburgh fields in Pennsylvania, and as far south as Birmingham, Alabama, including Kentucky, Indiana and substantially all the fields of Illinois, we did not fail in any case to make a high grade coke. . . . ."

---

[7] Anon., Knowles oven widens market for No. 5 Illinois screenings by turning fines into domestic coke: Coal Age, vol. 39, pp. 421-423, Nov. 1934.

McBride, R. S., Processing coal in Knowles coke ovens: Chem. and Met. vol. 42, No. 6, pp. 300-3, June 1925.

[8] Proceedings of the Symposium on Fuel and Coal. McGill University, pp. 186-187, 1931.

The Parr process is a two-stage process in which the coal is heated to within 50°C. of its plastic temperature in the first stage in an externally heated, rotating horizontal retort and then charged into a coke oven and coked. During the preheating stage the rise in temperature of the coal is slow until 100°C. has been passed and then rapid to 300°C. Coking is rapid in the second stage due to the fact that moisture and carbon dioxide have been driven off and the coal is almost at the fusion point. Coke oven operators claim that great difficulties arise in charging dry, preheated coal into hot coke ovens because the rapidly evolved gases in the oven carry much fine coal dust with them. This dust deposits in collector mains and tar-handling equipment and may plug them.[9] Even relatively small amounts of dust are undesirable, since they increase the so-called "free-carbon" content of the tar. It is said that in some cases practically the entire charge of dry, hot coal has been carried out of the oven. This process is not in commercial or experimental operation today. The experimental plant operated by Professor Parr has been dismantled.

Greene-Laucks process.—In the Greene-Laucks process the coal is propelled upwards on a worm conveyor shaft in a vertical retort. Heat is applied on the outside of the retort and inside the hollow conveyor. This method leaves 15 to 17 per cent volatile matter in the coke. A pilot plant having a rotary conveyor 3 feet in diameter and 18 feet long and a capacity of 15 tons of coal per day was operated in Waukegan, Illinois, for two years. No full size plant was built and the pilot plant has been dismantled.[10]

Other processes.—Illinois coals have been experimentally coked in the course of development or promotion of a large number of low-temperature carbonization processes. The reports of these tests are rarely given in sufficient detail to have any value. In most cases the process in question "successfully processes" the Illinois coal. Since most of these processes did not go beyond the experimental stage and since none are at present operating or appear likely to operate in the future with Illinois coal, they are not discussed further here.

Small scale carbonization tests on Illinois coals.—Because of the expense involved or the danger of damage to large scale equipment in conducting large scale coking tests, there is a natural tendency to try to determine the coking properties of coals on much smaller scales. The use of small scale equipment introduces the difficulty of so conducting the test that the results may be directly applied to full scale practice. However, even though the results may not be comparable to full scale work, small scale tests in many instances are comparable

---

[9] Discussion by Ulmer, C. D., Ramsburg, C. J., and Good, J. B., of paper by Warren, W. B., The relation of work of the laboratory to practical carbonization: Proceedings of Technical Meeting held under the auspices of the Coal Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Pa., Dec. 3, 1936, mimeographed, pp. 17-19.

[10] Greene, F. C., The Greene-Laucks process: Proc. First Int. Conf. on Bit. Coal, pp. 712-728, 1926.

among themselves. Small scale tests are especially valuable in studying coals about whose properties very little is known and for which there would be a very great risk of failure if they were tested on a large scale. It is impractical, if not impossible, to refer to and review all of the small scale carbonization tests which have been made on Illinois coals. Only those which appear to the author to be of most significance are referred to here.

The late S. W. Parr probably conducted more tests on the possibilities of producing a smokeless fuel from Illinois coal than any other man. The scale on which he worked was, as quoted above, gradually increased in the course of some twenty years work until his last tests were made in a commercial scale experimental oven. Professor Parr's efforts were largely directed towards the production of a low-temperature coke to supply a demand for a smokeless domestic fuel.[11]

As a result of Parr's investigation on the carbonizing characteristics of Illinois coals he came to the conclusion that these coals produced a much better coke if they were given a preliminary conditioning heat treatment in an inert atmosphere at a temperature just below the softening temperature, that is, at about 350°C. This conditioning treatment in an inert atmosphere is one of the important features in the Parr low-temperature coking process. The conditioned coal is charged into an oven similar in construction to a standard by-product oven and carbonization is finished at a temperature of from 750°C. to 800°C.

The United States Bureau of Mines in cooperation with the American Gas Association undertook a survey of the gas, coke, and by-product making properties of American coals during the course of which they examined coal from No. 6 bed at West Frankfort, Franklin County, Illinois.[12] This was the same coal used in the full sized tests at the plant of the Minnesota By-Product Coke Company, carried out cooperatively by the Bureau of Mines and the Bureau of Standards.[13] The results of their cooperative tests on the Orient (Illinois) coal are of special interest because this was one of a group of thirty or more coking coals from various parts of the United States to which the systematized tests

---

[11] Parr, S. W., Anthracizing bituminous coals: Illinois State Geol. Survey Bull. 4, 1907.

Parr, S. W., and Francis, C. K., Artificial modification of the composition of coal: Illinois State Geol. Survey Bull. 8, 1908.

Parr, S. W., and Francis, C. K., Univ. of Illinois Eng. Expt. Sta. Bull. 24, 1909.

Parr, S. W., and Olin, H. L., The coking of coal at low temperature with a preliminary study of the by-products: Univ. of Illinois, Eng. Expt. Sta. Bull. 60, 1912.

Parr, S. W., and Olin, H. L., The coking of coal at low temperatures, with special reference to the properties and composition of the products: Univ. of Illinois Eng. Expt. Sta. Bull. 79, 1915.

Parr, S. W., Low temperature carbonization of coal: Proceedings of the Second International Conference on Bituminous Coal, vol. 1, pp. 54-70, 1928.

[12] Fieldner, A. C. et al., Carbonizing properties and constitution of No. 6 bed from West Frankfort, Franklin Co., Illinois: U. S. Bureau of Mines Tech. Paper 524, 1932.

[13] McBride, R. S., and Selvig, W. A., Coking of Illinois coal in Koppers type oven: U. S. Bureau of Standards Tech. Paper 137, 1919.

were applied, providing truly comparable results. In comparing the carbonizing characteristics of the Orient coal from Franklin County, Illinois, with those of the eastern gas coals, Fieldner and his coworkers arrived at the following conclusions quoted from Technical Paper 524.

"Correlation of the chemical composition and physical properties of the Orient coal is of particular interest in view of its relatively low rank in the scale of classification of bituminous coal. The high oxygen content of Orient coal is reflected in its carbonizing properties as compared to eastern gas coals by:

"1. A higher yield of liquor.

"2. A lower yield of highly oxygenated tar, containing nearly 30 per cent of tar acids in the low-temperature tar.

"3. A moderate yield of gas rich in oxides of carbon and therefore of somewhat low calorific value, 2,650 B.t.u. in stripped gas per pound of coal at a carbonizing temperature of 900° C.

"4. A lighter and more porous coke.

"5. A lower agglutinating index.

"6. Less development of plasticity and a lower swelling coefficient."

They also conclude that:

"On blending with 25 per cent of low-volatile coal, however, it makes excellent metallurgical or domestic coke" and that:

"The physical properties of the coke obtained in the test retort at a carbonizing temperature of 900° C. were similar to those of coke obtained in tests of coal from the same mine in by-products ovens, and the oven yields of gas, coke, and tar were between those obtained in the retort tests at carbonization temperatures of 900° and 1000° C."

The United States Bureau of Standards also tested this coal on a small scale at about the time it carried on the large scale tests in the Koppers type ovens at St. Paul, Minn. Four pounds of coal were carbonized in a cast iron box placed in a special muffle furnace regulated so that the temperature of the vapor over the coal being coked was 840°, 775°, 700°, 600° and 605°C. in the five tests. By-product and coke yields and general observations on the quality of the coke are reported.[14] The investigators warn against making any conclusions from the tests.

CONCLUSIONS FROM THE HISTORICAL REVIEW OF THE ART OF PRODUCING COKE FROM ILLINOIS COALS

The production of coke from Illinois coals has occupied the attention of the coal industry and of investigators for more than sixty years. During this period the art of coking Illinois coal followed closely the art of coking the more easily coked coals. From about 1880 to 1900 some coke was produced from Illinois coals in beehive or similar ovens, the coke being sold for light industrial purposes. Illinois coals apparently are not nearly as well adapted to being coked in beehive ovens as are some of the Appalachian coals. In an effort to improve the pro-

---

[14] McBride, R. S., and Brumbaugh, I. V., Experimental retort tests of Orient coal: U. S. Bureau of Standards Tech. Paper 134, 1919.

duction of coke from Illinois coals various modifications of the beehive oven were introduced. In comparison with the total United States production of beehive oven coke, the production of beehive coke from Illinois coal was unimportant. The rapid loss of coking power on exposure to air and the high moisture and volatile matter content probably were the controlling factors in the manufacture of coke from Illinois coals by the beehive oven process.

The introduction of the by-product recovery coke oven into the Illinois area led to trials of Illinois coals in them. Again, the rapid loss of coking power and the much shrunken, fingery coke formed because of the high volatile matter content placed the coals from Illinois in an unfavorable position when compared with the eastern coking coals. As before, efforts were made to modify the by-product coke oven to make it more suitable for the coking of these coals. Until recent years coke was made primarily for metallurgical purposes and the quality and values of the coke produced were judged mainly from the standpoint of the blast-furnace operator.

A growing realization of the importance of the domestic fuel market for coke has led to a reconsideration of the coke which may be made from Illinois coals. Cokes which would be inferior for metallurgical uses may be well adapted for use as a domestic fuel. The mechanical problems of removing the fingered and shrunken coke from the oven may be and have been solved by modifications in oven construction. The new possibilities in the disposal of coke from Illinois coals makes desirable a survey of the coke-making properties of the coals found in Illinois, their improvement for coke-making purposes, and an investigation of the conditions under which they produce the best coke.

Present production of coke from Illinois coals is in an installation of sole-flue ovens, having characteristics quite different from those of the more common slot-type ovens. Because these ovens seem to be well suited to the carbonization of Illinois coals, certain features of their operation, particularly the temperature conditions throughout a charge during a coking cycle, have been investigated and are reported in detail together with analyses of the coke as produced (see pp. 179-203).

In these ovens a coke resembling slot-type, by-product oven coke or a coke grading from high-temperature coke at the bottom to a higher volatile, lower temperature coke at the top can be produced at will and easily removed from the oven. The higher volatile content cokes may have great advantage over low volatile cokes as domestic fuels.

CHAPTER VIII—THE REQUIREMENTS OF COKING COALS

The difficulties encountered in endeavors to coke Illinois coals indicate the desirability of a study of the requirements of coal for such use. Such a study should include consideration of the commonly accepted criteria for determining whether or not a coal is a coking coal, and also experimental studies with respect to the yield and characteristics of the products of carbonization of a variety of Illinois coals, even though such investigations may be possible only on a laboratory scale. Such studies and investigations will provide some answer to the questions concerning the nature of coking coals and the extent to which Illinois coals are coking coals.

CRITERIA FOR COKING COALS

Gluud's Handbook[1] defines coking coals as follows:

> "The commercial value of coke depends upon its purity, and upon such factors as size, strength and structure. These physical characteristics are determined not only by the kind of coal used, but also by the degree of pulverization and moisture content of the coal, the amount, nature and size of impurities such as slate, the temperature of the oven walls, dimensions of the oven, and other variables, all of which affect the coke at some stage of its formation. Therefore, it is not surprising to find that the only thoroughly reliable method to determine the quality of coke that can be made from a coal under particular conditions, is to subject the coal to a full-scale oven test under the desired conditions.
>
> "For practical purposes, a coal may be classed as a coking coal if it will yield a merchantable coke when carbonized by a commercial method in an existing type of coke oven. In order to be merchantable, a coke must, in general, be of adequate purity, size, strength and structure, for the uses to which it is to be put. Furthermore, the coke from any coal or coal mixture carbonized in by-product ovens must be strong enough and have sufficient shrinkage from the walls to permit its discharge from the ovens without difficulty. Coal mixtures are commonly used at by-product coke-oven plants, and this practice has resulted not only in the production of superior coke, but in the extensive utilization of coals which would not be very suitable for coke manufacture, if used separately."

It is to be noted that this definition indicates that a coke should be judged according to its suitability for the use to which it is to be put. In the past cokes from Illinois coals have almost always been judged as to their suitability for metallurgical purposes. These cokes should now be judged as to their suitability for use as a smokeless and convenient domestic fuel. Inasmuch as the requirements which must be met by the coke for domestic use are different and

---

[1] Gluud, W., International handbook of the by-product coke industry: American Edition by D. L. Jacobson. New York 1932, p. 156.

not so rigorous as for metallurgical purposes, it seems quite possible that a different valuation should be placed upon many Illinois coals as coking coals.

The American Society for Testing Materials has adopted standard specifications for gas and coking coals which may be used as a guide in the selection of coking coals. It should be emphasized that these specifications were set up with the production of metallurgical coke in slot-type by-product ovens primarily in mind and are rather rigorous; however they do furnish a means for evaluating coals comparatively. Coals which fall outside the limits for coking coals set up in this specification may still yield coke of good quality entirely satisfactory for many purposes. These specifications are reproduced here:[2]

STANDARD SPECIFICATIONS FOR GAS AND COKING COALS

A. S. T. M. Designation: D 166-24

"These specifications are issued under the fixed designation D 166; the final number indicates the year of original adoption as standard, or, in the case of revision, the year of last revision.

ISSUED AS TENTATIVE, 1923; ADOPTED, 1924

"1. Gas and coking coals must yield both merchantable gas and coke when distilled in a retort or oven by commercial methods. The type of coals may vary within rather wide limits according to the treatment in the retort and the market for the products. These specifications, therefore, merely give the limits within which gas and coking coals will usually fall, and indicate the circumstances under which further restrictive conditions should be imposed.

Sampling and Analysis

"2. The coal shall be sampled in accordance with the Standard Method of Sampling Coal (A.S.T.M. Designation: D 21) of the American Society for Testing Materials.

"3. Analyses of the coal, when required, shall be made in accordance with the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271) of the American Society for Testing Materials.

Chemical and Physical Properties

"The carbon ratio, that is, the ratio of fixed carbon to volatile matter, while not entirely reliable, is the best simple index to the behavior of the coal when carbonized. The carbon ratio in the case of gas coals will vary from 1.4 to 2.0 and for coking coals from 1.4 to 5.0. The latter includes a wide range of coals varying from high volatile gas coal to low volatile or 'smokeless' coal.

"4. (a) The percentage of moisture in the coal as mined shall be subject to agreement by the purchaser and the seller.

"(b) In the absence of a definite agreement between the purchaser and the seller, the mine moisture in the coal as mined shall not exceed 4.0 per cent. The moisture shall be determined by the general average composition of coal from the mine in question and an analysis of each shipment shall not be required.

---

[2] Amer. Soc. for Testing Materials: A. S. T. M. Designation D 166-24, A. S. T. M. Standards: pp. 379-381, 1936. Reprinted by permission of the Society from its copyrighted publications.

"5. The fusion temperature of ash of coal, the coke from which is intended for domestic and industrial use, shall not be below 2200°F. In the case of coke for use in the manufacture of water gas, the fusion temperature of the ash of the coal shall preferably be higher than 2300°F. The fusion temperature of the ash shall be determined in accordance with the method for determination of fusibility of coal ash appearing in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271) of the American Society for Testing Materials.

"NOTE.—The fusion point of ash is not usually important for metallurgical work. It is important, however, in the case of coke for domestic and industrial furnace use and for the manufacture of water gas.

A. Special Requirements for Gas Coals

"6. Gas coal shall contain not less than 35.0 per cent of volatile matter when determined on the moisture and ash-free basis.

"NOTE.—This is equivalent to 30.8 per cent volatile matter for a coal containing 12.0 per cent of combined ash and moisture.

"7. In the case of gas coals, the ash in the dry coal shall not be over 9 per cent.

"8. The composition of gas coal shall be such that the dry coke produced therefrom will contain not over 1.5 per cent of sulfur and the resultant gas will contain not more than 30 grains of sulfur, in the form of compounds other than hydrogen sulfide, per 100 cu. ft. of gas.

"9. Gas coal shall be such that the coke produced therefrom will be of sufficient size and strength to be suitable for domestic use or for the manufacture of water gas.

"NOTE.—These physical characteristics of coke are not amenable to simple explicit definition and must necessarily be left to the judgment of experienced operators."

B. Special Requirements for Coking Coals

"10. In the case of coking coals, the ash in the dry coal shall be not over 9 per cent.

"11. (a) If metallurgical coke is to be produced, the composition of the coking coal shall be such that the dry coke produced therefrom will not contain more than 1.0 per cent of sulfur in the case of foundry coke, and 1.3 per cent of sulfur in the case of blast-furnace coke.

"(b) If gas is to be sold for domestic use, the composition of the coking coal shall be such that the resultant gas will contain not more than 30 grains of sulfur, in the form of compounds other than hydrogen sulfide, per 100 cu. ft. of gas.

"12. A limitation as to phosphorus, which may be required when coke is used for metallurgical purposes, shall be subject to agreement between the purchaser and the seller.

"13. The composition of coking coal which is to be charged into a by-product oven without admixture shall be such that the coke produced therefrom will shrink sufficiently to permit of its being discharged from the oven without difficulty.

"NOTE.—The mixing of coals for by-product coke-oven use is widely practiced, and such mixtures usually contain, as a very important component, low volatile or 'smokeless' coking coals, which when carbonized alone would not give the requisite shrinkage.

"14. The composition of the coking coal shall be such that the coke produced therefrom will meet such requirements as to size, strength, and structure as are necessary for good practice in the industries using the coke.

"NOTE.—These physical characteristics of coke are not amenable to simple explicit definition and must necessarily be left to the judgment of experienced operators."

In the discussions which follow various county-average analytical values and values derived from them will be used to determine where Illinois coals stand when judged by the foregoing A.S.T.M. specifications. The values used are

TABLE 11.—COUNTY AVERAGE ANALYTICAL VALUES FOR ILLINOIS COALS

No.	County	Geographic location[a]	Coal	Moisture as received per cent	Ash (dry) per cent	Sulfur (dry) per cent	Fixed carbon (dry) per cent	Volatile matter (dry) per cent	Volatile matter (dry, ash-free) per cent	Fixed carbon (unit coal) per cent	Rank index	Ultimate analysis: Carbon (unit coal) per cent	Ultimate analysis: Hydrogen (unit coal) per cent	Ultimate analysis: Oxygen (unit coal) by difference per cent	Specific volatile index	Ratio: Fixed carbon to volatile matter
1	Bond	C	Herrin 6	11.9	12.2	3.8	47.9	39.9	45.4	55.9	123	82.9	5.5	11.6	122.2	1.20
2	Bureau	N	LaSalle 2	16.1	8.8	3.5	45.4	45.8	50.2	50.6	119	82.0	5.5	12.5	131.1	.99
3	Bureau	N	Sparland 7	17.7	16.4	3.8	44.3	39.3	47.0	54.6	115	82.4	6.1	11.5	127.5	1.13
4	Christian	C	Lower Assumption	11.3	10.0	2.6	46.2	43.8	48.7	52.1	129	....	...	....	136.3	1.05
5	Christian	C	Upper Assumption	13.0	8.0	3.6	47.0	45.0	48.9	52.0	126	....	....	....	135.0	1.04
6	Christian	C	Herrin 6	12.7	11.7	4.5	46.0	42.3	47.9	53.4	123	81.8	5.9	12.3	126.6	1.08
7	Clinton	C	Herrin 6	11.8	11.9	4.2	46.6	41.5	47.1	54.2	123	81.8	5.6	12.6	124.1	1.12
8	Edgar	C	Springfield 5?	12.5	11.2	3.5	46.4	42.4	47.7	53.4	126	83.9	5.8	10.3	134.4	1.09
9	Franklin	S	Herrin 6	9.2	9.5	1.6	53.2	37.3	41.2	59.5	131	84.3	5.4	10.3	129.9	1.42
10	Fulton	N	Rock Island 1	11.2	11.5	5.6	45.2	43.3	48.9	52.5	130	....	...	....	137.5	1.05
11	Fulton	C	Springfield 5	15.3	13.2	3.8	45.2	41.6	47.9	53.4	120	83.6	5.6	10.8	129.6	1.0$^{9}_{0}$
12	Fulton-Peoria	N	Herrin 6	15.9	10.7	3.6	50.5	38.8	43.4	57.8	119	82.9	5.6	11.5	126.8	1.3
13	Gallatin	S	Harrisburg 5	4.1	9.2	3.6	55.1	35.7	39.3	61.9	145	....	...	....	142.1	1.5^{4}
14	Gallatin	S	Harrisburg 5	4.9	11.7	3.6	50.9	37.4	42.4	59.0	141	....	...	....	137.0	1.3^{6}
15	Gallatin	S	Herrin 6	4.3	11.1	3.9	51.2	37.7	42.4	59.0	143	....	...	....	139.4	1.3^{6}
16	Greene	C	Summum 4?	14.4	11.0	4.6	46.8	42.2	47.4	53.8	122	83.2	5.6	11.2	130.7	1.10
17	Grundy	N	La Salle 2	17.1	7.0	3.3	47.9	45.1	48.5	52.3	118	82.7	5.7	11.6	132.0	1.06
18	Grundy	N	Verona 6?	14.0	12.4	4.6	45.3	42.3	48.3	53.1	123	81.7	5.8	12.5	131.3	1.07
19	Henry	N	Rock Island 1	15.9	10.8	5.7	45.6	43.6	48.9	52.5	119	81.9	6.1	12.0	129.2	1.04
20	Henry	N	Colchester 2	14.5	11.6	4.0	45.1	43.3	49.0	52.2	119	81.9	5.8	12.3	126.1	1.04
21	Henry	N	Herrin 6	19.1	15.1	4.8	47.4	37.5	44.2	57.6	112	83.1	5.6	11.3	124.4	1.26
22	Jackson	C	Murphysboro 1	9.3	6.3	1.4	56.2	37.5	40.0	60.6	133	....	...	....	136.4	1.50
23	Jackson	S	Herrin 6	9.4	10.9	2.4	51.0	38.1	42.8	58.3	131	83.6	5.7	10.7	130.9	1.34
24	Jefferson	C	Herrin 6	8.5	9.5	1.4	52.7	37.8	41.8	59.0	133	84.1	5.5	10.4	131.7	1.39
25	Knox	N	Rock Island 1	14.3	9.3	5.3	47.9	42.8	47.2	54.1	123	....	...	....	131.9	1.12
26	Knox	N	Summum 4	15.0	9.1	4.6	46.2	44.7	49.2	52.0	123	....	...	....	136.0	1.03
27	Knox	N	Herrin 6	17.9	13.0	3.8	47.5	39.5	45.4	56.0	116	84.0	5.5	10.5	129.0	1.20
28	La Salle	N	La Salle 2	14.5	9.7	3.9	45.4	44.9	49.7	51.3	122	83.5	5.6	10.9	132.2	1.01
29	La Salle	N	Herrin 6	14.8	11.3	4.0	40.2	48.5	54.7	46.3	120	....	...	....	130.5	.83
30	La Salle	N	Streator 6	13.2	9.9	4.2	44.5	45.6	50.6	50.5	125	82.6	5.7	11.7	133.9	.98
31	Livingston	N	Grape Creek	11.9	15.1	4.2	45.1	39.8	46.9	54.7	128	85.1	6.0	8.9	136.6	1.13
32	Logan	C	Springfield 5	13.5	12.3	3.6	45.1	42.6	48.6	52.6	122	83.3	5.6	11.1	128.3	1.06
33	McDonough	C	Colchester 2	16.6	10.0	3.5	49.0	41.0	45.6	55.6	119	....	...	....	131.6	1.20
34	McLean	N	La Salle 2	11.3	9.9	3.4	42.5	47.6	52.8	50.1	129	....	...	....	136.6	.89
35	McLean	C	Springfield 5	13.3	14.4	4.3	41.8	43.8	51.2	48.8	123	....	...	....	131.7	.95

36	Macon	C	Springfield 5	13.9	11.9	4.0	46.1	42.0	47.7	53.6	121	82.0	5.6	12.4	126.7	1.10
37	Macoupin	C	Herrin 6	13.3	11.3	4.6	45.6	43.1	48.6	52.7	121	80.9	5.9	13.2	124.3	1.06
38	Madison	C	Herrin 6	13.1	11.6	4.7	44.3	44.1	49.9	51.4	122	82.0	5.9	12.1	126.9	1.01
39	Marion	C	Herrin 6	10.3	12.3	4.3	46.9	40.8	46.5	54.9	128	83.4	5.8	10.8	129.7	1.15
40	Marshall	N	La Salle 2	15.1	8.5	3.3	45.5	46.0	50.3	50.6	123	....	...	....	137.2	0.99
41	Marshall	N	Sparland 7	15.3	16.7	4.1	41.7	41.6	49.9	51.5	119	83.3	5.9	10.8	130.7	1.00
42	Menard	C	Springfield 5	15.3	10.9	3.8	47.2	41.9	47.0	54.2	120	83.4	5.8	10.8	128.7	1.13
43	Mercer	N	Rock Island 1	15.6	10.6	5.3	43.7	45.7	51.1	50.1	120	....	...	....	131.6	.96
44	Montgomery	C	Herrin 6	13.2	11.8	4.8	46.5	41.7	47.3	54.1	121	82.3	5.6	12.1	124.5	1.12
45	Moultrie	C	Herrin 6	6.8	12.6	4.3	45.4	42.0	48.1	53.3	137	....	...	....	136.9	1.08
46	Peoria	C	Springfield 5	14.5	12.8	3.8	46.1	41.1	47.1	54.2	122	83.7	5.8	10.5	132.3	1.12
47	Perry	S	Herrin 6	10.3	10.5	1.0	52.7	36.8	41.1	59.7	128	83.2	5.3	11.5	126.2	1.43
48	Perry	C	Herrin 6	10.0	11.8	3.8	48.1	40.1	45.5	55.8	127	82.1	5.6	12.3	124.4	1.20
49	Randolph	C	Blair 5 (4?)	10.7	11.6	5.1	47.7	40.7	46.0	55.4	127	....	...	....	126.5	1.17
50	Randolph	C	Herrin 6	10.3	13.1	4.4	46.3	40.6	46.7	54.7	126	82.2	5.6	12.2	124.4	1.14
51	Rock Island	N	Rock Island 1	16.6	10.2	5.8	47.1	42.7	47.6	53.8	118	....	...	....	128.4	1.10
52	St. Clair	C	Herrin 6	11.3	12.4	4.2	44.6	43.0	49.1	52.2	126	83.0	5.5	11.5	128.7	1.04
53	Saline	S	Harrisburg 5 (4?)	6.6	9.0	2.7	54.3	36.7	40.3	60.7	137	85.3	5.6	9.1	134.2	1.48
54	Sangamon	C	Springfield 5	14.2	12.3	4.6	45.6	42.1	48.0	53.3	121	83.0	5.7	11.3	128.0	1.08
55	Sangamon	C	Herrin 6	13.9	11.6	4.8	45.5	42.9	48.5	52.8	121	82.2	5.6	12.2	126.4	1.06
56	Schuyler	C	Colchester 2	12.5	8.6	5.2	48.1	43.3	47.4	53.9	129	....	...	....	138.4	1.11
57	Schuyler	C	Springfield 5	15.2	11.6	3.1	47.5	40.9	46.2	54.8	122	....	...	....	134.0	1.16
58	Shelby	C	Springfield 5	11.2	12.1	4.0	48.2	39.7	45.2	56.2	126	82.8	5.9	11.3	126.9	1.21
59	Stark	N	Herrin 6	17.5	12.3	4.5	47.6	40.1	45.7	55.7	117	83.4	5.7	10.9	129.2	1.19
60	Tazewell	C	Springfield 5	15.1	11.2	3.8	46.7	42.1	47.4	53.8	120	83.9	5.7	10.4	129.6	1.11
61	Vermilion	C	Grape Creek 5	14.8	10.8	2.7	48.6	40.6	45.5	55.5	122	83.4	5.6	11.0	131.3	1.20
62	Vermilion	C	Danville 7	13.3	11.3	3.2	45.1	43.6	49.2	51.8	125	83.4	5.8	10.8	135.7	1.03
63	Warren	N	Rock Island 1	13.2	9.9	6.3	44.7	45.4	50.4	51.0	125	....	...	....	131.1	.98
64	Washington	C	Herrin 6	10.3	13.2	4.6	44.2	42.6	49.1	52.3	127	81.9	5.9	12.2	127.0	1.04
65	White	S	Herrin 6	8.5	9.8	3.1	51.5	38.7	42.9	58.2	133	84.5	5.7	9.8	131.8	1.33
66	Will	N	La Salle 2	15.4	6.0	1.9	53.5	40.5	43.1	57.6	120	81.8	6.0	12.2	127.6	1.32
67	Williamson	S	Harrisburg 5 (4?)	7.0	12.0	3.8	51.5	36.5	41.5	59.9	136	....	...	....	132.7	1.41
68	Williamson	S	Coal 6	8.0	10.0	2.4	53.4	36.6	40.7	60.4	133	84.4	5.6	10.0	130.0	1.46
69	Woodford	N	La Salle 2	14.5	7.1	2.2	52.2	40.7	43.8	56.9	124	84.2	5.6	10.2	134.3	1.28

---

[a] Samples from Northern, Central or Southern Illinois, see figure 24.

presented in Table 11, the original data for which come from or are derived from Table 4 of Bulletin 62 of the Illinois State Geological Survey.[3]

The ratios of fixed carbon to volatile matter for Illinois coals as shown by county-average analyses are shown in Table 11 and figures 13, 14, 15, and 16. County average ratios of fixed carbon to volatile matter of 1.4 or greater are found only in the southern part of the Illinois coal field in Franklin, Gallatin, Jackson, Jefferson, Perry, Saline, and Williamson counties. On the basis of the ratio limits given in this specification, Illinois coals are at the lower limit, some just managing to be included. Illinois coals as they exist in the bed all have higher moisture contents than 4.0 per cent. (See fig. 17.) The ash requirement of 9.0 per cent maximum can be easily met by careful preparation and cleaning. The county average ash values are given in figure 18. Illinois coals are less fortunate as regards sulfur. County average sulfur values are shown in figure 19. A detailed map of the low sulfur area[4] is shown in figure 20. If we assume that the sulfur content of the coke will be 0.8 of the sulfur content of the coal (see p. 127), coals having a sulfur content of less than 1.3 per cent and 1.6 per cent will be required to produce cokes having respectively sulfur contents of 1.0 per cent or less and 1.3 per cent or less. Coals having sulfur contents of 1.3 per cent or less are found in Illinois only in parts of Franklin, Jackson, Jefferson, and Perry counties. These counties also contain the only coals having sulfur contents of 1.6 per cent or less. For most non-metallurgical uses, the sulfur limit need not be so low or so rigorously adhered to as set forth in the A.S.T.M. specifications.

---

[3] Cady, G. H., Classification and selection of Illinois coals: Illinois State Geol. Survey Bull. 62, 1935.

[4] Cady, G. H., Low sulfur coal in Illinois: Illinois State Geol. Survey Bull. 38, fig. 58, p. 432, 1922.

Fig. 13.—COUNTY AVERAGE CARBON RATIOS FOR COAL NOS. 1, 4, AND 7 AND ASSUMPTION COAL IN CHRISTIAN COUNTY.

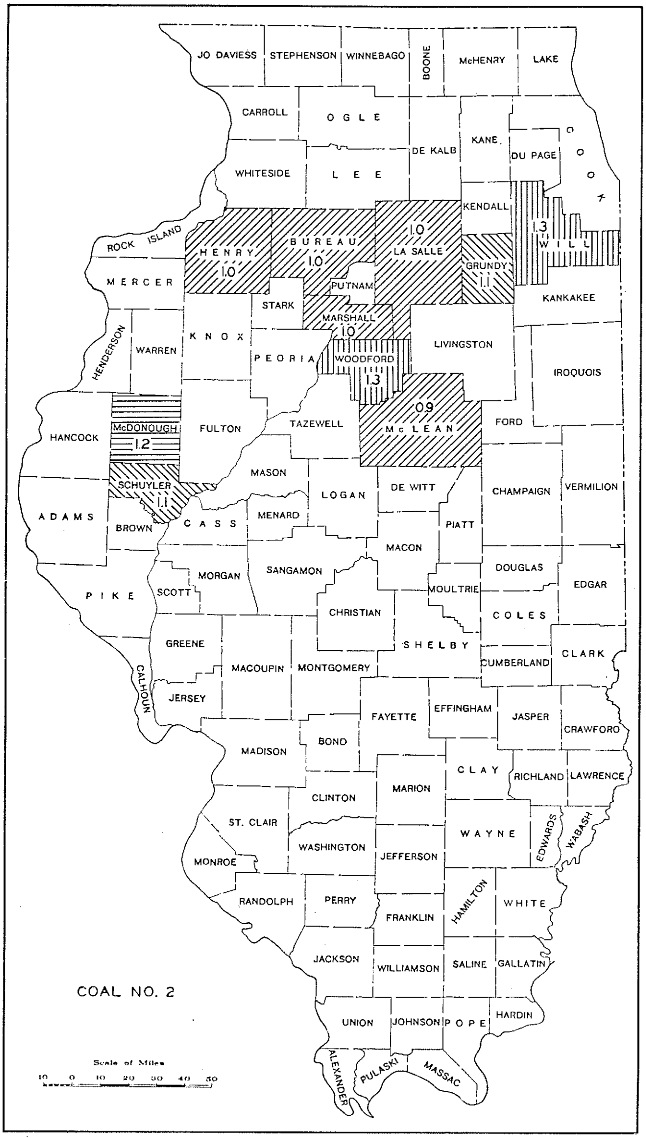


Fig. 14.—COUNTY AVERAGE CARBON RATIOS FOR COAL NO. 2.

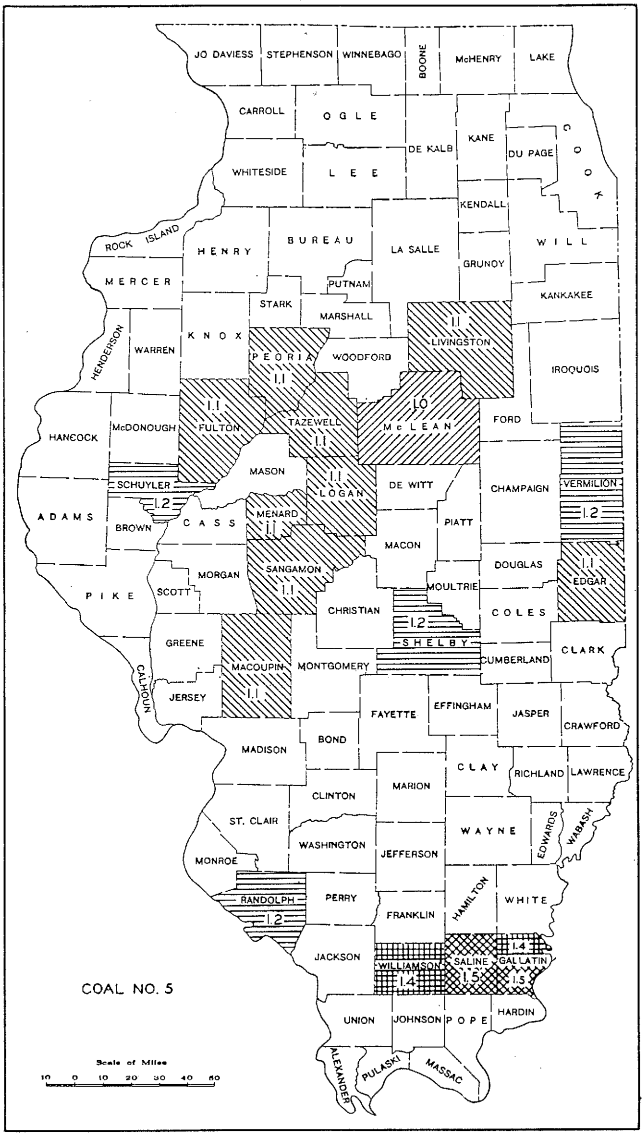


Fig. 15.—County Average Carbon Ratios for Coal No. 5.

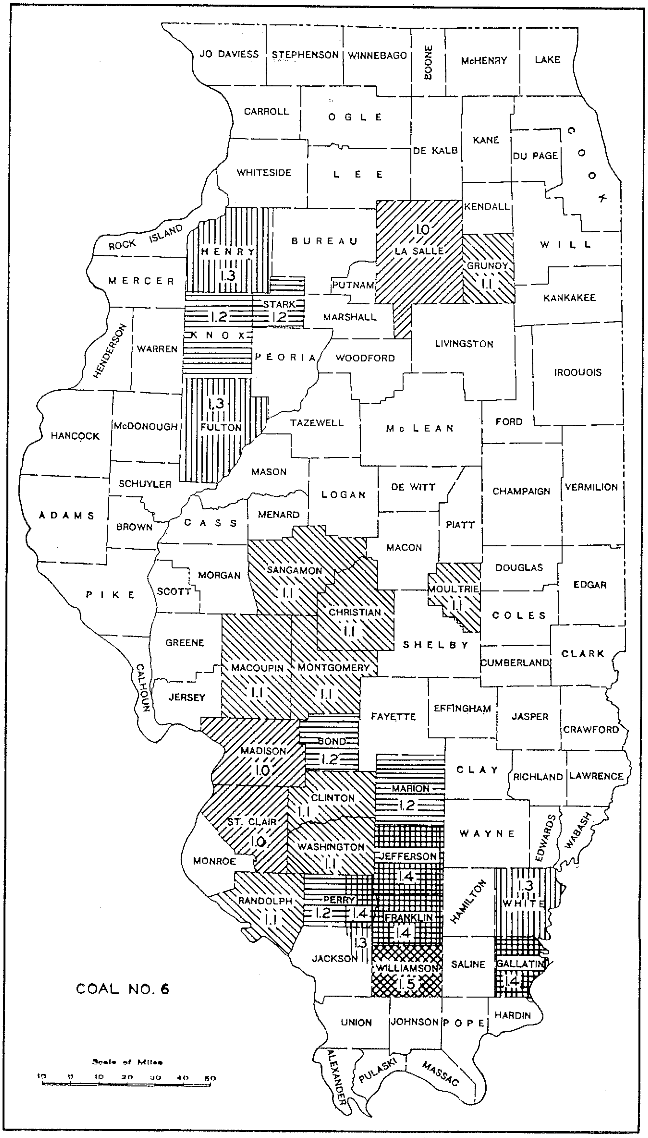


Fig. 16.—County Average Carbon Ratios for Coal No. 6.

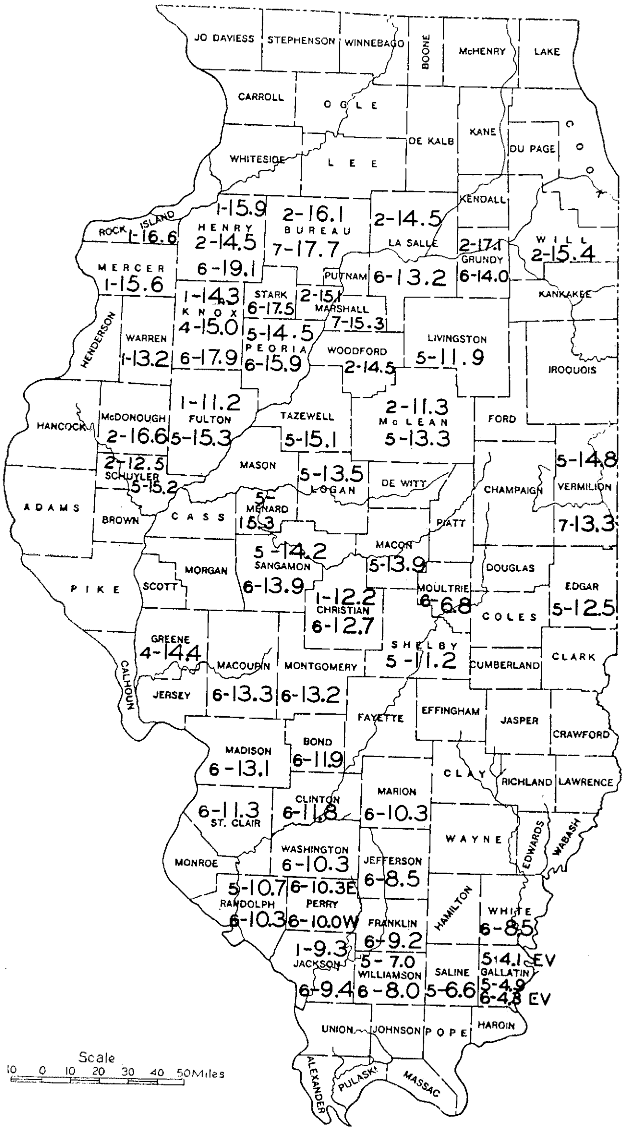


Fig. 17.—Coal Bed Numbers and County Average Moisture ("As Received") of Illinois Coal.

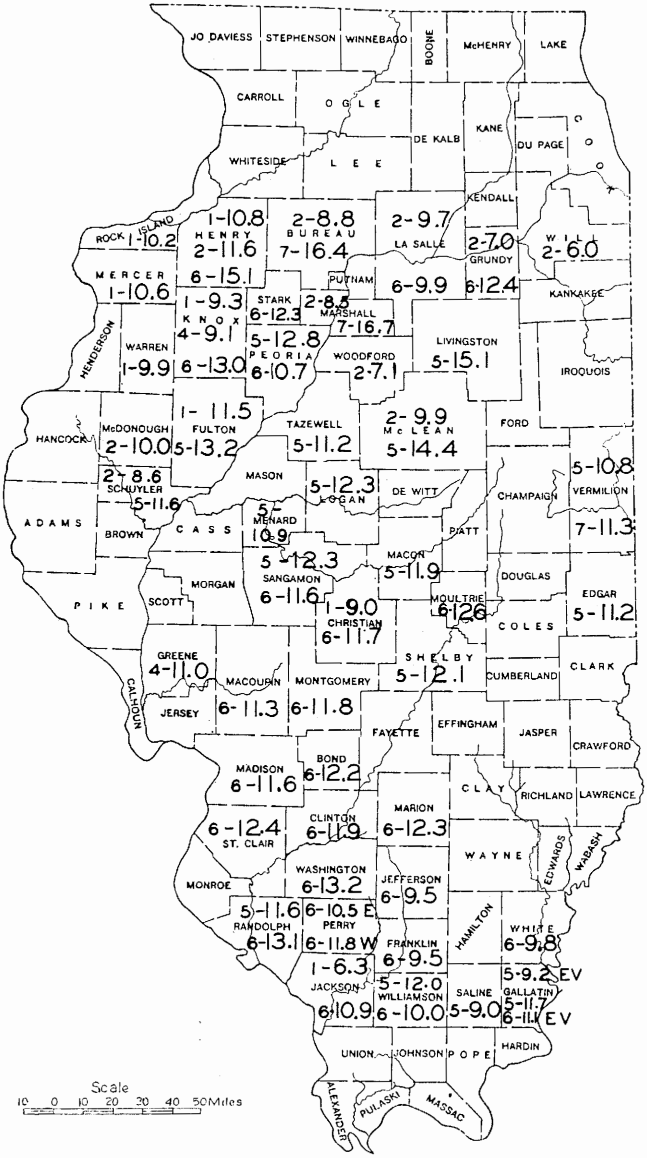


Fig. 18.—Coal Bed Numbers and County Average Ash (Dry Basis) of Illinois Coals.

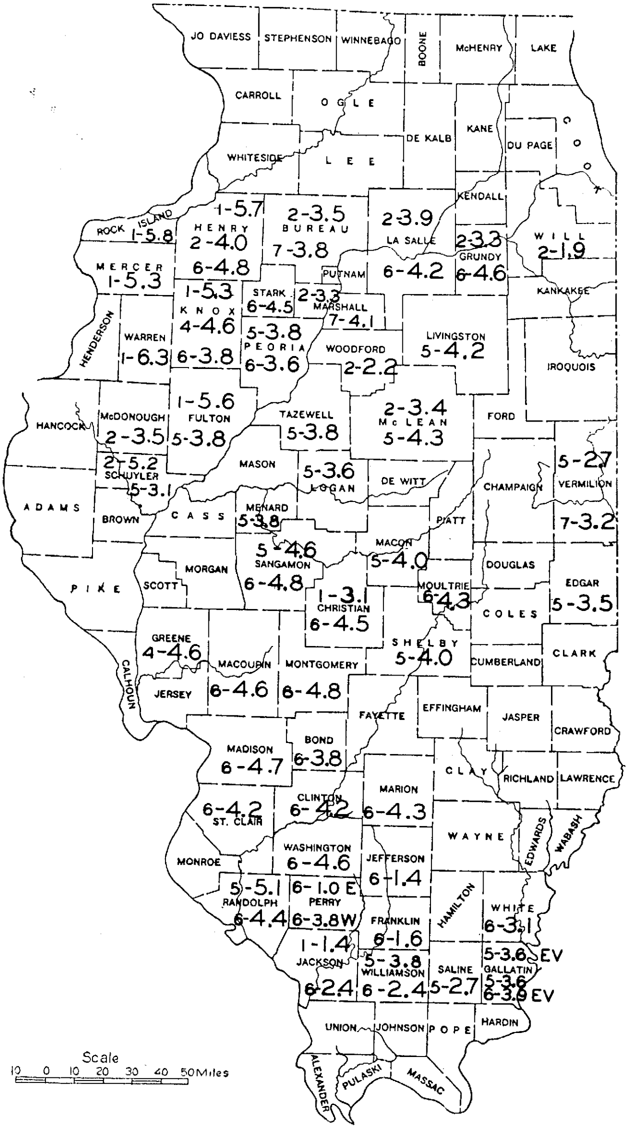


Fig. 19.—Coal Bed Numbers and County Average Total Sulfur (Dry Basis) of Illinois Coals.

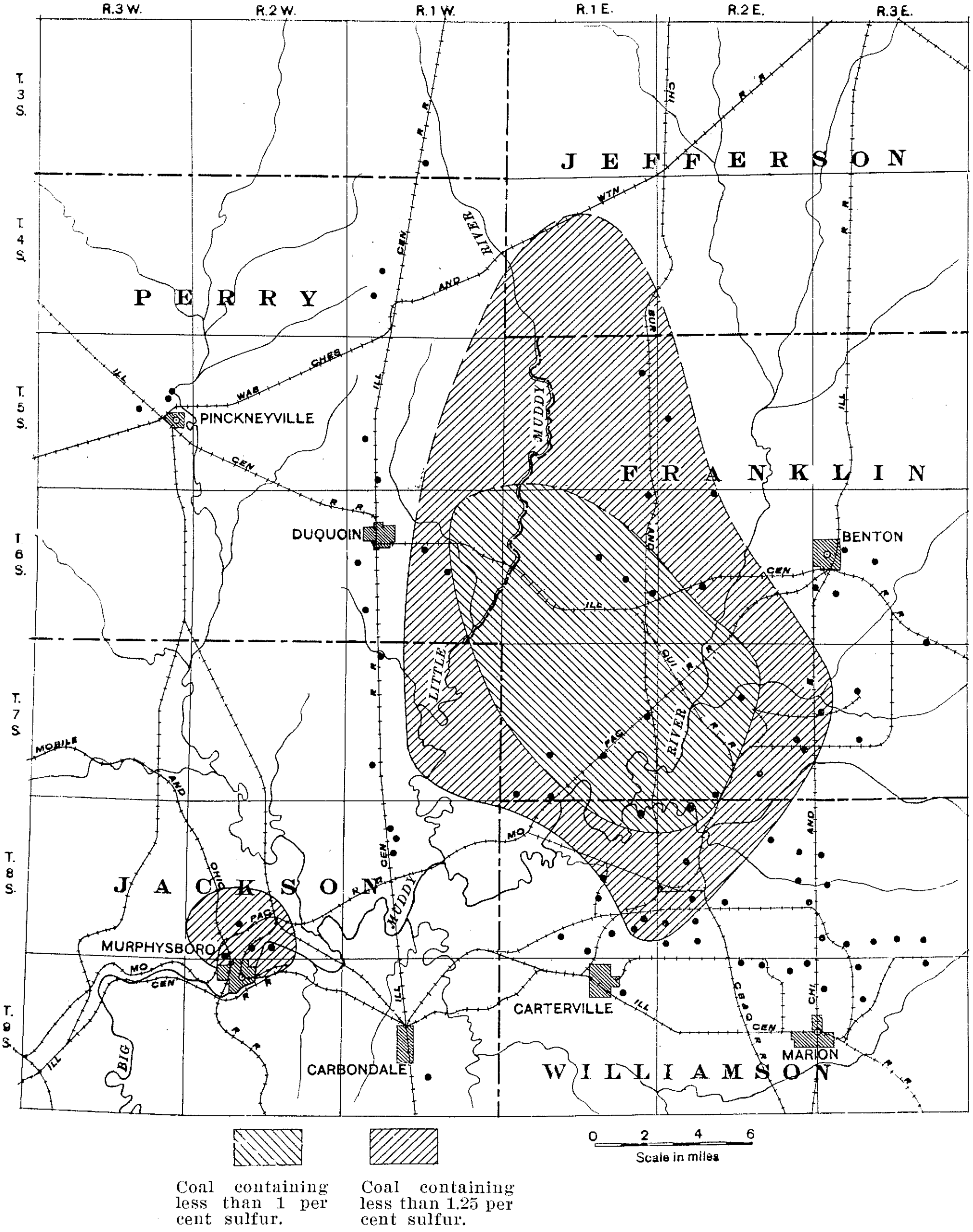


Fig. 20.—Map Showing Location of Low-Sulfur Coal in the Franklin-Williamson and Big Muddy Districts.

The Big Muddy District in Jackson County has been worked out. (*Cady, G. H., Op. cit.*)

CLASSIFICATION OF ILLINOIS COALS WITH REFERENCE TO COKE-MAKING PROPERTIES ON THE BASIS OF THEIR PROXIMATE AND ULTIMATE ANALYSES

Systems for classifying coals in groups having similar properties on the basis of their analyses or the results of various tests applied have been proposed as aids in the selection of coals for given purposes. Such classification systems have been found to be of considerable practical value and are being used to an increasing degree. Some of the proposed classification systems were applied to Illinois coals in an effort to determine the relative positions of Illinois coals with respect to each other and to other coals. The various classification systems which have been proposed are not of equal value in evaluating coals for the production of coke, and in some cases it is not certain whether the system will evaluate coals for coke making. An attempt is made to indicate the value of the classification systems to be discussed for purposes of estimating the coking characteristics of coals.

Rank of coal.—The position a coal occupies in the series from peat to anthracite is known as its rank. Increase in rank in coal is due to various geological factors which are not discussed here. Progressive change in the rank of coal causes a progressive change in properties, other things, such as type, being equal. Coals of the same type and the same rank can, in general, be assumed to have the same properties. The lower ranks of coal do not fuse and form a coherent cellular coke. This property appears with the bituminous rank and is one of the characteristics which distinguish bituminous from sub-bituminous coals. The coking characteristics of coals increase with rank through a maximum in the low volatile coals and disappears again in the anthracite rank.

With few exceptions, Illinois coals fall in the high-volatile B (moist mineral-matter-free calorific value of 13,000 or more, and less than 14,000 B.t.u. per pound) or high-volatile C (moist mineral-matter-free calorific value of 11,000 or more, and less than 13,000 B.t.u. per pound) bituminous groups of the A.S.T.M. tentative specifications for classification of coals by rank, D 388-36 T.[5]

To be placed in group C, rather than in the lower sub-bituminous group, coals must be either agglutinating or non-weathering. To be agglutinating they must form a coherent coke button during the standard volatile matter test. All Illinois coals so far tested are agglutinating coals. They are placed in the bituminous rather than in the sub-bituminous rank by virtue of their established coking quality.

Coals of the high-volatile B group are found only in the southern part of Illinois. Some coals belonging to the high-volatile A bituminous group (having

---

[5] Amer. Soc. for Testing Materials Tentative Specification for Classification of Coals by Rank, A.S.T.M. Designation D 388-36 T., A.S.T.M. Tentative Standards 1936, pp. 520-26, 1936.

a dry mineral-matter-free fixed carbon content of less than 69 per cent and a moist mineral-matter-free calorific value of 14,000 or more B.t.u. per pound)

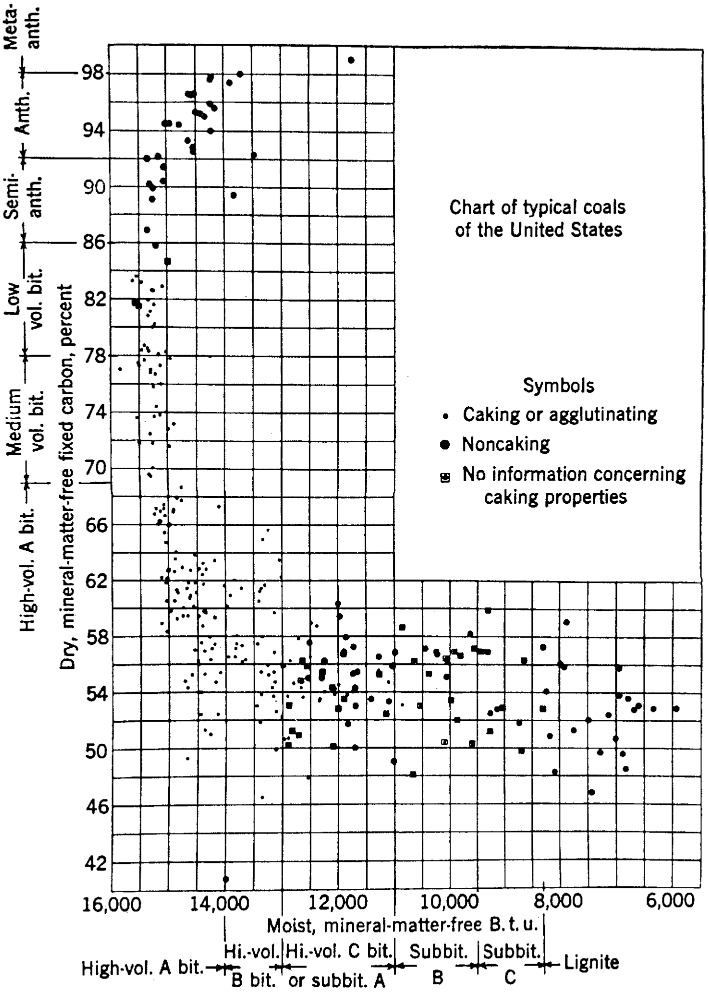


Fig. 21.—Classification Chart of Typical Coals of the United States. *(From Fieldner, Selvig, and Frederick, Op. cit.)*

are found in a small area in southern Gallatin and southeastern Saline county, known as Eagle Valley, where there are no shipping mines. There may be small bodies of such coal at other places along the southern margin of the coal basin.[6]

---

[6] Cady, C. H., Classification and selection of Illinois coals: Illinois State Geol. Survey Bull. 62, p. 33, 1935.

Rank may be indicated graphically on a chart upon which fixed carbon on the dry mineral-matter-free basis is plotted against the moist mineral-matter-free calorific values of a coal, as has been done by Fieldner, Selvig, and Frederic[7] (fig. 21). The plotting of a large number of values representing a wide variety of coals results in a chart upon which the positions of the coals lie within a fairly narrow band in the diagram. In this diagram the position of the high volatile bituminous coals should be observed, as this is the position occupied by Illinois coals. An enlarged portion is used to plot the position of the county

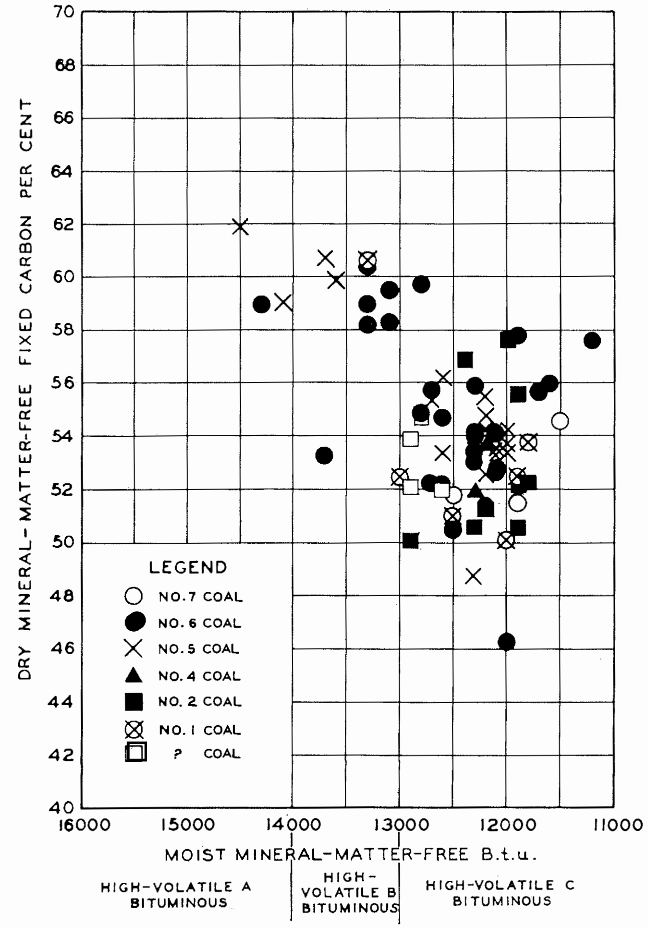


Fig. 22.—Classification Chart of Illinois Coals According to A. S. T. M. Tentative Specifications for the Classification of Coals by Rank, D 388-34T.

---

[7] Fieldner, A. C., Selvig, W. A., and Frederic, W. H., Classification chart of typical coals of the United States: U. S. Bureau of Mines Rpt. of Inv. 3296, 1935.

averages of Illinois coals, first in such a way as to differentiate the coal beds (fig. 22), and then to differentiate the position in the state, northern, central or southern (figs. 23 and 24).

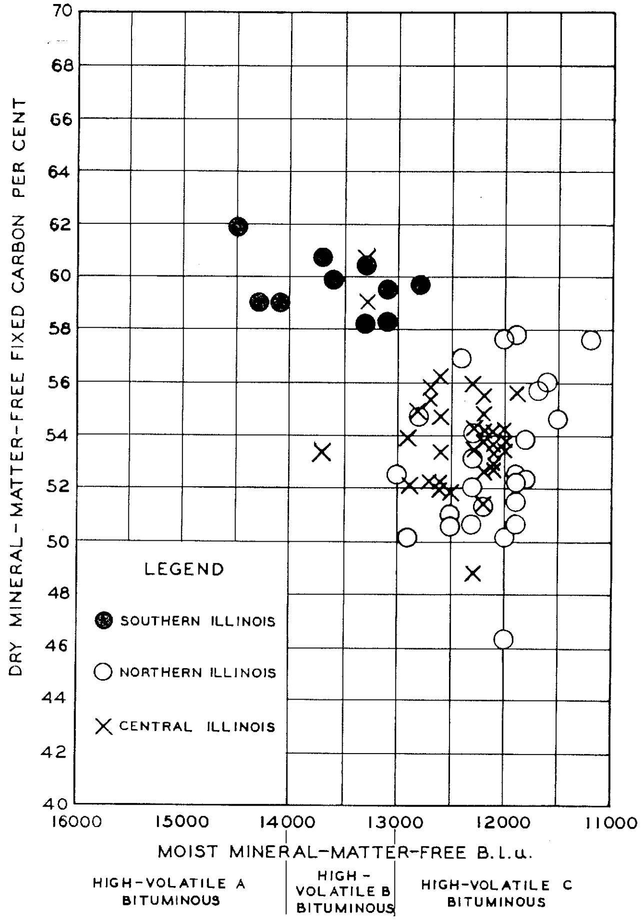


Fig. 23.—Classification Chart of Illinois Coals, by Geographic Location, According to A. S. T. M. Tentative Specifications for the Classification of Coals by Rank, D 388-34T.

Coals from the same parts of the state show a greater tendency to have the same rank than do coals from the same bed irrespective of their location. The Illinois coals which have received the most attention as possible coking coals have been from the southern counties and as a group are the coals of highest rank in the state.

Ultimate items.—When ultimate coal analyses are plotted on a triangular diagram, the coals arrange themselves across a part of the diagram in a narrow band with coals of high rank at one end and low rank at the other, while coals of similar properties are usually found together (fig. 25). Such a plot is most

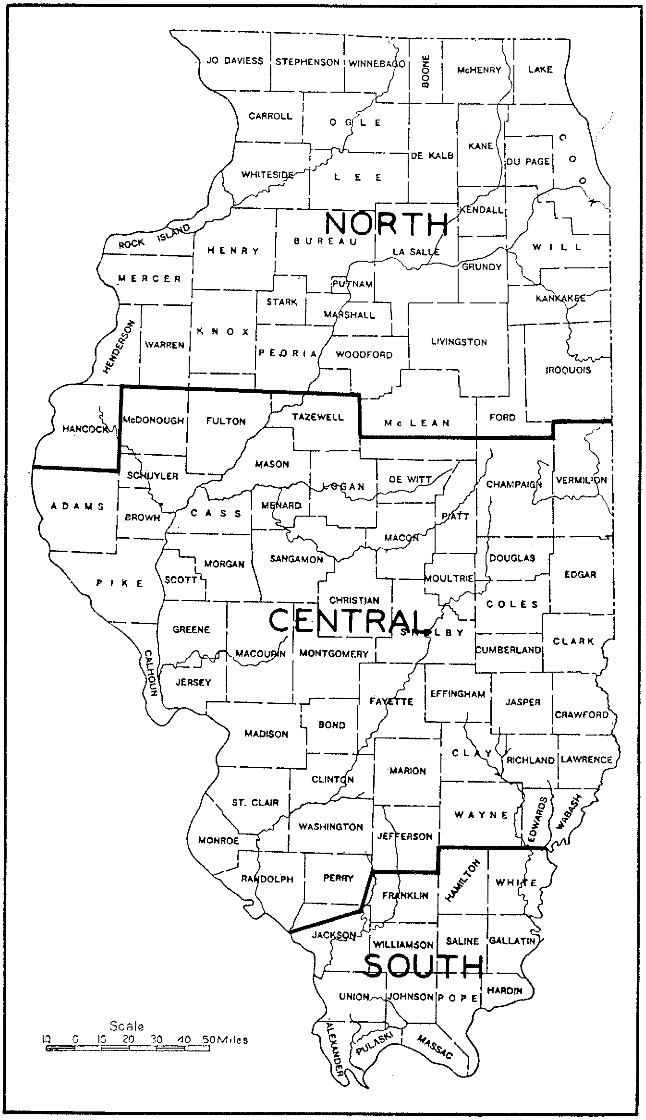


Fig. 24.—Division of State into North, Central and South Areas.

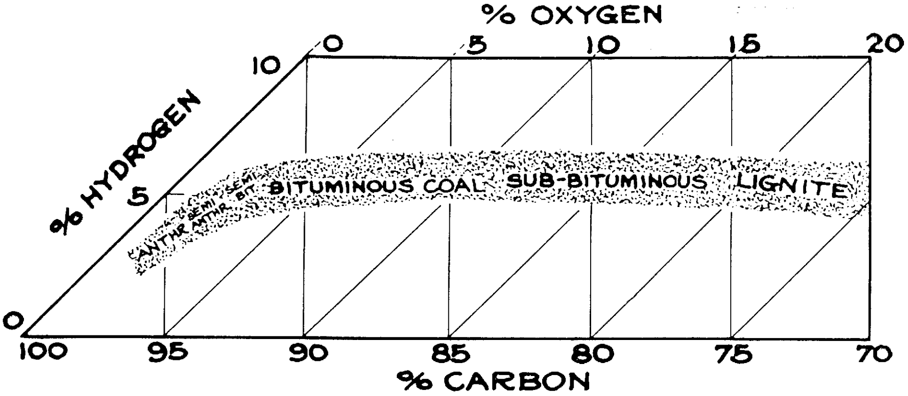


Fig. 25.—Three-component Diagram of Ultimate Analyses of United States Coals. *(From Rose, Op. cit.)*

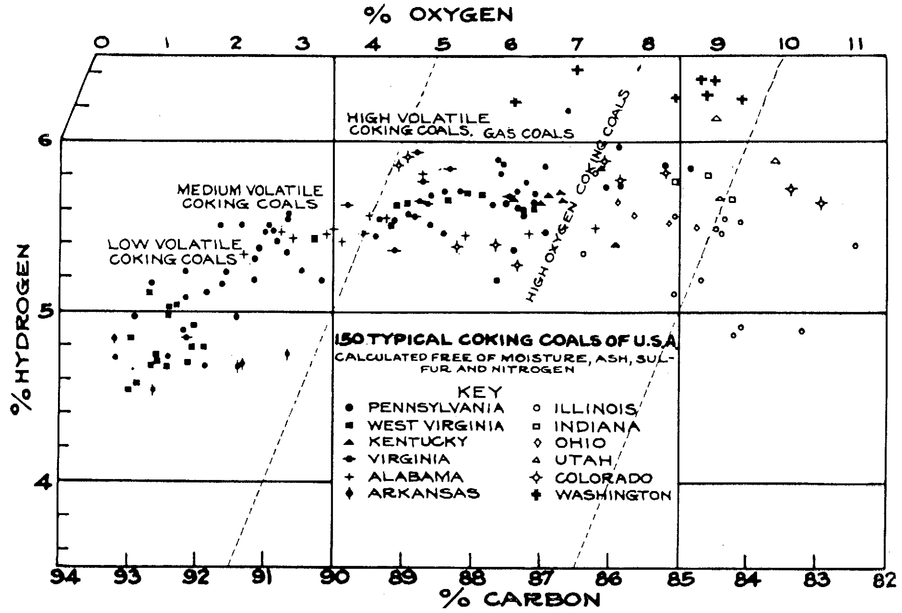


Fig. 26.—Three-component Diagram of Ultimate Analyses of 150 Coking Coals of the United States. *(From Rose, Op. cit.)*

useful if the analyses have first been calculated to a dry mineral-matter and sulfur- and nitrogen-free basis. H. J. Rose has published such a diagram showing 150 coking coals of the United States, with the states of origin of the coal indicated.[8] This diagram is reproduced in figure 26. Figure 27 is a similar plot showing the positions of the county average ultimate analyses of the various coals of Illinois for which analyses are available.[9] These analyses are also shown on an enlarged scale and differentiated according to coal bed in figure 28 and according to their geographic location in figure 29. Rose says: "All coking coals appear to fall within fairly definite chemical limits, so far as their ultimate analyses are concerned, and it is the writer's experience that from the location of a coal within these limits, the coking properties and by-product yields may be predicted with reasonable accuracy, *if all essential data on the chemical and physical characteristics of the coal as it will be charged, and the operating conditions which will exist, are known.*" The location of the coals in the United States which Rose, on the basis of his experience, classes as coking coals are shown in figure 30 taken from his paper[8] and may be compared with the coal fields of the United States shown in figure 31. It will be noted that Rose included only the coals of southern Illinois and Vermilion County with the coking coals. He may have discriminated on the basis of sulfur and ash content, since his area in Illinois includes only the low sulfur and lower ash coals. His area excludes a large area of Illinois coal from which satisfactory domestic and industrial fuel coke may be made.

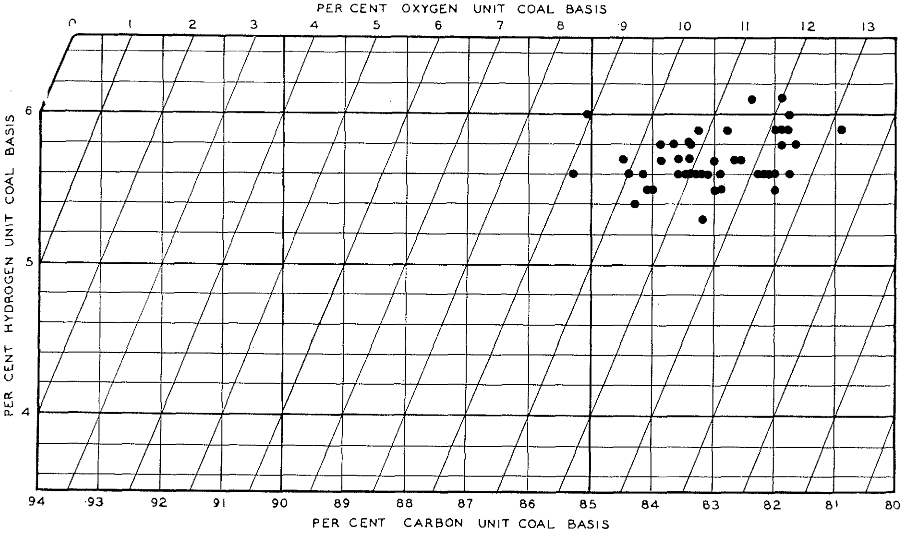


Fig. 27.—Three-component Diagram of Illinois Coal Ultimate Analyses.

---

[8] Rose, H. J., The selection of coals for the manufacture of coke: Trans Amer. Inst. Min. Met. Eng. vol. 74, pp. 600-636, 1926.

[9] Cady, G. H., op. cit. Table 4, pp. 314-26.

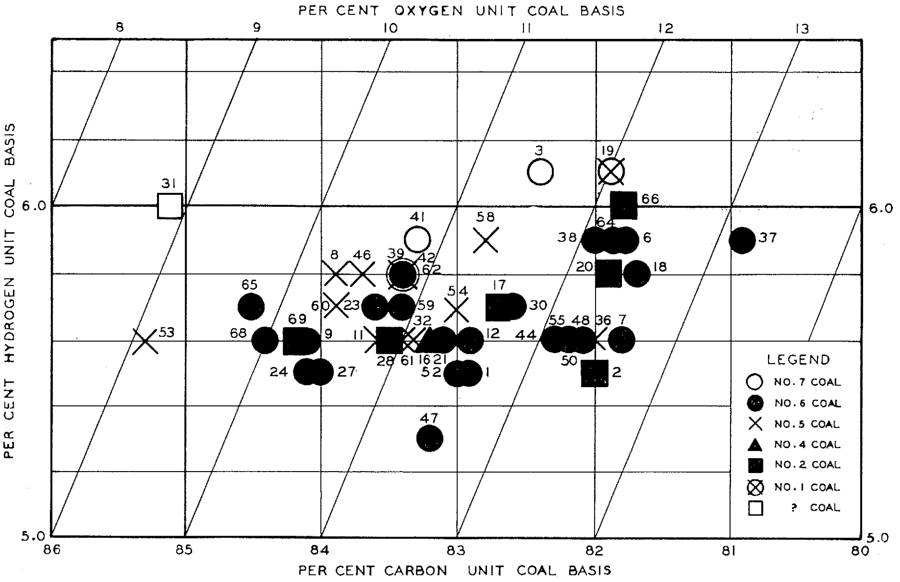


Fig. 28.—Three-component Diagram of Illinois Coal Ultimate Analyses with the Coal Bed Numbers Indicated.
Numbers beside points refer to lines in Table 11.

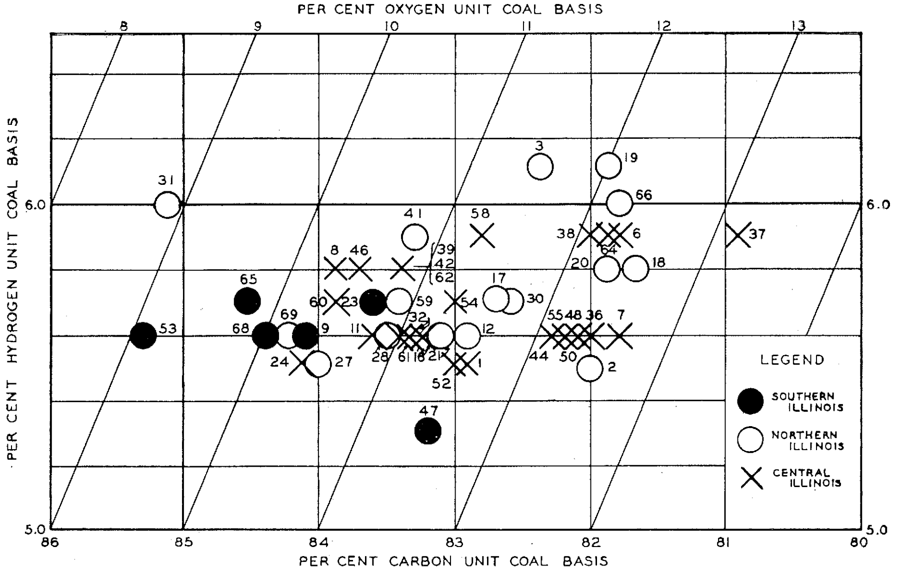


Fig. 29.—Three-component Diagram of Illinois Coal Ultimate Analyses with Location of Coals Indicated.
Numbers beside points refer to lines in Table 11.

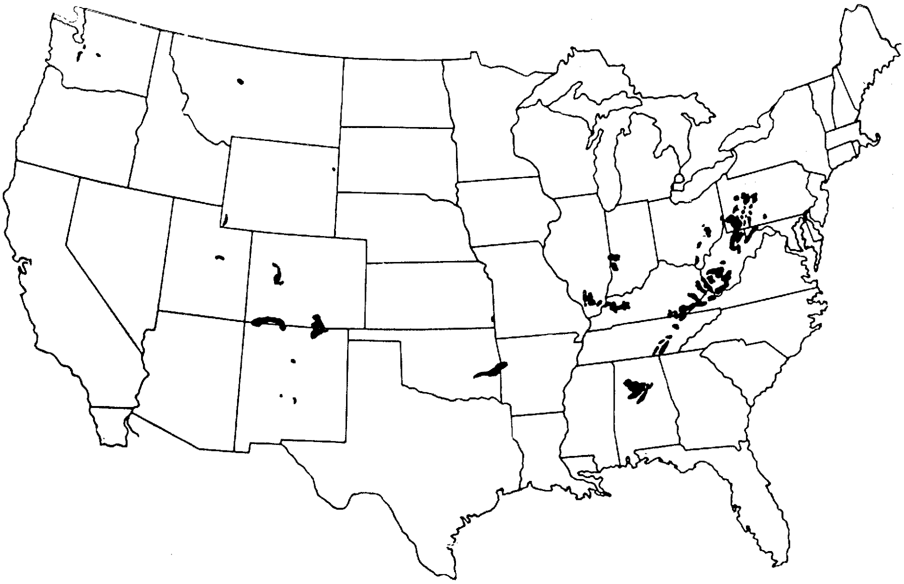


Fig. 30.—MINING DISTRICTS OR COAL AREAS OF THE UNITED STATES WHICH CONTAIN COKING COAL. *(From Rose, Op. cit.)*

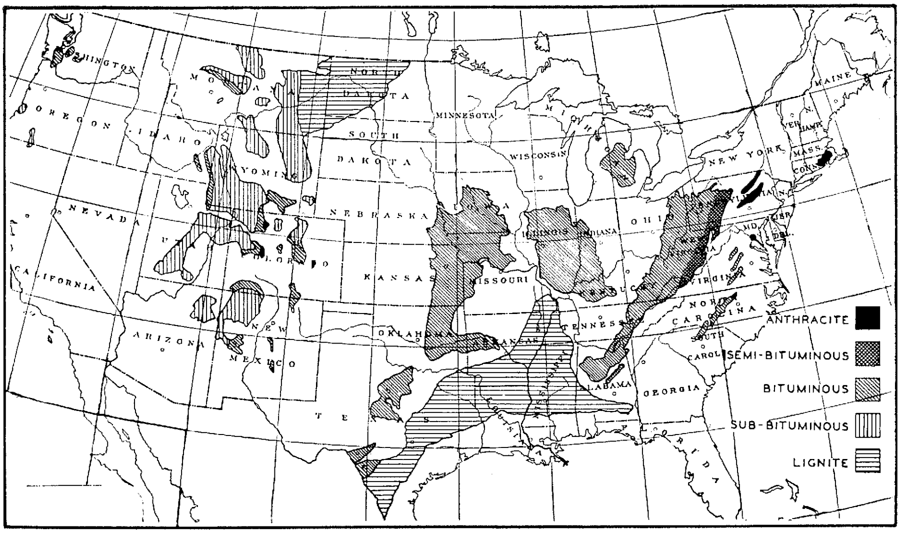


Fig. 31.—COAL FIELDS OF THE UNITED STATES.

From "The Design of Steam Generating Plants" Part II, "The Three Natural Fuels" by F. H. Rosencrants, published in "Combustion," May 1932, *p.* 37, *probably redrawn from original in colors by M. R. Campbell, U. S. Geological Survey.*

Specific volatile index.—For classifying coals, using the items of the proximate analyses, Burrough, Swartzman, and Strong have devised a new value which they term the "specific volatile index",[10] which is the calorific value in B.t.u. per pound of one per cent of the volatile matter in the coal. It is arrived at by subtracting the calorific value of the fixed carbon in the coal from the calorific value of the coal and dividing the result by the percentage of volatile matter in the coal, according to the following formula:

$$\text{S.V.I.} = \frac{\text{Determined B.t.u. in coal} - (14{,}500 \times \text{weight of F.C.})}{\text{Per cent of volatile matter}}$$

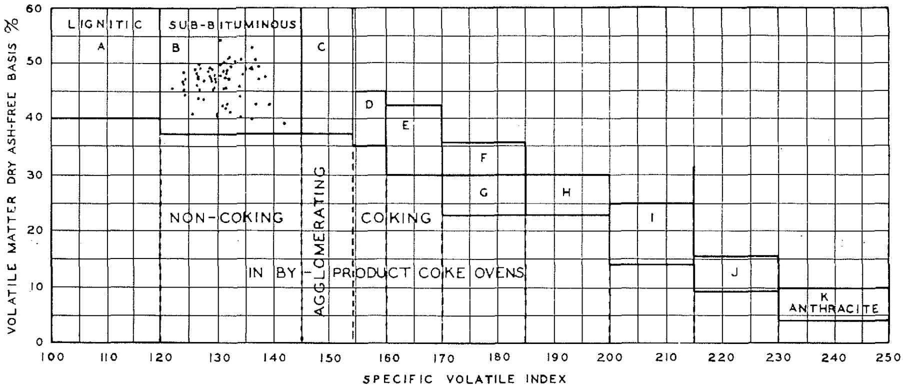


Fig. 32.—Classification of Illinois Coals on the Basis of Specific Volatile Index.

When the specific volatile index is plotted against the volatile matter content of the coal, as expressed on the dry ash-free basis, a continuous band of varying width which includes all coals is obtained. The authors of the system have divided the plot so obtained into various areas which include coals with similar coking characteristics. Their chart, on which the Illinois coals have been plotted using values obtained from county averages published by Cady,[11] is reproduced here in figures 32 and 33 in which the area occupied by Illinois coals has been enlarged.

In this system, as in the others, the points representing the various coals fall in a narrow band across the chart with the Illinois coals grouped closely together on the lower edge of the coking coals. This system does not differentiate coking coals from non-coking coals, some of the non-coking sub-bituminous coals from the United States and Canada lying in the same area with Illinois coals.

---

[10] Burrough, E. J., Swartzman, E., and Strong, R. A., Classification of coals using specific volatile index: Canada Dept. of Mines, Mines Branch No. 725, Fuels and Fuel Testing 1930-31, pp. 36-50, 1933.

[11] Cady, G. H., op. cit.

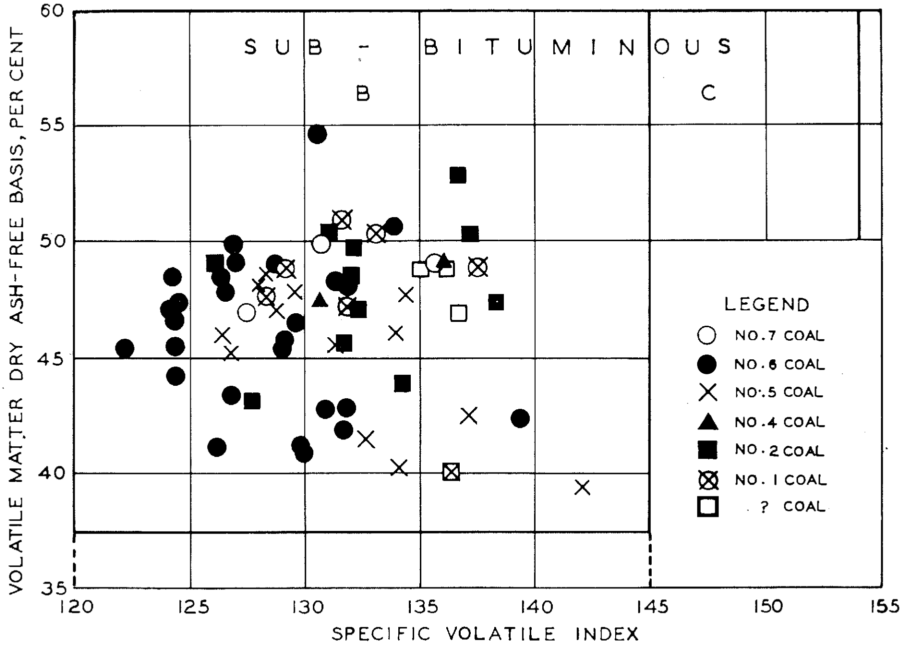


Fig. 33.—Classification of Illinois Coals on the Basis of Specific Volatile Index with the Coal Bed Numbers Indicated.

CONCLUSIONS FROM THE APPLICATION OF VARIOUS CRITERIA FOR COKING COALS TO ILLINOIS COALS

Definitions of coking coals and classifications which claim to provide definite criteria for separating coking from non-coking coals are based in general upon the average characteristics of coking coals. Since most coke has been prepared for metallurgical purposes, it is the characteristics of coals which produce that kind of coke that have provided the criteria for determining coking coals. Applying the criterion that coking coals are coals from which a saleable commercial coke can be made, much of the coal in Illinois has definitely been shown to be coking coal. This is especially so in view of the growing market for non-metallurgical coke for which the requirements are different than for blast-furnace coke. The coals from southern Illinois usually occupy a more favorable position with respect to the established coking coals when plotted in the various systems of classification than do the coals from central and northern Illinois. The differences between the various Illinois coals are, however, small in comparison to the range of variations possible among the coking coals.

Part IV — Experimental Investigation of the Production of Coke from Illinois Coals

By Gilbert Thiessen and Paul E. Grotts

CHAPTER IX—INTRODUCTION TO THE EXPERIMENTAL WORK

In spite of the work which has been done toward the production of coke from Illinois coal, very little definite information is available, particularly concerning the comparative coking characteristics of coals from different parts of the state. Many of the tests from which results are available were made in apparatus which would no longer be used, for example, in beehive or special types of ovens. Many tests were made in connection with the development or promotion of new types of carbonizing equipment, the conditions and results of which are not available or only in a general way. Many economic and technical changes have also occurred since the problem of the production of coke from Illinois coals was last reviewed by the Illinois State Geological Survey.[1] It appeared desirable, therefore, to assemble a systematic body of data in regard to the coking characteristics of a variety of Illinois coals, at the same time having the advantage of newer experimental methods, for the sake of providing fundamental information concerning the coking characteristics of these coals. Although these data provide general indications of the coking characteristics, much yet remains to be done before we can say that we thoroughly understand the mechanism of coke formation from any coal, not only Illinois coal.

In determining the coking quality of a coal, small-scale experimental tests provide essential information not supplied by the standard types of commercial analysis. Proximate and ultimate analyses provide values on which probabilities can be based, as was shown in the previous chapter; but for border-line coals, particularly such as those found in Illinois, additional factors are important. That is to say, differences among coals cannot be completely revealed by the standard types of analysis. This is particularly true with respect to those differences arising from variations in the physical constitution of the coal, known as type differences, since variations in chemical constitution have not been sufficiently well correlated with such differences. Experimental tests are,

---

[1] Ovitz, F. K., Coking of Illinois coals: U. S. Bureau of Mines Bull. 138, 1917.

therefore, necessary to determine the exact behavior of a coal, a condition which will continue until the relationships between type differences and the behavior of coals under the influence of heat are more definitely understood.

Because of the expense involved, full-scale tests are usually out of the question even if ovens were available. There is, however, some compensation in the use of small-scale tests in the fact that such tests permit much greater freedom in controlling conditions of operation and, therefore, in determining the extent to which the coking characteristics of a coal may be modified, at least in a general way, under a variety of conditions.

On the other hand, it is well to keep in mind the statement by H. J. Rose[2] that ". . . the only thoroughly reliable method to determine the quality of coke that can be made from a coal under particular conditions is to subject the coal to a full-scale oven test under the desired conditions."

---

[2] Rose, H. J., the selection of coals for the manufacture of coke: Trans. Amer. Inst. Min. Met. Eng. vol. 74, pp. 600-636, 1926. (Especially 604.)

CHAPTER X—METHODS USED IN THE EXPERIMENTAL WORK

SELECTION OF METHODS

Some methods for testing coal, particularly those used in the proximate and ultimate analyses, have been highly standardized and widely used. A high degree of standardization of methods is essential in coal testing if the results from different laboratories and from tests at different times are to be comparable, since most of the methods are highly empirical. Most of the methods used in studying the coking properties of coals have not been so standardized, partly because they are relatively new and untried as only a few laboratories have use for them, and in many cases the essential factors in the methods have not yet been discovered. The investigator must, therefore, select his methods cautiously. One of the factors considered in the selection of the methods used in this work was the extent to which they had been adopted and used in the United States. Those adopted include methods set forth in the standard and tentative standard methods of the American Society for Testing Materials, in the "Methods of the Chemists of the United States Steel Corporation for the Testing of Coal, Coke and By-Products," and in the reports of the United States Bureau of Mines on the work in cooperation with the American Gas Association on the coke, gas and by-product making properties of American coals.

CHEMICAL ANALYSES

The methods for the chemical analysis of coal have been well standardized in the United States. Methods used in this investigation were the standard methods as set forth in the A.S.T.M. Standard Methods of Laboratory Sampling and Analysis of Coal and Coke, A.S.T.M. Designation: D 271-30. These methods were carried out in strict conformity with the methods as set forth, both as to procedure and equipment. As a check on the accuracy of the work, samples were periodically exchanged with the Coal Analysis Laboratory of the United States Bureau of Mines at Pittsburgh.

The proximate analyses, calorific values and sulfur contents of all the coals used in these tests were determined in order to evaluate them as to quality and rank and to make easier their comparison with other coals.

TESTS INDICATING THE BEHAVIOR OF COAL UNDER THE INFLUENCE OF HEAT

Coal when heated loses first moisture, then a portion of its volatile matter, and then it softens and becomes plastic, at which stage decomposition increases greatly and volatile matter is liberated. In the plastic stage the individual particles may completely fuse together, may merely sinter (stick together), or may remain completely free. The behavior of the coal while plastic is dependent upon whether or not the particles fuse together. When complete fusion occurs at the same time that a considerable proportion of the volatile matter is given off a much swollen coke is produced which, if permitted to expand freely, may be several times the volume of the original sample. If fusion does not take place the charred product will be smaller in volume than the original coal, due to loss of volatile matter. As heating is continued the sample loses more volatile matter and finally reaches a temperature at which the plastic coal, regardless of whether fusion has occurred, sets to a solid. Further heating of the coke results in its devolatilization and in a slight shrinkage. The temperatures at which the coal particles are most plastic and the temperatures at which the coal decomposes most rapidly may not be the same. Their relationship to each other is an important factor in production of coke from a given coal. The temperature range during which a coal is plastic is also important when blends of coal are coked, because the various coals in the blend should be plastic at similar temperature ranges.

From the standpoint of operating practice, the yields and quality of the coke, gas, tar, light oil, and other by-products of those coals which will produce coke are important. From the standpoint of the present investigation the ultimate interest concerns whether or not coke of quality suitable for use as a domestic fuel may be produced from a given coal. Certain conventional tests are used in judging the quality of coke.

On the basis of the above considerations, tests were selected to yield the following information:

(1) The yields of coke, gas, tar, and other by-products obtainable from a given coal, together with some information concerning the analysis of the coke and gas

(2) The temperature at which the coals tested became plastic

(3) The temperatures at which the plastic coals set to form coke

(4) The coking or agglutinating power of the coals, that is, the ability of the individual coal particles to form a coherent mass

(5) The quality of the coke obtainable under experimental conditions from a variety of Illinois coals and blends of coals

(6) The influence and behavior of various impurities in coal on the quality of the resulting coke

Gas, coke, and by-product yields.—Coke and by-product yields were determined using a procedure and equipment adopted by the laboratories of the United States Steel Corporation[3] with a few modifications developed in the course of this work to facilitate duplication of results.

A detailed description of the apparatus and method as used and the procedure adopted is given in Appendix A (pp. 209-223).

Transparent fused quartz combustion tubes were used in place of the hard glass combustion tubes, as it was found difficult to obtain glass which would retain its shape at the required temperature of 900°C. The quartz tubes were exactly the same dimensions as specified for the glass tubes. One alteration of procedure consisted of the introduction of a snugly fitting piece of transparent fused quartz tubing 128 mm. long into the open end of the combustion tube. Removal of this tube with the tar deposited in it took the place of cutting off this portion of the glass tube in the ordinary procedure. The tar collecting between the two snugly fitting tubes was recovered with negligible loss. The inner tubes projected into the tar filter.

The directions call for heating the portion of the charged combustion tube containing the crushed silica brick to the constant temperature of 720°C. It was found difficult to maintain this temperature uniformly with gas heat. A small electric heater was built to take the place of the first four burners. The use of this electric heater greatly simplified the operation of the equipment. Four thermo-couples were used to measure and automatically record on a 4-point recording potentiometer pyrometer the temperatures in the electric furnace and at the two ends and center of the coal charge. Light oil was recovered by the freezing tube method using a mush of acetone and dry ice to produce a low temperature. When these tests were first started the laboratories were supplied with artificial gas which produced a hot concentrated flame. Shortly after the tests started, however, the gas supplied was changed to natural gas which gave a slow flame that made it difficult to localize the heat. To overcome this difficulty arrangements were made for mixing air and gas before the mixture was passed into the burner manifold. Both the air and gas lines were equipped with orifice flow meters. The proportion of air and gas which would give the required rise of temperature to 900°C. in ten minutes was found by trial and this proportion was maintained during subsequent tests. The mixture of air and gas had burning characteristics similar to the artificial gas previously supplied. The greatest difficulty encountered in carrying out these carbonization tests was in obtaining tar yields comparable to those obtained in by-product oven practice. In the results presented in another section it will be noticed that the tar yields are low by about one-half.

---

[3] Methods of the chemists of the U. S. Steel corporation for the sampling and analyses of coal, coke, and by-products, 3rd Edition, 1929. Dry distillation test for coal, pp. 130-143.

Softening and swelling characteristics.—The apparatus and procedure used for determining softening and swelling properties of the coals which include a determination of the initial softening temperature, the initial active decomposition temperature, the temperature of maximum swelling, the temperature of volatilization, and the temperature range during which the coal was plastic was that designed by the United States Bureau of Mines after Agde and Damm,[4] illustrated in figure 34. A detailed description of the apparatus and procedure as will be found in Appendix B (pp. 225-226). The relationship between the plastic temperature range and the liberation of volatile matter from the coal was determined as follows:

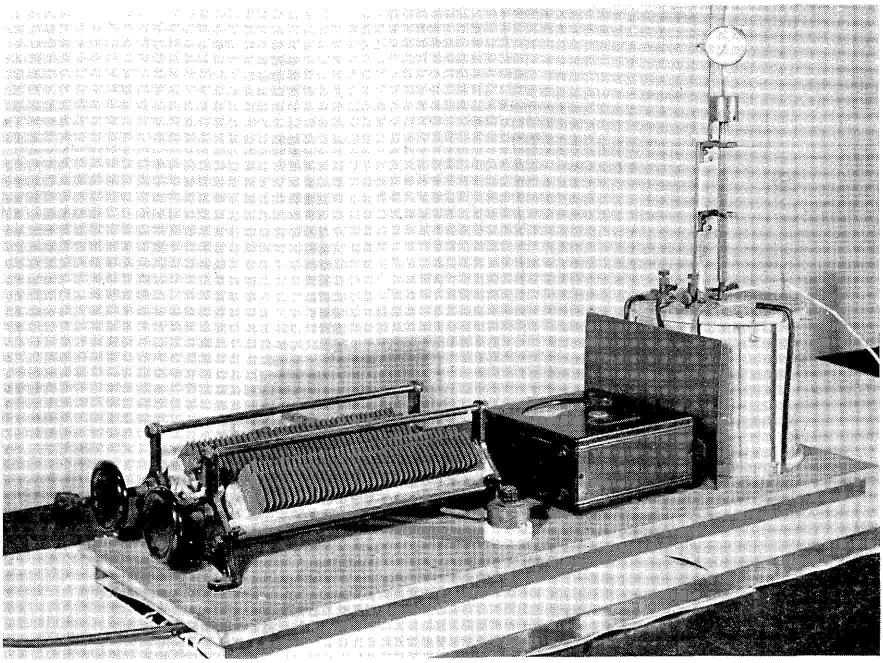


Fig. 34.—AGDE-DAMM APPARATUS.

---

[4] Fieldner, A. C., Davis, J. D., Thiessen, R., Kester, E. B., and Selvig, W. A., Methods and apparatus used in determining the gas, coke, and by-product making properties of American coals: U. S. Bureau of Mines Bull. 344, pp. 14-16, 1931.

A support was constructed so that a Westphal specific gravity balance could be supported with its hook directly above the opening of a standard coal volatile matter furnace. The standard volatile-matter platinum crucible was suspended centrally in the furnace from a long stirrup attached to the balance hook. Convection currents were minimized by covering the furnace opening with a series of perforated mica plates. One gram of coal was placed in the crucible, the capsule lid firmly put into place and the crucible suspended by the stirrup in the cold furnace. By means of weights the beam was put into balance. The temperature of the furnace was gradually raised at the uniform rate of 6°C. per minute. At intervals of one minute the beam was put into balance and the loss of weight determined. The temperature in the furnace was measured at the same time by means of calibrated chromel-alumel thermocouples. From these readings a graph was plotted which showed the evolution of volatile matter with temperature change. The apparatus is illustrated in figure 35.

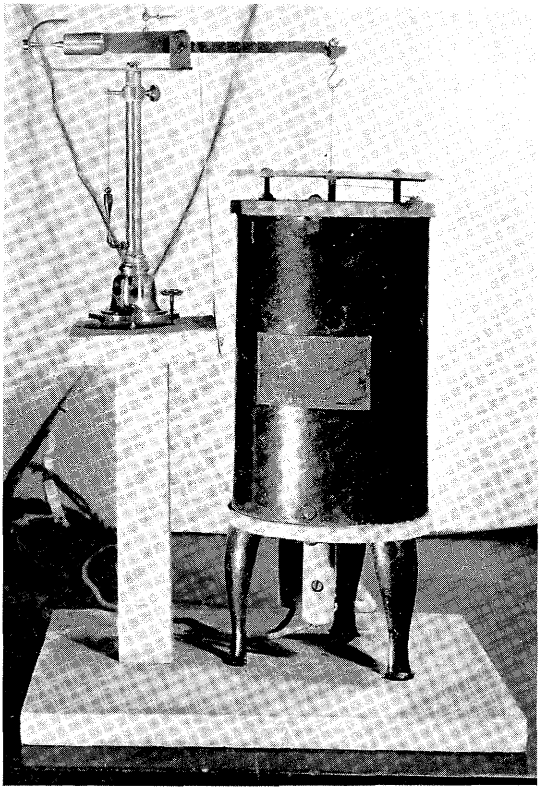


Fig. 35.—Apparatus for Determining Volatile Matter Loss.

Agglutinating or caking power.—The caking or agglutinating power of a coal is its ability to cement itself and inert material into a coherent mass upon carbonization. To measure this property the finely ground coal must be diluted with a sufficient amount of suitable inert matter until only the cementing power of the coking coal is effective. Insufficient dilution permits the formation of coke cells which cause anomalous and untrustworthy results. A number of such test procedures have been described in the literature.[5]

This type of test has been extensively studied at the U. S. Bureau of Mines[6] with the result that a modification of the Marshall-Bird test was devised and published by the A.S.T.M. as a proposed method.[7] Wider use of this test brought to light the fact that the values obtained by use of the test are very dependent upon the surface condition of the sand grains used as inert material and that it is practically impossible to supply a sand which would be satisfactory except at a prohibitive price. Accordingly, results by various laboratories are comparable among themselves as long as the same batch of sand is used. Values varying two-fold have been obtained using different sands. The Marshall-Bird test[8] was first used in the study of Illinois coals until the Bureau of Mines modification was developed and adopted. This method as used is described in detail in Appendix C.

Coke quality tests: retort for carbonizing two- to three-kilogram charges of coal.—As only small amounts of coke were obtainable from the United States Steel Corporation by-product apparatus, it was necessary to construct a special retort to yield a sufficient quantity of coke from which an estimate of its quality could be obtained. A cylindrical steel retort was constructed from a piece of 6-inch steel pipe 9 inches long. A sheet of steel of the same thickness as the walls of the pipe was welded to one end to form a bottom, the other end of the pipe was threaded to take a 6-inch pipe cap. Two holes were drilled in the cap cover, one centrally located to take a 1-inch pipe fitting and the other 2 inches from the center to take a ¾-inch fitting. A one-inch nipple carrying a pipe cross at the other end was screwed into the centrally located hole.

---

[5] Anonymous, Methods of analysis of coal: Dept. of Sci. and Ind. Research, Great Britain, Fuel Research, Physical and Chemical Survey of the National Coal Resources, No. 7, pp. 10-12, 1927 (reprinted 1929).

Gray, T., The determination of the caking power of coal: Fuel, vol. 2, No. 3, pp. 42-45, 1923.

Kattwinkel, R., The examination of coking coals and estimation of their value: Fuel vol. 5, No. 8, pp. 347-355, 1926.

Marshall, S. M., and Bird, B. M., Test for measuring the agglutinating power of coal: Trans. Amer. Inst. Min. Met. Eng. Coal Division 1930, vol. 88, pp. 340-388, 1930.

Marshall, S. M., and Bird, B. M., Agglutinating, coking and by-product tests of coals from Pierce County, Washington: U. S. Bureau of Mines Bull. 336, 1931.

[6] Selvig, W. A., Beattie, B. B., and Clelland, J. B., Agglutinating value test for Coal: Proc. Amer. Soc. Testing Materials, vol. 33, pt. II, pp. 741-760, 1933.

[7] Report of Committee D-5 on Coal and Coke. Proc. Amer. Soc. Testing Materials, vol. 34, pt. I, pp. 457-, 1934.

[8] Marshall, S. M., and Bird, B. M., op. cit.

Pieces of pipe screwed into the horizontal openings of the pipe cross served to support the retort in the gas-heated pot furnace and to carry off the carbonization gases. Stuffing boxes were fitted into the top of the cross and into the ¾-inch opening to hold fused quartz thermocouple tubes in place. Temperatures in the retort were continuously recorded during the course of the test by means of a recording potentiometer pyrometer and chromel-alumel thermocouples in the thermocouple tubes so that the couple was about half way down the charge. No effort was made to recover the gases or other by-products. Several of these retorts were kept on hand so that one could be charged while another was being heated.

Coke shatter tests.—The resistance of the coke to shattering was determined by means of equipment similar to the A.S.T.M. shatter test for coke but made smaller in order to be comparable to the size of the sample. The box holding the coke was 12 inches square and 6 inches deep fitted with two hinged doors which open from the center upon the release of a catch. The coke was dropped 6 feet onto a steel plate. Before the test the coke samples were sized on three screens having square openings of 2 inches, 1 inch, and ½ inch. The shatter test was made on the coke retained by the 1-inch screen, and after each of the four drops the coke was sized and the fractions weighed. From these weights curves were drawn which showed the degradation of the coke.

CHAPTER XI—SAMPLES USED IN THE INVESTIGATION

In the course of the work carried on in the present study of the coke making properties of Illinois coals, samples of coals from 18 counties in Illinois were used. The various tests to which the samples of coal from the various counties were subjected are indicated in Table 12 which also indicates where the results of the various tests may be found in the report. In almost all cases samples from several places in each county were used. The tests indicated as having been

TABLE 12.—TABULATION OF TESTS APPLIED TO THE ILLINOIS COALS INVESTIGATED AND LOCATION OF THE RESULTS IN THE TEXT
(X indixates that test was applied)

County[a]	Coal No.	Dry distillation	Caking value	Plastic range	6-inch retort[b]	Basket test in Knowles oven
Bureau	7	—	X	X	—	—
Christian	6	X	—	—	—	—
Franklin	6	X	X	X	X	X
Grundy	7-Streator	—	X	X	—	—
Henry	6	—	X	X	—	—
Knox	6	—	X	X	—	—
Macoupin	6	X	—	—	—	—
Menard	5	—	X	X	—	—
Montgomery	6	X	—	—	—	—
Perry	6	X	—	—	X	—
Randolph	6	X	X	X	X	—
St. Clair	6	X	—	—	—	—
Saline	5	X	X	X	X	—
Stark	6	—	X	X	—	—
Vermilion	6	X	—	—	—	—
Vermilion	7	—	X	—	—	—
Washington	6	X	X	—	—	—
Williamson	6	X	—	—	—	—
Woodford	2	—	X	X	X	—

[a] More than one sample from each county may be represented.
[b] These coals were also used in blends as well as separately.

Test	Results discussed on pages—	Results presented in	
		Table No.	Figure No.
Dry distillation	99	13	38 – 40
Caking value	111	15	.. – ..
Plastic range	109–111	15	.. – ..
6-inch retort	141–165	24, 25, 26	47 – 53
Basket in Knowles oven	166	27	55 – 60

made on samples of coal from any one county were not necessarily, and probably were not, made on the same sample. The reason for this is that in many cases the various tests were in use at different times during the time the work was under way. Since these Eastern Interior basin coals are rapidly influenced by exposure to air and since most of the tests must be made on fresh samples of the coal if the results are to be used in evaluating the coals for coke making, samples

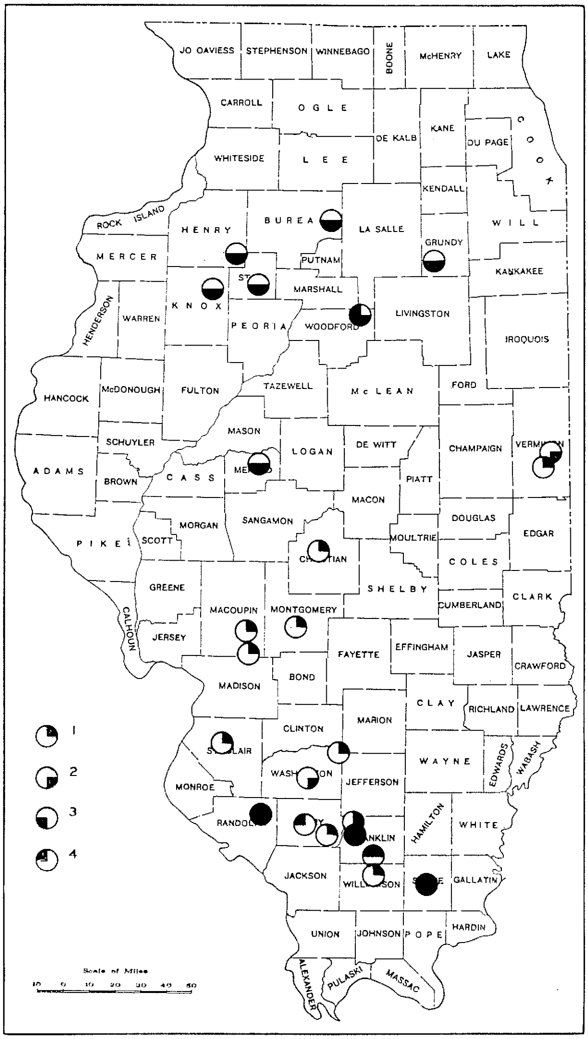


Fig. 36.—Location of the Mines from which Samples were Taken, and the Tests which were Applied.

1. Dry distillation (coal carbonization assay)
2. Caking power (agglutinating value test)
3. Plastic range (Agde-Damm test)
4. Small scale coking test (six-inch retort)

obtained for one series of tests were usually too old or changed by exposure to be suitable for later tests. The uniformity of Illinois coals over geographical areas of considerable magnitude permits a considerable latitude in the selection of samples which may be considered equivalent for many purposes.

The locations of the mines from which the samples used in this investigation were taken and the tests to which these samples were subjected are indicated in figure 36.

CHAPTER XII—COKE, GAS, AND BY-PRODUCT YIELDS

The coke, gas, and by-product yields were determined on samples of No. 6 bed coal from 15 mines and for samples of No. 5 coal from two mines. The proximate analyses, calorific value, rank index,[1] and results of the carbonization tests for these samples are given in Table 13 and the locations of the mines in figure 37.

VARIATION IN YIELDS WITH GEOGRAPHIC LOCATION

Three lines can be drawn so that most of the samples studied lie along or close to them. One of these lines A-A′ (fig. 37) is drawn in a northwest-southeast direction through Belleville in St. Clair County and Harrisburg in the center of Saline County, another, B-B′, in an almost north-south direction from Springfield in Sangamon County to Marion in the center of Williamson County, and the third, C-C′, from Taylorville in Christian County to Belleville in St. Clair County. Distances along these lines have been used as abscissae against which the rank indices, yields of ash-free coke in per cent of ash-free coal, and yields of gas per ton of ash-free coal expressed in therms have been plotted as ordinates in figures 38, 39 and 40. Certain trends are evident from these graphs. Along the line A-A′ the rank indices and coke yields increase and gas yields decrease in going toward Franklin County. The coke yield increases and the gas yield decreases greatly as the DuQuoin anticline is crossed in the eastern part of Perry County. Samples 13 and 14 are from the No. 5 coal and are not strictly comparable to the others.

Along B-B′ (fig. 37) the rank index rises slowly but regularly as does coke yield, while the gas yield drops sharply after the DuQuoin anticline is passed at the western edge of Marion County. From north to south along C-C′ (fig. 37) the rank index rises very gradually carrying with it a very gradual increase in coke yield and a decrease in gas yield.

The well known improvement in rank of Illinois coals in passing from west to east across the position of the DuQuoin anticline and as one continues southeast reveals itself likewise in the results of the determinations of the yields of coke, gas, and by-products. Inasmuch as the more valuable and probably highest grade portion of these coals is rapidly nearing exhaustion, supplies of coal for coke-making purposes will before many years have to be obtained from

---

[1] Cady, G. H., Classification and selection of Illinois coal: Illinois State Geol. Survey Bull. 62, pp. 29-30, 1935.

lower grade or lower rank coals or both. It is important to note, however, that higher gas yields in terms of therms per ton characterize the lower rank No. 6 coal found west of the DuQuoin anticline, although coke yields are lower.

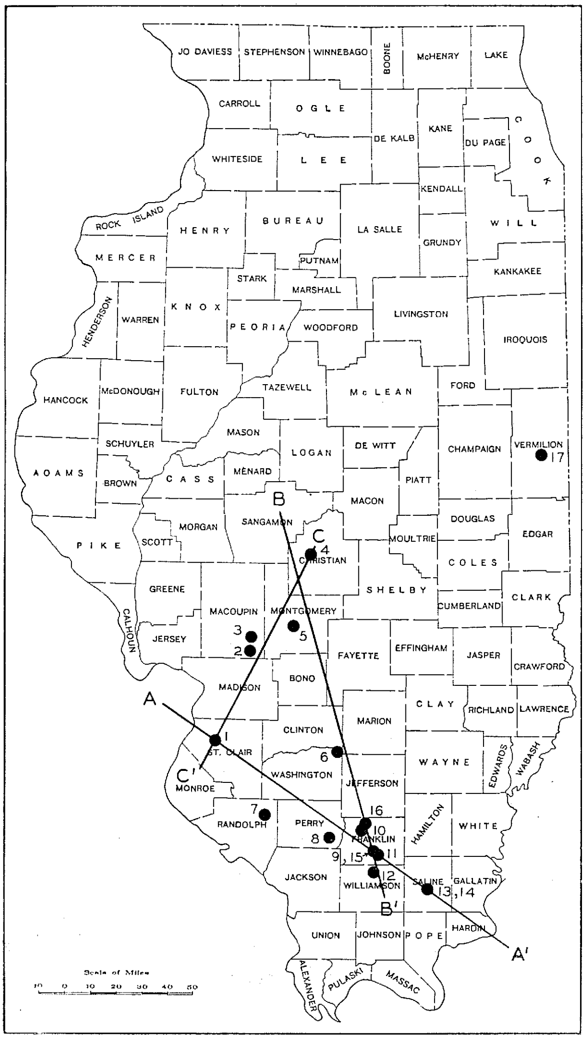


Fig. 37.—Locations of Mines from which Samples were Tested for Coke, Gas, and By-product Yields.

Graphical comparison of coke yields with rank indices and with fixed carbon contents shows that there is only a general trend towards increasing coke yields with increase in fixed carbon content or rank index.

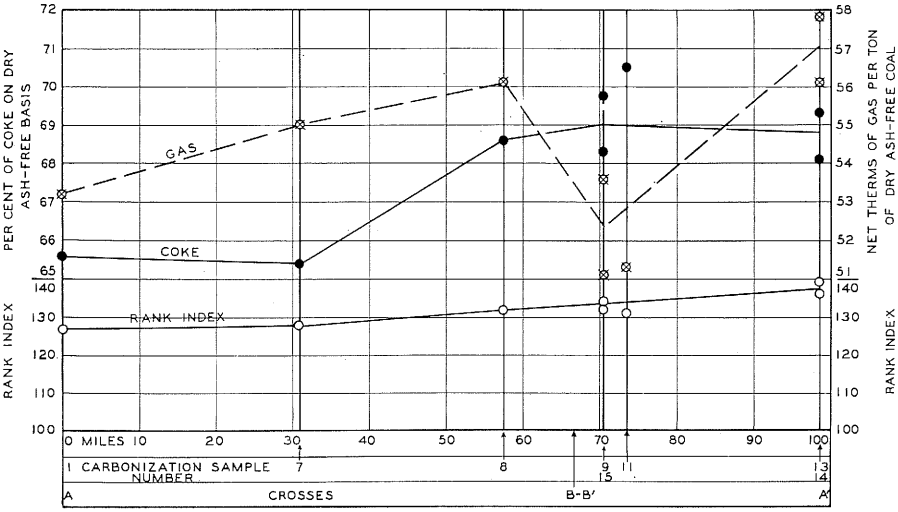


Fig. 38.—Gas, Coke and Rank Indices along the Line A-A' (Fig. 37).

COMPARISON OF RESULTS OF TESTS BY SEVERAL LABORATORIES ON NO. 6 COAL FROM OR NEAR ORIENT MINE

The value of empirical tests, such as the present, lies largely in the comparison of results obtained from a considerable number of coals subjected to the same set of conditions. That exactly similar results will be obtained in other laboratories is not probable, even though the specifications for making the tests are scrupulously followed, because of local and uncontrollable differences in procedure and equipment. However, where it is possible to compare the results of empirical tests by two or more laboratories such comparison is of interest since it furnishes a means of bridging the gap between different series of tests by different organizations.

The best available values for comparison are those obtained on coal from the No. 6 bed from the Orient No. 1 mine in Franklin County or from a mine very close to it which because the geological conditions in the region are very uniform should be practically the same. Determinations of coke, gas, and by-product yields using the United States Steel Corporation dry distillation test were made by the Illinois State Geological Survey on coal from the Orient mine and from an adjacent mine. A modification of the dry distillation test was used as one of the small-scale tests by the United States Bureau of Mines in its cooperative study with the American Gas Association of the gas, coke, and by-product making properties of American coals,[2] coal from Orient No. 1 mine[3] being one of those tested. The coal was also carbonized in the larger sized retort

---

[2] Fieldner, A. C., Davis, J. D., Thiessen, R., Kester. E. B., and Selvig, W. A., Methods and apparatus used in determining the gas, coke and by-product making properties of American coals: U. S. Bureau of Mines Bull. 344, 1931.

[3] Fieldner, A. C. and others, Carbonizing properties and constitution of No. 6 bed coal from West Frankfort, Franklin County, Ill.: U. S. Bureau of Mines Tech. Paper 524, 1932.

used in that cooperative survey. Coal from this mine had previously been used in the large-scale tests at St. Paul on the practicability of producing coke from

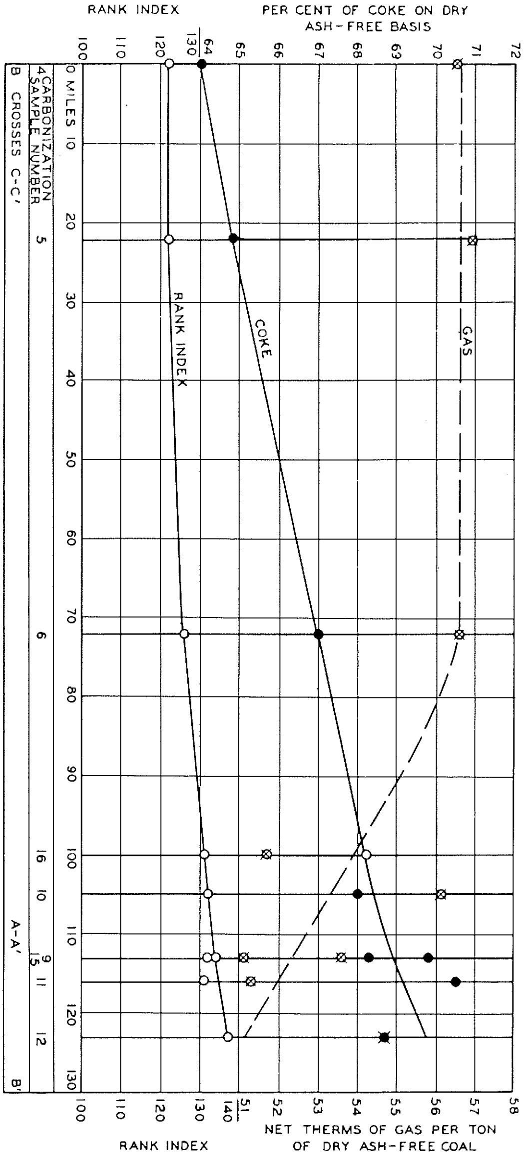


Fig. 39.—Gas, Coke, and Rank Indices along the Line B-B′ (Fig. 37).

Illinois coals in Koppers type ovens.[4] The quantitative yields of coke, gas, tar, and other products obtained in the various tests have been compiled for comparison in Table 14.

---

[4] McBride, R. S., and Selvig, W. A., Coking of Illinois coals in Koppers type oven: U. S. Bureau of Standards Tech. Paper 137, 1919.

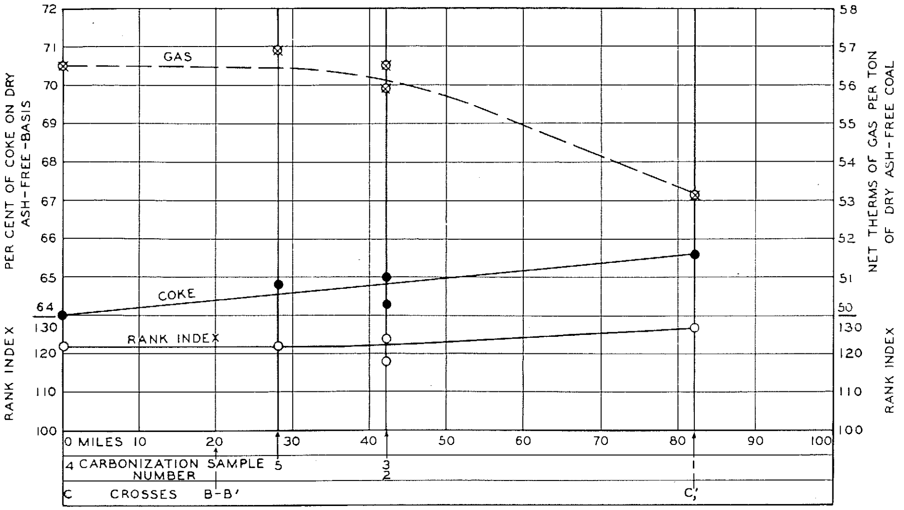


Fig. 40.—Gas, Coke, and Rank Indices along the Line C-C′ (Fig. 37).

The agreement between the results given by the United States Steel Corporation carbonization test in the two laboratories is very good, especially when consideration is given to the fact that the samples were taken years apart and probably from places quite far apart in the mine. They point to the uniformity of Illinois coals.

COMPARISON OF RESULTS ON COALS FROM OTHER STATES

No tests were made on coals from other states during the course of the present investigation. More than 30 coals have been tested by this method by the Bureau of Mines in its cooperative study with the American Gas Association. In view of the good agreement between the tests at the Illinois State Geological Survey and at the United States Bureau of Mines on the Orient coal there is good reason to assume that the tests reported by the Bureau of Mines[5] may be directly compared with results on Illinois coals reported here. In making such comparisons it is well to keep in mind the difference in moisture and ash content of the various coals. Attention has already been called (page 57) to the conclusions arrived at by the Bureau of Mines from a comparison of the Bureau of Mines, American Gas Association carbonization test results for the Illinois coals with the results for eastern coals. The Bureau of Mines finds a close correlation between the results of the Bureau of Mines, American Gas Association test at 900°C. and the results of the United States Steel Corporation carbonization test.

---

[5] Fieldner, A. C., and Davis, J. D., Gas-, coke-, and by-product making properties of American coals and their determination: U. S. Bureau of Mines Mon. 5, p. 119, 1934.

TABLE 13.—RESULTS OF CARBONIZATION ASSAY TESTS ON ILLINOIS COALS

	Coal Analysis										
	Coal sample number	Bed No.	County	Moisture per cent	Moisture-free basis						Rank index
					Ash per cent	Sulfur per cent	Fixed carbon percent	Volatile matter per cent	Calorific value B.t.u. per pound		
1	C–5	6	St. Clair..........	9.3	16.5	4.9	45.6	37.9	11,680		127
2	C–1934	6	Macoupin........	14.1	11.3	4.8	50.1	38.6	12,180		118
3	C–7	6	Macoupin........	12.3	12.5	4.8	46.3	41.2	12,350		124
4	C–1935	6	Christian.........	13.2	12.8	5.5	48.6	38.6	12,230		122
5	C–9	6	Montgomery......	13.0	12.2	4.3	47.4	40.4	12,300		122
6	C–1936	6	Washington.......	10.8	14.8	4.7	48.6	36.6	12,000		126
7	C–383	6	Randolph.........	9.6	16.2	3.9	45.9	37.9	11,750		128
8	C–197	6	Perry............	6.2	14.8	1.1	49.7	35.5	11,930		132
9	C–11	6	Franklin..........	7.3	10.7	1.3	53.2	36.1	12,850		134
10	C–1937	6	Franklin..........	8.1	9.7	1.2	55.8	34.5	12,960		132
11	C–198	6	Franklin..........	8.9	10.2	1.2	54.3	35.5	12,920		131
12	C–12	6	Williamson.......	6.0	11.1	2.9	52.5	36.4	12,860		137
13	C–1938	5	Saline............	6.3	7.6	2.7	56.8	35.6	13,630		139
14	C–199	5	Saline............	5.7	7.1	2.0	54.1	38.8	13,370		136
15	C–1939	6	Franklin..........	8.8	8.3	1.1	58.4	33.3	13,290		132
16	C–493	6	Franklin..........	9.2	9.8	2.4	53.9	36.3	12,940		131
16[a]	C–497	6	Franklin..........	9.1	9.7	.8	54.8	35.5	12,920		130
16[b]	C–509	6	Franklin..........	9.6	1.0	.8	61.6	37.5	12,124		130
17	C–1940	6	Vermilion.........	14.7	10.6	1.6	53.4	36.0	12,940		122

[a] Below blue band.
[b] Bright coal (anthraxylon)

TABLE 13.—RESULTS OF CARBONIZATION ASSAY TESTS ON ILLINOIS COALS (continued)

	Carbonization test yields, per cent by weight										
	Coke	Tar	Light oil	Fixed NH_3	Free NH_3	Total NH_3	CO_2	H_2S	Liquor	Gas	Gas dry cu. ft. per ton, 30″ Hg. 60°F
1	70.8	2.3	1.20	.01	.33	.34	3.87	1.74	7.5	12.3	9,140
2	69.0	2.3	1.47	.03	.34	.37	3.70	1.71	8.3	13.2	10,530
3	68.9	2.1	1.38	.04	.34	.38	3.96	1.66	8.0	13.6	10,380
4	69.2	2.3	1.59	.10	.27	.37	3.65	1.85	7.8	13.2	10,370
5	70.1	1.4	1.69	.08	.27	.35	4.10	1.29	7.7	13.4	10,830
6	71.8	2.1	1.25	.10	.28	.38	3.13	1.19	7.9	12.3	9,580
7	71.6	2.5	1.39	.01	.36	.37	3.37	1.43	7.1	12.2	9,120
8	73.7	1.2	1.39	.02	.41	.43	3.14	.29	7.0	12.9	9,440
9	70.6	4.8	1.10	.03	.42	.45	2.46	.33	7.7	12.6	9,120
10	71.4	3.5	1.12	.06	.37	.43	2.47	.33	7.9	12.9	9,680
11	73.5	2.4	.94	.04	.43	.47	3.04	.34	7.5	11.8	9,100
12	71.6	3.4	1.23	.02	.43	.45	2.63	.91	7.6	12.2	9,410
13	71.7	3.9	1.23	.06	.36	.42	1.94	.93	6.2	13.7	9,680
14	70.6	5.2	1.65	.03	.34	.37	1.47	.74	6.3	13.7	9,620
15	72.4	3.0	1.32	.05	.29	.34	2.42	.29	7.1	13.1	9,820
16	72.4	3.5	1.26	.04	.37	.41	2.73	.41	7.1	12.2	9,820
16[a]	71.8	3.4	1.30	.03	.50	.53	3.19	.16	6.3	13.3	10,580
16[b]	70.6	4.1	1.46	.08	.37	.45	2.13	.31	7.4	13.6	10,820
17	71.0	2.9	1.21	.03	.36	.39	3.66	.29	7.8	12.8	9,570

TABLE 13.—RESULTS OF CARBONIZATION ASSAY TESTS ON ILLINOIS COALS (continued)

	Gas analysis per cent by volume				Heating value				
	CH_4	CO	Illuminants	H_2	B.t.u. per cu. ft., gross	B.t.u. per cu. ft., net	Therms, gross	Therms, net	Sp. Gr. gas, dry at 30″60°F from constituents
1	25.2	12.4	3.1	59.2	545	486	49.8	44.4	.336
2	24.3	12.9	2.7	60.2	533	476	56.1	50.1	.332
3	23.6	13.4	2.7	60.1	527	471	54.7	48.9	.333
4	23.6	12.8	3.0	60.5	532	475	55.2	49.3	.331
5	23.2	13.7	2.2	60.7	517	461	56.0	49.9	.329
6	26.1	12.1	3.9	57.4	562	503	53.8	48.2	.347
7	26.5	12.4	3.8	57.4	565	505	51.5	46.1	.350
8	26.0	12.5	4.1	57.3	566	506	53.4	47.8	.352
9	29.1	10.6	4.1	56.1	587	525	53.5	47.9	.350
10	28.8	11.4	4.1	55.7	585	524	56.6	50.7	.356
11	27.5	12.4	3.5	56.6	567	507	51.6	46.1	.352
12	28.8	10.9	3.6	56.9	578	517	54.4	48.6	.346
13	30.0	9.7	4.4	56.1	599	535	58.0	51.8	.350
14	32.1	9.2	5.1	53.5	623	558	59.9	53.7	.363
15	23.9	11.8	3.1	61.2	536	478	52.6	46.9	.325
16	22.2	10.6	3.7	63.4	534	475	52.4	46.6	.312
16[a]	23.3	11.1	2.5	62.9	522	465	55.2	49.2	.309
16[b]	21.9	11.1	3.3	63.6	525	468	56.8	50.6	.311
17	26.7	11.4	3.3	58.7	559	499	53.5	47.8	.337

Table 13.—Results of Carbonization Assay Tests on Illinois Coals (concluded)

Sample No.	Coke analysis		By-product yield per ton			Coke yield, ash-free, per cent of ash-free coal	Gas, net therms per ton ash-free coal
	Sulfur per cent	Ash per cent	Light oil, gallons	Tar, gallons	Ammonium sulfate pounds		
1	4.49	22.6	3.3	4.6	26.4	65.6	53.2
2	3.91	16.5	4.0	4.6	28.7	65.0	56.5
3	3.97	18.4	3.8	4.2	29.5	64.3	55.9
4	4.83	19.4	4.3	4.6	28.7	64.0	56.5
5	3.87	18.8	4.6	2.8	27.2	64.8	56.9
6	4.05	70.5	3.4	4.2	29.5	67.0	56.6
7	3.67	23.5	3.8	5.0	28.7	65.4	55.0
8	1.47	20.7	3.8	2.5	33.4	68.6	56.1
9	1.20	13.6	3.0	9.6	34.9	68.3	53.6
10	1.16	14.0	3.0	7.0	33.4	68.0	56.1
11	1.32	13.9	2.6	4.8	36.5	70.5	51.3
12	2.45	14.7	3.3	6.8	34.9	68.7	54.7
13	2.22	10.7	3.3	7.8	32.6	69.3	56.1
14	1.79	10.4	4.5	10.4	28.7	68.1	57.8
15	1.02	11.6	3.6	6.0	26.4	69.8	51.1
16	1.64	15.0	6.1	7.0	31.8	68.2	51.7
16[a]	.91	11.6	3.5	6.8	41.1	70.3	54.5
16[b]	.66	3.3	3.9	8.2	34.1	68.9	51.1
17	1.79	15.9	3.3	5.8	30.3	66.8	53.5

TABLE 14.—COMPARISON OF GAS, COKE, AND BY-PRODUCT YIELDS OBTAINED IN VARIOUS TESTS ON ORIENT COAL, NO. 6 BED, WEST FRANKFORT, FRANKLIN CO., ILLINOIS

(All results on dry ash-free basis)

	Yields, per cent				Gas cu. ft. per ton	Tar gallons per ton	Ammonium sulfate lbs. per ton	Gross therms per ton
	Coke	Gas	Tar	Liquor				
U. S. Steel Corp. assay test by Bureau of Mines...	69.3	17.5	3.1	7.6	12,155	6.4	38.1	61.4
U. S. Steel Corp. assay test of Illinois Geol. Survey.	69.9	17.2	3.3	7.7	11,229	6.5	28.8	59.9
Same test by Illinois Geol. Survey on coal from adjoining mine..............................	67.1	17.2	5.4	8.6	10,877	10.8	39.1	53.6
Tests in Bureau of Mines retort at 900° C........	64.8	20.8	5.4	7.9	12,356	11.1	24.8	66.5
Tests in Bureau of Mines retort at 1000° C.......	64.4	22.9	4.3	4.9	14,781	8.8	16.2	64.0
Test in Koppers type ovens at St. Paul..........	65.0	23.0	4.7	...	13,178	9.4	31.9	69.5

CHAPTER XIII—BEHAVIOR OF ILLINOIS COALS UNDER HEAT AS SHOWN BY THE RESULTS OF EXPERIMENTAL WORK

SOFTENING TEMPERATURES AND TEMPERATURE RANGE OF PLASTICITY

To illustrate the effects which the Agde-Damm test measures, data from a typical test on a feebly swelling coal have been plotted on figure 41 and on figure 42 from the same coal after it had been oxidized until it was a non-swelling coal. In these graphs dial readings in thousandths of an inch starting from an arbitrary dial setting are plotted against temperatures which have been corrected for differences due to lag. Temperatures measured in the copper block will be higher, and temperatures measured in the plunger lower than those actually present in the center of the coal. Additional corrections must also be made for changes in dial reading due to expansion of the apparatus if quantitative measurements of the amount of shrinkage or swelling are desired. The temperature of initial contraction or initial plasticity is determined by the intersection of an extension of the horizontal portion of the curve with the extension of the uniform portion of the contraction period. (Point A, figs. 41 and 42). The temperature of initial swelling which is also the temperature of active decomposition is determined by the point at which contraction stops and swelling starts. The temperature intervals between the point of initial contraction and active decomposition has been called the initial contraction interval. The temperature at which expansion ceases marks the end of the plastic interval.

It is very difficult or even impossible to determine the temperature of active decomposition by this method when the coal particles do not coalesce and the coal does not swell. In the case of Illinois coals such a condition is likely to exist, because of the ease with which these coals oxidize, unless the tests are made very soon after the coal has been taken from the mine and immediately after grinding. Grinding should preferably be done in an inert atmosphere.

Curves showing the loss of volatile matter with increasing temperature for the fresh and weathered No. 2 coal were also plotted on figures 41 and 42. The position of the point of most active loss of volatile matter is seen to correspond very closely to the temperature of initial expansion. In the case of the weathered coal, contraction is still taking place at the time of the maximum rate of volatile matter evolution.

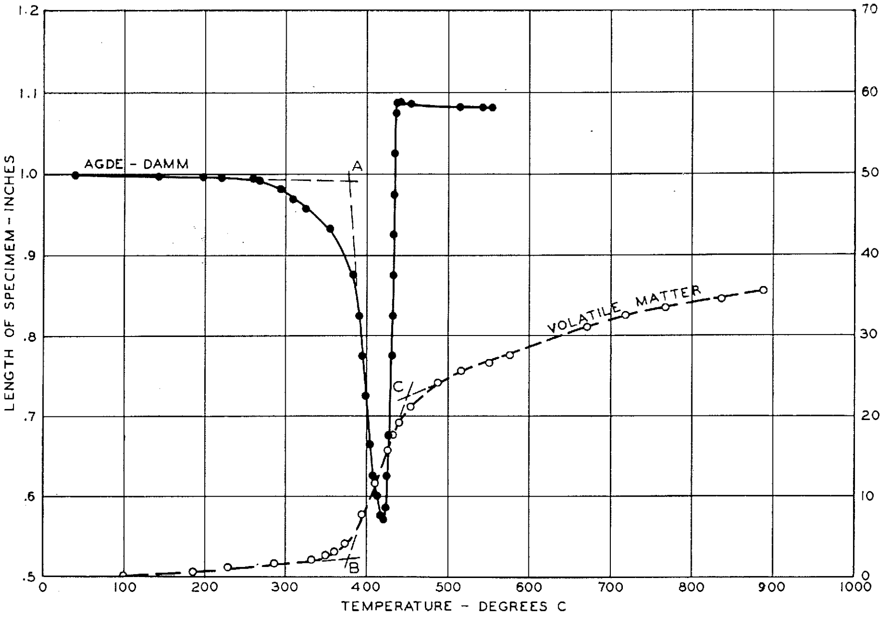


Fig. 41.—Adge-Damm and Volatile Matter Evolution Curves for Fresh No. 2 Coal.

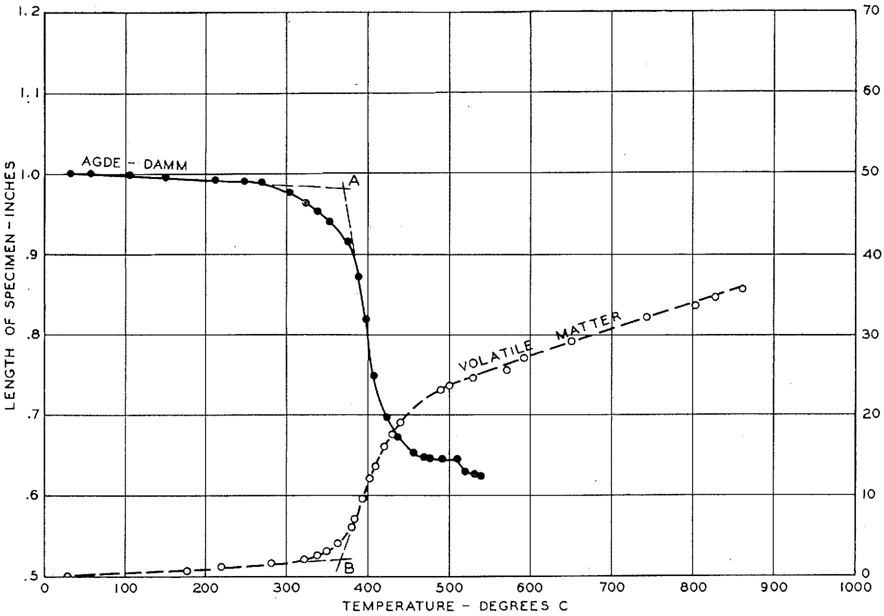


Fig. 42.—Agde-Damm and Volatile Matter Evolution Curves for Weathered No. 2 Coal.

SIGNIFICANCE OF THE PLASTIC RANGE TEMPERATURE

Coke quality apparently cannot be definitely correlated with the plastic range values obtained by the Agde-Damm method. Certain rather broad trends do occur. Brewer and Atkinson[1] say, "Data from the modified Agde-Damm test method provide valuable knowledge on the pre-plastic temperature range, but have limited significance, except for the less fusible coals, in the plastic range." They find that the temperature of initial expansion corresponds to the temperature at which the mass of the coal becomes plastic and begins to offer resistance in the Davis plastometer. Brewer and Atkinson say further, "The strength of the coke has been shown to increase with decreases in the Agde-Damm 'contraction-interval' in the length of the 'plastic range' and the length of the 'high fluidity' as observed with the Davis plastometer."

SOFTENING TEMPERATURE AND PLASTIC TEMPERATURE INTERVALS OBTAINED FROM ILLINOIS COALS

The temperatures at which various Illinois coals show initial contraction, initial expansion, final expansion (or contraction if non-fusing) and the initial contraction and plastic range intervals are given in Table 15, which also gives rank indices, dry mineral-matter-free carbon, and agglutinating values. For comparison, values for other coals, taken from reports of the United States Bureau of Mines have been added. The initial contraction temperature for the No. 6 coal from Franklin County (No. 10) found by the Bureau of Mines is much lower than were found in this investigation. The initial expansion temperature checks our value closely. The values for the Pocahontas coals found by the two laboratories are in close agreement.

AGGLUTINATING OR CAKING STRENGTH VALUES FOUND FOR ILLINOIS COALS

Because of the factors of oxidation of the coal after removal from the mine and during preparation and the influence of sand character, the values obtained for the agglutinating power of Illinois coals have little fundamental value except in some cases as an indication of order of magnitude. Table 15 presents the agglutination values obtained for a number of Illinois coals tested by the Bureau of Mines method as soon as possible after removal from the mine. These values were shown by interlaboratory check to be approximately one-half the values obtained by the Bureau of Mines. This difference in magnitude was shown by tests to be due to differences in the surface characteristics of the sands used. The intervals between the time the samples were taken at the mine and the time the tests were run in the laboratory were not the same for all of the coals for which values are given in Table 15 and in that respect the values are not strictly comparable.

---

[1] Brewer, R. E., and Atkinson, R. G., Plasticity of coals: Ind. Eng. Chem. Anal. Ed. vol. 8, No. 6, pp. 443-9, Nov. 1936.

TABLE 15.—PLASTIC RANGE TEMPERATURES AND AGGLUTINATING VALUES FOR ILLINOIS AND OTHER COALS

Sample No.[a]	State	County	Coal bed	Rank index[c]	Dry mineral-matter-free fixed carbon per cent	Softening temperature °C	Decomposition temperature °C	Initial contraction interval °C	Setting temperature °C	Plastic interval °C	Agglutinating value, Kg. B. of M. method
					Values Obtained in the Illinois State Geological Survey Laboratories						
C 153–4. .	Illinois.	Bureau.	7	115	54.0	354	. . .	. .	. . .	. .	4.4
C 493	Illinois.	Franklin.	6	131	60.7	. . .	n.d[d]	. .	. . .	. .	3.6
C 613	Illinois.	Franklin.	6	131	59.9	376	. . .	. .	438	62	3.2
C 146–7	Illinois.	Grundy.	7–Streator	123	52.3	336	401	65	431	95	5.6
C 138–9	Illinois.	Henry.	6	113	56.7	359	406	47	448	89	1.0
C 135–6	Illinois.	Knox.	6	117	55.3	356	415	59	440	84	2.1
C 149–51	Illinois.	Menard.	5–Springfield	122	54.9	354	418	64	441	87	5.1
C 648	Illinois.	Randolph. . . .	6	125	55.6	352	. . .	. .	432	80	4.3
C 748	Illinois.	Saline.	5	137	61.2	378	441	63	464	86	3.7
C 140	Illinois.	Stark.	6	117	54.6	349	407	58	441	92	4.8
C 141	Illinois.	Stark.	6	116	55.0						2.8
C 144	Illinois.	Stark.	6	117	55.4	352	411	59	443	91	5.0
C 803	Illinois.	Vermilion. . . .	7	125	52.3	. . .	n. d.	. .	. . .	. .	5.7
C 368	Illinois.	Washington. .	6	127	52.9	. . .	n. d.	. .	. . .	. .	2.9
C 612	Illinois.	Woodford. . . .	2	126	56.7	374	420	46	440	66	4.0
C 303	Kentucky.	Perry.	3–Hazard	146	56.3	. . .	n. d.	. .	. . .	. .	4.5
C 611	West Virginia		3–Pocahontas	153	82.7	406	452	46	481	75	9.9
Coal No. [b]					**Values Obtained by the U. S. Bureau of Mines**						Marshall Bird method
1	Pennsylvania. . .	Allegheny. . . .	Pittsburgh.	151	63.0	320	390	70	. . .	. .	6.7
2	Kentucky.	Letcher.	Elkhorn.	148	62.0	340	394	54	. . .	. .	4.3
3	Virginia.	Wise.	Taggart.	152	62.5	314	387	73	. . .	. .	5.7
7	Alabama.	Jefferson. . . .	Mary Lee.	152	67.6	322	386	64	. . .	. .	11.3
10	Illinois.	Franklin. . . .	No. 6.	133	60.8	275	414	139	. . .	. .	4.3
14	West Virginia. .	Logan.	Chilton.	149	61.6	304	388	84	. . .	. .	8.4
19	Utah.	Carbon.	L. Sunnyside.	140	57.0	295	394	99	433	138	3.9
20	Pennsylvania. . .	Allegheny. . .	Thick Freeport. . .	149	60.9	293	380	87	. . .	. .	10.2
23	West Virginia. .	McDowell. . .	Pocahontas 4. . . .	157	84.2	407	462	55	490	83	16.3

---

[a] Illinois State Geological Survey Coal Analysis number.

[b] Refers to coal numbers in Table 1, U. S. Bureau of Mines Monograph 5, 1934.

[c] "Rank index" moist mineral-matter-free calorific value in hundreds of B.t.u. per pound is used for classifying by rank only those coals having a rank index of less than 140. For coals having a rank index of 140 or above, the dry mineral-matter-free fixed carbon value is used.

[d] n. d.=not determined.

CHARACTERISTICS OF THE COAL WHICH AFFECT ITS AGGLUTINATING VALUE

Mineral impurities.—The removal of mineral matter from coals high in ash should improve their agglutinating powers. A Washington County coal having an ash content of 13.7 per cent had an agglutinating value of 2.9 kg. After cleaning at a specific gravity of 1.35 by the float-and-sink procedure the coal had an ash content of 6.4 per cent and an agglutinating value of 3.2 kg. The 28.8 per cent ash refuse had an agglutinating value of less than 0.2 kg. With an increase in agglutinating value proportional to the ash reduction a value of 3.1 kg. would have been obtained. This is within experimental error of the value 3.2 kg. found experimentally.

Freshness.—The agglutinating values of Illinois coals are variously decreased by oxidation. Exposure of the mine face for some weeks permits oxidation sufficient to affect the agglutinating values of channel samples. For example, a sample of Franklin County coal taken from a fresh face had an agglutinating value of 3.64 kg., while a sample from a face that had been exposed for several weeks had a value of 2.75 kg.

Fusain content.—In attempts to reduce the formation of shrinkage cracks and to produce blockier coke, dedusting plant dust or coke breeze was added as low volatile carbonaceous matter. The dedusting plant dust contained about 50 per cent fusain and had no agglutinating value. The addition of dedusting plant dust or fusain to coke oven charges to improve coke quality has been patented by Mott and Wheeler[2] in England on the claims that fusain is "wet" by and becomes better incorporated into the coal than coke. Some agglutinating values of blends of coal coked in experiments reported elsewhere in the report are of interest in that connection and are presented in Table 16.

TABLE 16.—AGGLUTINATING VALUES OF VARIOUS COALS AND BLENDS OF COAL AND COKE OR OF COAL AND DEDUSTING PLANT DUST

Composition of sample	Agglutinating value, kg.
100% Franklin Co. coal	3.2
80% Franklin Co. coal 20% dust	4.0
80% Franklin Co. coal 20% coke	0.4
75% Franklin Co. coal 20% coke, 5% dust	0.4
100% Randolph Co. coal	4.3
80% Randolph Co. coal 20% coke	0.0
100% Woodford Co. coal	3.3
80% Woodford Co. coal 20% dust	3.8

---

[2] Mott, R. A., and Wheeler, R. V., and The National Federation of Iron and Steel Manufacturers (Corporate Organization) Limited. "Improvements In and Relating to the Manufacture of Coke." British Patent 351,546, accepted June 29, 1931.

Conclusions.—Illinois coals have relatively little caking or agglutinating power. The agglutinating power they have is easily destroyed by oxidation. Oxidation also destroys the ability of the plastic coal particles to coalesce to form the viscous mass which ultimately would form the coherent cellular coke. For these reasons Illinois coals should be coked as soon as possible after they are mined, and should be taken from freshly worked places in the mine. Inert material such as mineral matter adversely affects the agglutinating power of these coals and should be present, therefore, in amounts as small as possible.

CHAPTER XIV—EFFECT OF THE BANDED COMPONENTS AND IMPURITIES OF COALS ON COKE STRUCTURE

Though the usual analyses and tests treat coal as a homogeneous substance, it is far from being so. Even casual examination of a piece of coal with the naked eye reveals the fact that the common varieties of coal are made up of bands of materials, some having a vitreous, black appearance, others a finely striated, silky appearance. Fracture surfaces parallel to the bedding plane frequently are covered with a charcoal-like, friable, cellular material often having the appearance of small chips of carbonized wood. These bands are not merely the same substance in different forms but are actually different materials which had their origin from different parts or different mixtures of parts of plants. The impurities, likewise, are not uniformly distributed throughout the mass of coal. During the mining and preparation of the coal the banded components and impurities, because of their different properties, may be present in the various sizes of prepared coal in different proportions than they existed in the coal as it occurred in the bed. Since the various components and ingredients behave differently during carbonization and since the situation is more or less common to all coals, they may be treated in a general way.

BANDED COMPONENTS

In Illinois coals the banded components are mainly of three kinds: (1) a bright, vitreous material known to be derived from larger pieces of woody material, variously called "anthraxylon"[1] or "vitrain"[2]; (2) non-vitreous appearing, striated materials, sometimes having a silky lustre, which forms the bulk of the coal and which separates the bright bands from one another, derived from plant debris of all kinds, but mainly of small size and called "attritus" or "clarain"; and (3) a charcoal-like material practically always present and in minor amount, derived in some unknown manner from woody material and called "fusain". The banded structure of Illinois coals is largely, if not entirely, due to the presence of the bright bands which range in thickness from fine slivers to infrequently as much as one inch. A less common variety of banded coal is composed of finely banded material having a dull gray or slaty appearance.

---

[1] Thiessen, R., and Francis, W. R., Terminology in coal research: U. S. Bureau of Mines Tech. Paper 446, 1929.

[2] Stopes, M. C., The studies on the composition of coal: The Four Visible Ingredients in Banded Bituminous Coal, Proc. Royal Soc. Lon. 90B, p. 470, 1919.

These coals are characterized by toughness and a blocky fracture and are known as "splint coals", dull coal "durain", or in England also as "hards". Cannel coal, also a less frequent variety of coal, is not banded and exhibits a waxy lustre and a conchoidal fracture. The varying nature of these components are reflected in their carbonizing properties both with respect to their behavior when heated and the yields of products. Conclusions from a correlation of the petrographic compositions of coals and the results of certain carbonizing tests have been presented by R. Thiessen and Sprunk[3] in which one of the coals considered is an Illinois coal from the No. 6 bed in Franklin County.

Carbonizing properties.—Of the four components, fusain alone does not form a coke. Splint coal will form coke but the individual pieces of coal tend to form individual pieces of coke. Bright coal is the only satisfactory coke-forming coal. Tests show that of the two ingredients of bright coal, the anthraxylon (or vitrain) by itself tends to form a frothy, thin-walled coke. Its separation in a high state of purity does not appear to be very desirable for coke making purposes. The carbonizing properties of the bright attritus or clarain depend on its composition in terms of plant fragments, such as resins, waxy, or humic materials. In using fine sized coal for coke making, the questions of ash and fusain content are far more important than are the proportions of anthraxylon and attritus (vitrain and clarain). Fusain tends to make the coke more blocky in structure, but should not be present in sufficient quantity to make the coke soft and friable.

Composition of Illinois coals in terms of banded components.—Illinois coals are predominantly bright coals. Occurrences of splint and cannel coals are localized and may be neglected in a consideration of Illinois coals for coke making purposes.

The average petrographic composition of the No. 6 bed in Illinois as determined from a megascopic examination of 21 columns, each of which represented the entire seam and which averaged 6 feet 8 inches in thickness, was found by McCabe to be 75.9 per cent clarain (bright attritus), 13.8 per cent vitrain (anthraxylon), 5.6 per cent fusain (mineral charcoal) and 4.7 per cent non-coal impurities.[4] The petrographic composition of No. 6 coal from Orient No. 6 mine, West Frankfort, Franklin County, is discussed in detail in the report of the United States Bureau of Mines, American Gas Association cooperative investigation of that coal.[5] This coal is quite uniform from top to bottom.

---

[3] Sprunk, G. C., and Thiessen, R., Relation of microscopic composition of coal to chemical, coking and by-product properties: Ind. Eng. Chem. vol. 27, No. 4, pp. 446-451, April 1935.

[4] McCabe, L. C., The lithology of coal No. 6: Trans. Illinois State Acad. Sci. vol. 25, No. 4, pp. 149-50, 1934.

[5] Fieldner, A. C., and others, Carbonizing properties and constitution of No. 6 bed coal from West Frankfort, Franklin County, Ill.: U. S. Bureau of Mines Tech. Paper 524, 1932.

Compared to other coals from the United States studied in that investigation it contained much anthraxylon (vitrain). McCabe, Mitchell, and Cady[6] have reported the petrographic composition of other Illinois coals.[7]

Because coal components differ considerably in their resistance to crushing and breakage, the various components tend to concentrate in different sized fractions of the coal. Fusain, being the most friable, is found concentrated in the very fine dust, the minus 200-mesh material containing 50 per cent or more of it. Vitrain is the next most friable material and is also found concentrated in the fine sizes.[8] Because vitrain (anthraxylon) tends to have a lower specific gravity because of its freedom from ash forming material, it is also found concentrated in the light float-and-sink fractions. The concentration of fusain in the very fine sizes of coal recovered from dedusting plants has been studied by G. Thiessen[9] who found that the minus 200-mesh fraction was composed largely of fusain.

IMPURITIES

The impurities which seriously affect the value of a coal for the manufacture of coke are ash, sulfur, moisture, and in some cases phosphorus. Moisture is undesirable because of the heat required to evaporate and drive it out of the coal and because the resulting liquor must be condensed and handled in the by-product recovery system. The ash is objectionable because it becomes concentrated in the coke, decreasing its fuel value, and because it may impair coke structure. Sulfur is objectionable not only because part of the sulfur remains in the coke and affects the value of the coke for practically all of its uses but also because part of the sulfur is volatilized and is found partly in the tar, where it is relatively innocuous, and partly in the gas where its presence is serious and from which it must be removed if the gas is to be sold. Phosphorus is important only in certain metallurgical applications of coke. The effects of the impurities on the coking process and on the character of the resulting coke and by-products, and the methods which may be employed to eliminate those impurities or to minimize their harmful effects are discussed.

MOISTURE

Moisture is an undesirable component of coking coal because of the large amounts of heat required to vaporize it. The high latent heat of vaporization of water keeps the temperature beyond the plastic zone in a coking charge of coal down to the boiling point of water. Much of the heat used to vaporize this

---

[6] McCabe, L. C., Concentration of the banded ingredients of Illinois coals by screen sizing and washing: Amer. Inst. Min. Met. Eng. Tech. Pub. 684, Class F, Coal Division No. 73, Table 2, 1936.

[7] McCabe, L. C., Mitchell, D. R., and Cady, G. H., Contributions to the Study of Coal—Banded ingredients of No. 6 coal and their heating values as related to washability characteristics: Illinois State Geol. Survey Rept. Inv. 34, 1934.

[8] McCabe, L. C., Op. cit.

[9] Thiessen, G., Fusain content of coal dust from an Illinois dedusting plant: Amer. Inst. Min. Met. Eng. Tech. Pub. 664, Class F Coal Div. No. 70, 1936.

moisture comes from hot products of carbonization. The presence of moisture does, however, require the addition of an increased quantity of heat to the charge, and this heat, coming through the oven walls is expensive and decreases the oven capacity by increasing coking time. Koppers[10] states that each two per cent moisture corresponds to an increase of one hour in coking time. A large excess of water may cool the oven refractories sufficiently to damage them. The evaporated moisture must later be condensed and provided for in by-product recovery system capacity and finally disposed of without creating a nuisance.

The moisture contents of Illinois coals (Table 11, pp. 62-3; fig. 17, p. 69) are relatively high when compared with the eastern coals commonly used for coke making. Wet coal-cleaning processes add more water to the fine sized coal and introduce further difficulties. Drying processes commonly employed which use heated gases may seriously decrease the coke forming properties of these coals through oxidation. The natural tendency of these coals to oxidize is accelerated by the increased temperature, the small size of the coal particles, and the rapid circulation of air through the mass of coal.

ASH OR MINERAL MATTER

Ash is objectionable in metallurgical coke because its presence reduces the furnace capacity and increases the quantity of slag. For domestic or industrial fuel, ash is objectionable in coke for the same reason that it is objectionable in coal, with the added fact that coke ashes have a tendency to be more dusty when not clinkered. Furthermore the large clinkers which may form due to the higher temperatures attained in a bed of burning coke are undesirable. The particle size of coal as charged into a coke oven is usually rather small, less than ½ inch in most cases, the small size being obtained either by crushing larger coal or through the use of small screenings. The small size of the coal particles limits the size of the pieces of mineral matter or ash forming streaks in the coke and makes the ash distribution more homogeneous. The swelling and flowage of the coal during its plastic stage further disrupts the ash forming materials. The result is that the ash residue is powdery and frequently fluffy when coke is burned at a temperature below the fusion point of the ash.

Although the more uniform distribution of the ash forming material in coke as compared with coal ordinarily results in less frequent formation of clinkers,[11] such clinkers as do form tend to be large, vitreous, and relatively strong. Cokes made from coals having ashes with low fusion temperatures are most likely to product such clinkers, since the ash forming components are quite uniformly distributed and the attainable temperatures in the fuel bed are high.

---

[10] Koppers Mitteilungen 1921, No. 2, p. 58—quoted in Gluud, W., (Jacobson, D. L.): International Handbook of the By-product Coke Industry. American Edition, New York, 1932, p. 96.

[11] Thiessen, G., Ball, C. G., and Grotts, P. E., Coal ash and coal mineral matter. Ind. Eng. Chem. vol. 28, No. 3, pp. 355-361, 1936.

TABLE 17.—SEGREGATION OF LARGER IMPURITIES IN SMALLER SIZES OF COKE PRODUCED BY SHATTER BREAKAGE

Laboratory Nos.	Description of sample	Ash content, per cent on dry basis		
		Coke +½ inch	Coke −½ inch	Difference
C-1557–C 1550	2 x ¾ inch washed at 1.55 sp. gr. and crushed to −⅛ inch	14.4	14.8	+0.4
C 1558–C 1551	¾ x 0 inch screenings raw	13.9	22.7	+8.8
C 1561–C 1554	¾ x 0 inch, washed at 1.5 sp.gr.	10.7	14.3	+3.6
C 1562–C 1555	5/16 x 0 inch screenings raw	16.5	24.8	+8.3
C 1559–C 1552	6/16 inch x 10 mesh washed at 1.5 sp.gr.	9.7	11.6	+1.9
C 1560–C 1553	−10 mesh screenings raw	17.7	19.5	+1.8

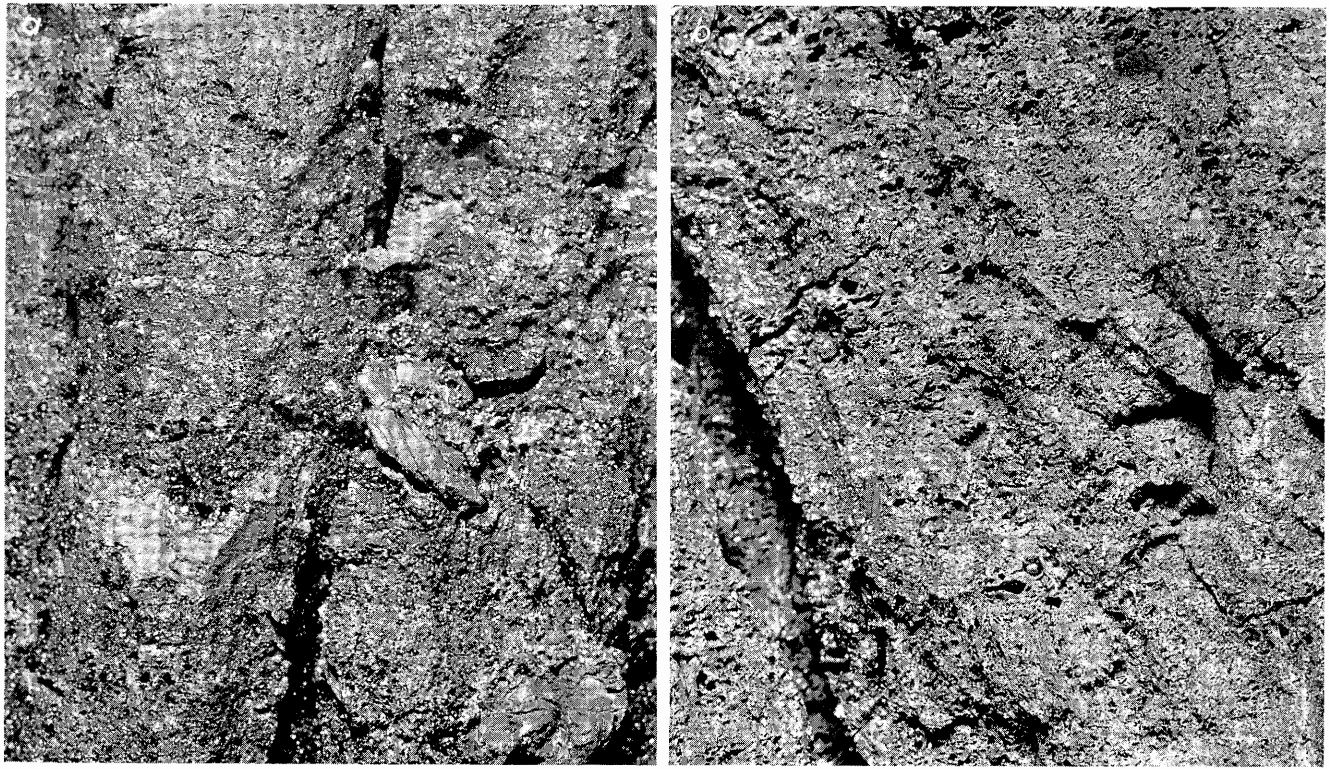


Fig. 43. *a.*—Coke made from Raw Screenings; Note Pieces of Loosely Held Shale
b.—Coke made from Washed Screenings; Note Freedom from Shale

The mineral matter in coal is further troublesome because of the effect it may have on coke structure. Free mineral matter is non-caking material which must be consolidated into the coke mass if the coke is to be strong. If the agglutinating strength of the coal is already low, sufficient quantities of such inert matter may weaken the coke. Even in small amounts the effects of such free mineral matter are apparent when a piece of coke is examined and compared with a piece of coke made from the same coal from which such inert matter has been removed. This is illustrated by figure 43 in which two pieces of coke are pictured side by side, one made from raw -¾ inch screenings, the other from screenings which had been cleaned at a specific gravity of 1.5. It will be noted that, in the coke from the raw screenings, the cracks in the coke either begin, or end, at a piece of loosely held shale. That these larger pieces of mineral matter are only loosely held in the coke is evident from a comparison of the ash content of the larger sized (+½ inch) and smaller sized (-½ inch) pieces resulting from breakage during the shatter test (Table 17).

Ash forming materials tend to segragate in the naturally produced finer sized screenings from Illinois coals.[12] This tendency is more pronounced in some coals than in others but must always be considered as a possible serious problem if small sized screenings are to be used as raw material for coke manufacture. As examples of how the ash content of the various sized fractions from coals may vary, the ash content of the face samples and of sized fractions from naturally formed screenings from three of the mines studied by McCabe and his co-workers are presented in Table 18.

TABLE 18.—VARIATIONS IN ASH CONTENT WITH SIZE IN NATURALLY PRODUCED SCREENINGS FROM ILLINOIS COAL

Size	Ash content, per cent on dry basis		
	Mine B[a]	Mine G[a]	Mine J[a]
Face sample	6.5	11.4	8.4
1¼ x 0-mesh	19.4	18.1	11.8
1¼ x ¾ inches	11.0	15.5	9.7
¾ x ⅜ inches	14.3	16.9	10.2
⅜ inch x 10-mesh	19.7	19.3	11.7
10 x 48-mesh	30.5	25.8	18.5
Minus 48-mesh	36.5	25.2	16.8

[a] Mine B. Northern Illinois No. 2 coal.
Mine G. St. Clair County No. 6 coal.
Mine J. Saline County, Harrisburg, No. 5 coal.

The available ash analyses of face samples of coal from Illinois mines, mine and county averages, and available ash softening temperature values have

---

[12] McCabe, L. C., Mitchell, D. R., and Cady, G. H., Proximate analyses and screen tests of coal mine screenings produced in Illinois: Illinois State Geol. Survey Rept. Inv. 38, 1935.

been presented by Cady.[13] County average ash values have previously been presented in this report in figure 18 and Table 11.

Since the quality of coke produced from Illinois coal is materially affected by the ash content of the coal used, it is very important that the ash content be reduced as much as possible. The cleaning of coals as a means of reducing the mineral matter even for coal used as a fuel in the raw state is becoming important in the Eastern Interior coal field and much attention is being given to the problems involved. Only the larger pieces of free mineral matter or the higher-ash-content pieces of coal can be removed by physical means. Finer crushing frequently permits a more complete removal of the ash forming material. The lower limit to which the ash content of a coal may be reduced is the so-called inherent ash content of the coal. This "inherent ash" is the ash which is finely disseminated through the coal substance or in chemical combination with it and which entered the coal during its formative peat stage. Studies on the washability of Illinois coals have been made by Mitchell,[14] by Callen and Mitchell,[15] and by McCabe, Mitchell, and Cady.[16] An early account of coal washing in Illinois has been given by Lincoln.[17] Statistics covering recent coal washing developments in Illinois as well as in the rest of the United States have recently been published by Plein of the United States Bureau of Mines.[18] Descriptions of coal cleaning plants in Illinois may be found in the technical literature.

Regarding the cleaning of coal in general, it may be said that each coal offers its own problems which must be solved by experimentation. Coal cleaning is a problem by itself, the consideration of which does not fall within the field of the present investigation.

SULFUR

Sulfur is an especially undesirable impurity to the coke and gas industries. It distributes itself during coal carbonization between the volatile materials, gas, light oils and tar, and the solid coke residue and is undesirable in all of them. Sulfur in blast furnace and foundry coke is easily picked up by iron. If the sulfur content of blast furnace coke is unusually high very close watch must be kept of the slag composition and the slag must be made more basic by the addition

---

[13] Cady, G. H., Illinois State Geol. Survey Bull. 62, 1935.

[14] Mitchell, D. R., The possible production of low ash and sulphur coal in Illinois as shown by float-and-sink tests: Univ. of Illinois Eng. Exp. Sta. Bull. 258, 1933.

[15] Callen, A. C., and Mitchell, D. R., Washability tests of Illinois coals: Univ. of Illinois Eng. Exp. Sta. Bull. 217, 1930.

[16] McCabe, L. C., Mitchell, D. R., and Cady, G. H., Banded ingredients of No. 6 coal and their heating values as related to washability characteristics: Illinois State Geol. Survey Rept. Inv. 34, 1934.

[17] Lincoln, F. C., Coal washing in Illinois: Univ. of Illinois Eng. Exp. Sta. Bull. 69, 1913.

[18] Plein, L. N., Statistical analysis of the progress in mechanical cleaning of bituminous coal from 1927 to 1934: U. S. Bureau of Mines Economic Paper 18, 1936.

of limestone, which reduces seriously the capacity of the furnace. If the coke is used for burning limestone, the sulfur is absorbed by the lime, contaminating it for chemical use. If the coke is burned for domestic or commercial heating the problem of sulfur is not so serious, but even here it is undesirable from the standpoint of atmospheric pollution. The ashes from a coke high in sulfur may also give off enough sulfurous gases after removal from the furnace ash pit to be a nuisance and even a danger to health.

If the gas is to be sold for domestic use and for many industrial uses the sulfur compounds in the gas must be thoroughly removed. Sulfur enters the gas mainly as hydrogen sulfide and in organic compounds such as carbon bisulfide and mercaptans. All of these have unpleasant odors, tarnish silver and many other metals, and yield sulfur dioxide when burned. Hydrogen sulfide is a deadly poison. The cost of purification is an important item in gas manufacture.

Occurrence in Illinois coals.—Sulfur occurs in coals as the iron disulfides, pyrite and marcasite, as part of the organic coal substance, and as the sulfate minerals which occur in appreciable quantities only in weathered coals. Pyrites may be found in all sizes from small, finely disseminated particles to large masses, lenses, or bands. Pyritic sulfur may be partly removed during coal preparation. Organic sulfur cannot be removed without destruction of the coal.

The county average sulfur contents of Illinois coals and the low-sulfur coal area have already been discussed. (Table 11, figs. 19 and 20, pp. 62, 71, 72). The available values for pyritic and organic sulfur contents of Illinois coals as determined on face samples have been presented by Cady[19] who has also discussed[20] in detail the distribution of sulfur in Illinois coals, and the low sulfur area in Southern Illinois.[21] The distribution of sulfur in Illinois coals between varieties has also been studied by Yancey and Fraser.[22] As stated before, Illinois coals in general, and except for small areas, have sulfur contents higher than is thought generally to be desirable in coals for the manufacture of metallurgical coke.

Removal from Illinois coals.—Coal cleaning or preparation procedures remove only the inorganic forms of sulfur, more particularly the pyritic sulfur occurring in larger aggregations. The problem of sulfur reduction has received considerable attention because of its economic importance. Callen and Mitchell,[23]

---

[19] Cady, G. H., Illinois State Geol. Survey Bull. 62, Table 6, 1935.

[20] Cady, G. H., Distribution of sulfur in Illinois coals and its geological implications: Illinois State Geol. Survey Rept. Inv. 35, p. 25, 1935.

[21] Cady, G. H., Low sulfur coal in Illinois: Illinois State Geol. Survey Bull. 38, pp. 432-434, 1922.

[22] Yancey, H. F., and Fraser, T., The distribution of the forms of sulphur in the coal bed: Univ. of Illinois Eng. Exp. Sta. Bull. 125, 1921.

[23] Callen, A. C., and Mitchell, D. R., Washability tests of Illinois coals: Univ. of Illinois Eng. Exp. Sta. Bull. 217, 1930.

Mitchell,[24] and McCabe, Mitchell, and Cady[25] have presented results of washability studies of Illinois coals which include work on the effect of gravity separation on the reduction of the sulfur content of the coal. These tests show that, with some exceptions, not much reduction in sulfur can be expected through washing Illinois coals. This is due, first to the relatively high organic sulfur content of these coals and, second to the fact that much of the pyritic sulfur is often found in a finely disseminated form[26] too small to be removed except after grinding the coal to extreme fineness. The possible reduction of the sulfur content of the coal produced at each mine or group of contiguous mines presents a new problem which must be solved experimentally.

Behavior of coal sulfur during coal carbonization.—The conclusions which could be drawn from a review of the pertinent literature[27] were that: (1) about half the sulfur in the coal appears in the coke but the percentage of sulfur in the coke will be about 80 per cent of that in the coal because of the loss of volatile matter during coke formation; (2) there is no unanimity of opinion as to the relative importance of organic and pyritic sulfur as a source of sulfur in the coke; and (3) the sulfur found in the coke appears to be directly proportional to the total sulfur content of the coal from which the coke was made.

The conclusions from this review of the literature indicated that it would be desirable to carry out investigations on the distribution of coal sulfur between the volatile matter and the residual coke or fixed carbon and to correlate this distribution with the distribution of sulfur in the coal between pyritic and organic forms in an attempt to find a way to estimate the probable sulfur content of a coke when the sulfur content and distribution of the sulfur between the two varities in the coal is known.

In the experimental work as carried out, the coke was prepared in platinum crucibles under conditions prescribed for the standard volatile matter test for

---

[24] Mitchell, D. R., The possible production of low ash and sulphur coal in Illinois as shown by float-and-sink tests: Univ. of Illinois Eng. Exp. Sta. Bull. 258, 1933.

[25] McCabe, L. C., Mitchell, D. R., and Cady, G. H., Banded ingredients of No. 6 coal and their heating values as related to washability characteristics: Illinois State Geol. Survey Rept. Inv. 34, 1934.

[26] Thiessen, R., Occurrence and origin of finely disseminated sulfur compounds in coal: Amer. Inst. Min. Eng. Bull. 153, pp. 2431-2444, 1919. Also discussed in Univ. of Illinois Eng. Exp. Sta. Bull. 125, pp. 54-63, figures 18-24, 1921.

[27] Thiessen, G., Behavior of sulfur during coal carbonization: Ind. Eng. Chem. vol. 27, No. 4, pp. 473-478, 1935.

Parr, S. W., The coals of Illinois: their composition and analysis: Univ. of Illinois Bull. vol. 1, No. 20, July 15, 1904; The University Studies Vol. 1, No. 7.

Powell, A. R., A study of the reactions of coal sulfur in the coking process: Ind. Eng. Chem. vol. 12, No. 11, p. 1069, 1920.

Campbell, J. R., Effect of aeration and "watering out" on sulfur content of coke: Bull. Amer. Inst. Min. Met. Eng. No. 109, pp. 177-80, 1916.

McCallum, A. L., The action of organic sulphur in coal during the coking process: The Chemical Engineer vol. 11, No. 1, pp. 27-28, Jan. 1910.

Sperr, F. W., Gas purification in relation to coal sulphur: Proc. Second Int. Conf. on Bit. Coal, pt. II, pp. 37-60, 1928.

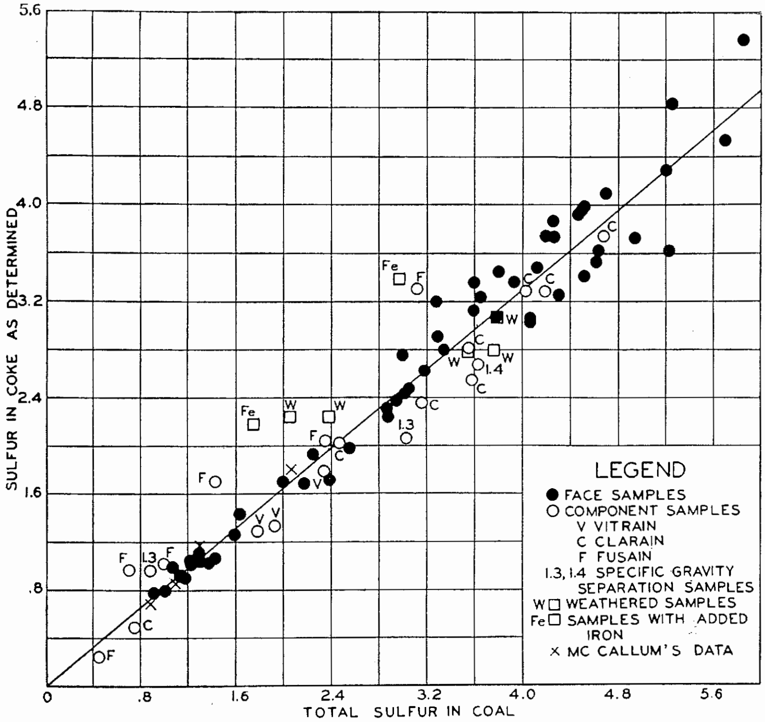


Fig. 44.—Relation of Sulfur Content of Coke to Sulfur Content of Coal. *(From "Behavior of Sulfur during Coal Carbonization," by G. Thiessen, Ind. and Eng. Chem. vol. 27, April, 1935.)*

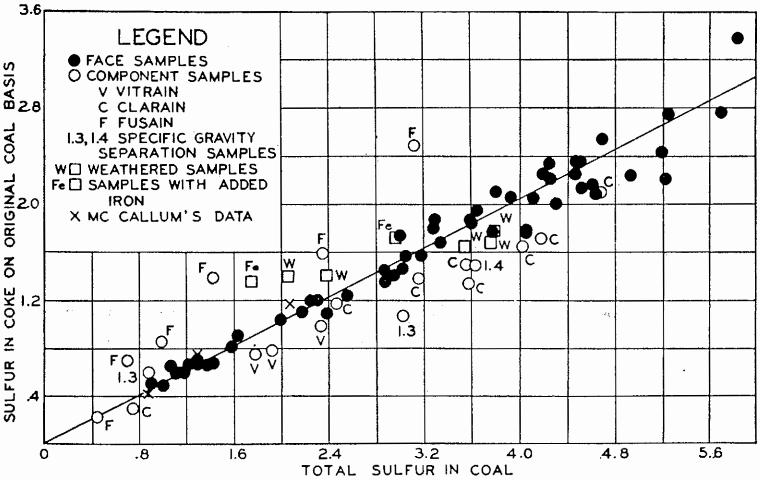


Fig. 45.—Sulfur Content of Coke, Expressed on Basis of Original Coal, as Function of Total Sulfur Content of Coal. *(From "Behavior of Sulfur during Coal Carbonization," by G. Thiessen, Ind. and Eng. Chem. Vol. 27, April, 1935.)*

coal. Four buttons were prepared from each sample except where rechecks were required. Analyses for pyritic and sulfate sulfur were made according to the method of Powell and Parr,[28] for other values by standard A.S.T.M. methods. A total of 82 samples was studied, including 55 mine face samples, 17 samples of banded coal components comprising four samples of anthraxylon (vitrain), seven samples of attritus (clarain), and six samples of fusain, three samples of coal treated by float-and-sink procedure, five samples of the same bed taken at points successively nearer the outcrop to determine the effect of weathering, and two samples of coal to which iron oxide had been added in order to simulate one of the characteristics of weathered coals.

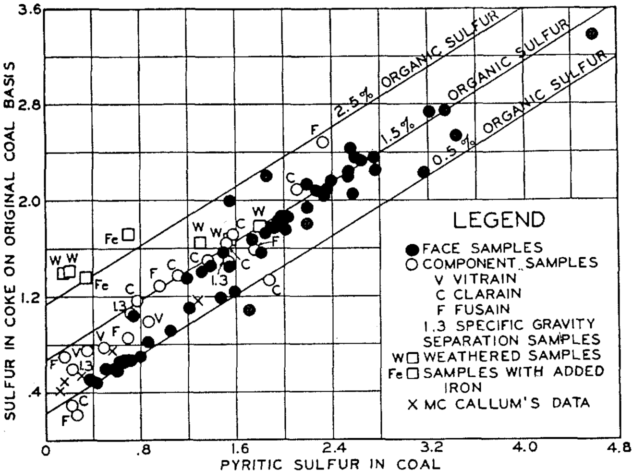


Fig. 46.—Sulfur Content of Coke, Expressed on Basis of Original Coal, as Function of Pyritic and Organic Sulfur Contents of Coal. *(From "Behavior of Sulfur during Coal Carbonization," by G. Thiessen, Ind. and Eng. Chem. vol. 27, April, 1935.)*

Studies on Face Samples.—The data pertaining to the face samples, including the percentage of organic, pyritic, and sulfate sulfur in the original coal samples are presented in Table 19. Better to visualize relationships which might exist, the data were plotted as follows: (1) Sulfur in the coke, as determined, against the total sulfur content of the coal (fig. 44); (2) the sulfur content of the coke expressed as a percentage of the original coal against the

---

[28] Powell, A. R., with Parr, S. W., A study of the forms in which sulphur occurs in coal: Univ. of Illinois Eng. Exp. Sta. Bull. 111, 1919.

Powell, A. R., The analysis of sulphur forms in coal: U. S. Bureau of Mines Tech. Paper 254, 1921.

Powell, A. R., Determination of sulfur forms in coal: Ind. Eng. Chem. vol. 12, No. 9, p. 887, 1920.

total sulfur content of the coal (fig. 45); and (3) the sulfur content of the coke, on the original coal basis, against the pyritic sulfur content of the coal (fig. 46). With few exceptions the points in all three cases fall close to straight lines. The two curves for which the total sulfur content of the coal is one of the variables (figs. 44 and 45) pass through the origin.

Sulfur in coke and total sulfur in coal.—When values for the sulfur in the coke were plotted against the values for the total sulfur content of the corresponding coal (fig. 44), it was found that the points fell reasonably well along a straight line whose equation is:

$$S_{\text{coke}} \text{ as determined} = 0.83\ S_{\text{coal}}$$

In figures 44 to 46 only those points representing face samples were used in determining the curves. These values are represented by solid dots whose radius is equal to the permissible error between laboratories for sulfur determinations on coke (0.05 per cent.)[29]

Table 19 shows the individual values for the ratio of S_{coke} to S_{coal}. These values range from 0.695 to 0.982 and average 0.831. This value is in agreement with the statement of Sperr[30]: "With coals containing from 0.5 to 3.0 per cent sulfur, the per cent sulfur in the furnace coke averages about 80 per cent of that in the coal." The agreement and the general similarity of figure 44 with the graph illustrating the relationship between coke and coal sulfur presented by Sperr permit the conclusion that the results of this laboratory study are directly applicable to standard by-product coke-oven practice.

Sulfur in coke (on basis of original coal) and total sulfur in coal.—By calculating the sulfur content of the coke to the basis of the original coal, it is possible to find the proportion of coal sulfur which remained in the coke and the proportion which entered the volatile matter. For the face samples studied it was found (fig. 45) that 51.9 per cent of the coal sulfur remained in the coke, the values ranging from 42.3 to 62 per cent. These points also lay reasonably close along a straight line whose equation was:

$$S_{\text{coke}} \text{ (coal basis)} = 0.519\ S_{\text{coal}}$$

The value given by Parr[31] was 48.5 per cent. The general statement that approximately half of the total coal sulfur remains in the coke is borne out by the present results.

---

[29] Amer. Soc. Testing Materials, Standards, Pt. II, pp. 387-426, Method D271-30, 1936, Amer. Standards Assoc. No. K18-1930, especially p. 426.

[30] Sperr, S. W., Op. cit., especially p. 51.

[31] Parr, S. W., The coals of Illinois: Op. cit.

TABLE 19.—SULFUR DATA FOR FACE SAMPLES OF ILLINOIS COALS[a]

1 Coke sample No.	2 Coal sample No.	3 County and coal No.	4 S_T per cent	5 S_{SO_4} per cent	6 S_P per cent	7 S_O per cent	8 F. C. per cent	9 Ash per cent	10 F. C. + Ash per cent	11 S-COKE per cent	12 S-COKE on coal basis per cent	13 Ratio S-COKE to S-coal on coal basis	14 Ratio S-coke detd. to S-coal	15 Ratio S_O/S_P	16 Ratio S_P/S_T	17 CO_2 per cent
846	11	Franklin 6	1.29	0.02	0.80	0.47	53.2	10.7	63.9	1.09	0.70	0.543	0.845	0.588	0.620	0.53
847	48		1.21	0.05	0.66	0.50	56.0	9.7	65.7	1.02	0.67	0.553	0.843	0.758	0.545	....
843	49		1.14	0.05	0.58	0.51	54.4	11.2	65.6	0.92	0.60	0.526	0.807	0.879	0.509	....
844	50		0.91	0.03	0.38	0.50	54.7	11.3	66.0	0.77	0.51	0.560	0.846	1.316	0.418	....
845	51		1.38	0.04	0.66	0.68	59.6	3.8	63.4	1.03	0.65	0.471	0.746	1.030	0.478	....
848	309		1.13	0.17	0.51	0.45	55.2	10.5	65.7	0.91	0.60	0.531	0.805	0.882	0.451	....
849	492		1.42	0.01	0.72	0.69	55.7	7.5	63.2	1.06	0.67	0.472	0.746	0.958	0.507	0.33
881	493		2.38	0.01	1.71	0.66	53.9	9.8	63.7	1.71	1.09	0.458	0.718	0.386	0.718	0.56
850	494		1.01	0.00	0.43	0.58	54.9	7.4	62.3	0.79	0.49	0.485	0.782	1.349	0.426	0.19
858	495		1.18	0.04	0.61	0.53	56.1	9.0	65.1	0.90	0.59	0.500	0.763	0.869	0.517	0.40
851	507		1.22	0.03	0.66	0.53	56.3	8.4	64.7	1.04	0.67	0.549	0.852	0.803	0.541	0.40
852	508		1.63	0.00	1.05	0.58	54.6	8.9	63.5	1.43	0.91	0.558	0.877	0.552	0.644	0.33
434	139	Henry 6	5.83	0.08	4.58	1.17	45.4	17.0	62.4	5.38	3.38	0.576	0.923	0.255	0.786	1.10
440	145		4.69	0.08	3.43	1.18	46.9	15.2	62.1	4.11	2.55	0.544	0.874	0.344	0.731	....
430	135	Knox 6	3.80	0.04	2.35	1.41	48.4	12.2	60.6	3.46	2.10	0.553	0.911	0.600	0.618	1.06
431	136		3.58	0.08	1.97	1.53	45.5	13.9	59.4	3.14	1.87	0.522	0.877	0.777	0.550	1.99
432	137		3.64	0.06	2.19	1.39	47.1	13.1	60.2	3.25	1.95	0.536	0.893	0.635	0.602	....
902	27	Macoupin 6	5.69	0.13	3.34	2.22	44.5	16.2	60.7	4.54	2.76	0.485	0.798	0.665	0.587	0.49
904	306		5.22	0.57	1.85	2.80	48.1	12.9	61.0	3.63	2.21	0.423	0.695	1.514	0.354	....
900	24	St. Clair 6	4.61	0.16	2.39	2.05	46.1	15.0	61.1	3.55	2.17	0.471	0.770	0.858	0.518	0.61
901	26		4.51	0.30	2.19	2.02	46.6	15.6	62.3	3.43	2.14	0.475	0.761	0.922	0.486	0.37
903	305		4.30	0.56	1.54	2.20	47.0	14.2	61.2	3.27	2.00	0.465	0.760	1.428	0.358	....
435	140	Stark 6	4.47	0.05	2.75	1.67	45.7	14.0	59.7	3.95	2.36	0.528	0.884	0.607	0.615	1.37
436	141		4.11	0.04	2.33	1.74	48.1	10.6	58.7	3.49	2.05	0.499	0.849	0.747	0.567	0.70
437	142		4.25	0.05	2.53	1.67	46.7	12.2	58.9	3.75	2.21	0.520	0.882	0.660	0.595	....
439	144		4.19	0.04	2.76	1.39	46.9	13.1	60.0	3.76	2.26	0.539	0.897	0.505	0.659	0.74

912	369	Washington 6	4.50	0.02	2.59	1.89	44.3	14.8	59.1	3.99	2.36	0.524	0.887	0.730	0.576	0.73
521	520		5.19	0.11	2.55	2.53	42.1	14.6	56.7	4.30	2.44	0.470	0.829	0.992	0.491	1.05
853	29	Williamson 6	2.17	0.07	1.21	1.91	54.7	10.3	65.0	1.69	1.10	0.507	0.779	1.579	0.558	0.25
855	30		2.86	0.16	1.54	1.15	52.9	9.7	62.6	2.31	1.45	0.507	0.808	0.747	0.538	0.76
854	31		3.28	0.15	2.03	1.10	52.2	11.9	64.1	2.92	1.87	0.570	0.890	0.542	0.619	0.94
856	32		3.04	0.15	1.81	1.07	52.7	10.8	63.5	2.48	1.57	0.516	0.816	0.591	0.595	0.77
857	37		1.58	0.03	0.86	0.69	54.9	9.8	64.7	1.26	0.82	0.519	0.797	0.802	0.544	....
861	192		2.24	0.05	1.47	0.72	56.8	4.7	61.5	1.93	1.19	0.531	0.862	0.490	0.656	0.21
862	193		1.29	0.01	0.68	0.60	54.0	10.6	64.6	1.04	0.67	0.519	0.806	0.882	0.527	0.24
863	194		1.07	0.01	0.63	0.43	52.1	13.2	65.3	0.99	0.65	0.607	0.925	0.683	0.589	1.05
859	39	Fulton 5	4.93	0.04	3.16	1.73	45.4	14.4	59.8	3.74	2.24	0.454	0.759	0.547	0.641	....
860	40		4.24	0.06	2.63	1.55	44.2	16.2	60.4	3.88	2.34	0.552	0.915	0.589	0.620	....
444	149	Menard 5	3.33	0.02	1.73	1.58	48.5	11.2	59.7	2.81	1.68	0.505	0.844	0.913	0.520	1.00
445	150		2.94	0.01	1.31	1.62	46.5	12.9	59.4	2.38	1.41	0.480	0.810	1.237	0.446	1.82
446	151		2.87	0.02	1.19	1.66	45.4	14.6	60.0	2.25	1.35	0.470	0.784	1.395	0.415	2.00
447	152		3.01	0.02	1.38	1.61	46.7	12.9	59.6	2.45	1.46	0.485	0.814	1.167	0.458	....
873	631		4.06	0.02	2.01	2.03	47.8	10.2	58.0	3.04	1.76	0.579	0.749	1.010	0.495	0.61
874	199	Saline 5	1.99	0.34	0.74	0.91	54.1	7.1	61.2	1.70	1.04	0.612	0.854	1.230	0.372	....
876	361		2.55	0.04	1.59	0.92	54.4	8.4	62.8	1.98	1.24	0.626	0.776	0.579	0.624	....
519	518		2.99	0.19	1.84	0.96	54.3	8.2	62.5	2.76	1.73	0.579	0.923	0.522	0.615	....
888	733	Sangamon 5	4.06	0.02	1.91	2.13	46.1	11.8	57.9	3.07	1.78	0.438	0.756	1.115	0.470	0.86
877	734		4.63	0.03	2.27	2.33	44.2	13.3	57.5	3.63	2.09	0.576	0.784	1.026	0.490	1.23
448	153	Bureau 7	3.92	0.03	2.57	1.32	43.7	17.1	60.8	3.38	2.06	0.526	0.862	0.514	0.656	0.86
449	154		3.17	0.03	1.49	1.65	44.6	15.1	59.7	2.63	1.57	0.495	0.830	1.107	0.470	1.15
441	146	Grundy 7 (?)	5.24	0.09	3.21	1.94	43.2	13.5	56.7	4.85	2.75	0.525	0.926	0.604	0.613	1.70
442	147		3.77	0.06	1.88	1.83	46.3	11.0	57.3	3.09	1.77	0.470	0.820	0.973	0.499	1.00
443	148		4.46	0.07	2.53	1.86	45.0	12.2	57.2	3.94	2.25	0.505	0.883	0.735	0.567	....
913	599	Woodford 2	3.27	0.06	3.19	1.02	48.7	7.8	56.5	3.21	1.81	0.554	0.982	0.466	0.670	0.51

---

[a] The columns have the following designations:
4. Total sulfur content of coal.
5. Sulfate sulfur content of coal.
6. Pyritic sulfur content of coal.
7. Organic sulfur content of coal.
8. Fixed carbon content of coal.
9. Ash content of coal.
10. Fixed carbon plus ash content of coal.
11. Sulfur in coke.
12. Sulfur in coke calcd. to basis of original coal (column 10 times column 11).
13. Ratio of fixed to total sulfur in coal (12 divided by 4).
14. Ratio of sulfur in coke to sulfur in coal (11 divided by 4).
15. Ratio of organic to pyritic sulfur in coal.
16. Ratio of pyritic to total sulfur in coal.

Sulfur in coke (on basis of original coal) and pyritic sulfur in coal.—When the values for the sulfur in the coke, calculated to the coal basis, were plotted against the pyritic sulfur content of the original coals, it appeared (fig. 46) that there were two groups of points, each defining a straight line. Reference to analyses of the coals corresponding to the points in the lower group showed that the organic sulfur contents of all these samples was remarkably uniform at about 0.5 per cent and that for the upper group, especially those which lay closest to the straight line, the organic sulfur contents were about 1.5 per cent (values from 0.76 to 2.0 averaged). This leads to the conclusion that the organic and pyritic forms of sulfur in the coal contribute proportionately to the sulfur in the coke. The equations for the curves were found by calculation from the individual values to be:

$$\text{Lower curve: } S_{\text{coke}} = 0.61\ S_p + 0.26$$

$$\text{Upper curve: } S_{\text{coke}} = 0.63\ S_p + 0.58$$

The sulfur in the coke may be represented by the equation:

$$S_{\text{coke}}\ (\text{coal basis}) = a\ S_p + b\ S_o$$

The constant (a) is 0.61 and 0.63 in the foregoing equations, and (b) is found to be 0.52 or 0.37, respectively, on the assumption that the organic sulfur contents were 0.5 and 1.5, respectively. The average equation would be:

$$S_{\text{coke}} = 0.62\ S_p + 0.45\ S_o$$

If the organic sulfur value, S_o, is varied by finite steps, this equation may be represented on Cartesian coordinates by a series of straight lines, since there is a straight line for each value of S_o chosen. Three such lines have been drawn on figure 46 at $S_o = 0.5$, 1.5, and 2.5 per cent, respectively.

The almost direct proportionality between coke sulfur and total coal sulfur is therefore probably due to the fact that approximately constant proportions of the pyritic and the organic sulfurs remain in the coke; the proportions are as shown in the foregoing—apparently 62 per cent of the pyritic and 45 per cent of the organic sulfur. From a practical standpoint these results are of interest in showing that it is the total sulfur reduction which is important in the coal cleaning, and that, since it is impossible to remove the organic sulfur content of the coal by coal cleaning processes, the organic sulfur content determines the minimum approachable sulfur value.

To test the validity of the above relationship, the sulfur contents of the cokes, on the coal basis, were calculated from the pyritic and organic sulfur contents of the coals by means of the equation given above. The results of these calculations are shown in Table 20.

TABLE 20.—COMPARISON OF DETERMINED AND CALCULATED COKE SULFUR CONTENTS

Coal No.	Sp per cent	So per cent	0.62 Sp + 0.45 So	S-coke on coal basis detd.	Difference
Face Samples					
11	0.80	0.47	0.71	0.70	+0.01
48	0.66	0.50	0.63	0.67	—0.04
49	0.58	0.51	0.59	0.60	—0.01
50	0.38	0.50	0.46	0.51	—0.05
51	0.66	0.68	0.72	0.65	+0.07
309	0.51	0.45	0.52	0.60	—0.08
492	0.72	0.69	0.76	0.67	+0.09
493	1.71	0.66	1.36	1.09	+0.27
494	0.43	0.58	0.53	0.49	+0.04
495	0.61	0.53	0.62	0.59	+0.03
507	0.66	0.53	0.65	0.67	—0.02
508	1.05	0.58	0.91	0.91	0.00
139	4.58	1.17	3.37	3.38	—0.01
145	3.43	1.18	2.66	2.55	+0.11
135	2.35	1.41	2.09	2.10	—0.01
136	1.97	1.53	1.91	1.87	+0.04
137	2.19	1.39	1.98	1.95	+0.03
27	3.34	2.22	3.07	2.76	+0.31
306	1.85	2.80	2.41	2.21	+0.20
24	2.39	2.05	2.40	2.17	+0.23
26	2.19	2.02	2.27	2.14	+0.13
305	1.54	2.20	1.94	2.00	—0.06
140	2.75	1.67	2.46	2.36	+0.10
141	2.33	1.74	2.23	2.05	+0.18
142	2.53	1.67	2.32	2.21	+0.11
144	2.76	1.39	2.34	2.26	+0.08
369	2.59	1.89	2.46	2.36	+0.10
520	2.55	2.53	2.72	2.44	+0.28
29	1.21	1.91	1.61	1.10	+0.51
30	1.54	1.15	1.47	1.45	+0.02
31	2.03	1.10	1.75	1.87	—0.12
32	1.81	1.07	1.60	1.57	+0.03
37	0.86	0.69	0.84	0.82	+0.02
192	1.47	0.72	1.24	1.19	+0.05
193	0.68	0.60	0.69	0.67	+0.02
194	0.63	0.43	0.58	0.65	—0.07
39	3.16	1.73	2.74	2.24	+0.50
40	2.63	1.55	2.33	2.34	—0.01
149	1.73	1.58	1.78	1.68	+0.10
150	1.31	1.62	1.54	1.41	+0.13
151	1.19	1.66	1.48	1.35	+0.13
152	1.38	1.61	1.58	1.46	+0.12
631	2.01	2.03	2.16	1.76	+0.40
199	0.74	0.91	0.87	1.04	—0.17
361	1.59	0.92	1.40	1.24	+0.16

TABLE 20.—COMPARISON OF DETERMINED AND CALCULATED COKE SULFUR CONTENTS (concluded)

Coal No.	Sp per cent	So per cent	0.62 Sp + 0.45 So	S-coke on coal basis detd.	Difference
518	1.84	0.96	1.57	1.73	—0.16
733	1.91	2.13	2.14	1.78	+0.36
734	2.27	2.33	2.46	2.09	+0.37
153	2.57	1.32	2.19	2.06	+0.13
154	1.49	1.65	1.67	1.57	+0.10
146	3.21	1.94	2.86	2.75	+0.11
147	1.88	1.83	1.99	1.77	+0.22
148	2.53	1.86	2.41	2.25	+0.16
599	2.19	1.02	1.82	1.81	+0.01
				Average	0.13
		Weathered Samples			
542	1.79	1.91	1.97	1.78	+0.19
543	1.28	1.87	1.64	1.65	—0.01
544	1.54	1.98	1.85	1.68	+0.27
545	0.20	1.99	1.02	1.41	—0.39
546	0.15	1.78	0.89	1.40	—0.51
385	0.71	2.30	1.48	1.07	+0.41
385+Fe_2O_3	0.69	2.24	1.44	1.72	—0.28
356—516	0.35	1.40	0.85	0.75	+0.10
356–516+ Fe_2O_3	0.34	1.37	0.83	1.36	—0.53
				Average	0.30
		Component Samples			
48	0.66	0.50	0.68	0.67	+0.01
66	0.23	0.62	0.42	0.60	—0.18
508	1.05	0.58	0.91	0.91	0.00
503	0.23	0.52	0.38	0.30	+0.08
506	0.27	0.17	0.24	0.22	+0.02
350	1.75	0.53	1.32	1.59	—0.27
354	0.49	1.42	0.94	0.78	+0.16
355	0.77	1.68	1.23	1.17	+0.06
356–516	0.35	1.40	0.85	0.75	+0.10
135	2.35	1.41	2.09	2.10	—0.10
134	0.69	0.27	0.55	0.86	—0.31
369	2.59	1.89	2.46	2.36	+0.10
385	0.71	2.30	1.48	1.07	+0.41
387	1.53	2.07	1.88	1.49	+0.39
372	0.16	0.50	0.32	0.70	—0.48
376	0.96	0.41	0.78	1.39	—0.61
377	7.72	0.75	5.12	6.50	—1.38
370	1.51	2.50	2.06	1.65	+0.41
373	1.57	2.60	2.14	1.72	+0.42
374	2.10	2.54	2.45	2.10	+0.35
378	1.36	2.12	1.82	1.50	+0.32
381	1.11	2.02	1.60	1.38	+0.22
				Average	0.29

COAL COMPONENTS.—Samples of vitrain (anthraxylon, fusain, and clarain (attritus) were included in this study for the purpose of determining whether or not the sulfur in any of the components behaved differently from that in the others. The values for these samples are presented in Table 21 and are represented on the curves by open circles with letters (V, F, C) designating the component. Although the points representing these samples are reasonably close to the curves indicated by the face samples, certain general trends are evident. The cokes from fusain have higher sulfur contents and cokes from vitrain and clarain have lower sulfur contents than do cokes from face samples of the same sulfur content. The high sulfur retention of the fusain can be explained by the frequent high calcite and pyrite content of fusain. Similarly, because the vitrain and clarain samples were hand-picked, it would be probable that these samples would be low in adventitious mineral matter such as cleats, partings, and pyrite nodules.

Samples of coal of low specific gravity obtained by float-and-sink procedure are also included in this group of samples, and, although not sufficient in number or variety to permit an accurate judgment, the results appear to conform with those for face samples.

EFFECT OF WEATHERING.—The samples used in this study were made available in the course of studies on the weathering of coal under other supervision and were peculiar in the quite wide variation in the proportion of organic, pyritic, and sulfate sulfur. Analysis of the coke buttons yielded anomalous results. The anomalies are thought to be the result of variations in the effects of weathering to which the coal had been subjected, the samples having been taken at points 82, 60, 40, 25, and 15 feet from the entrance of the drift. The analyses show that the organic sulfur content of these samples (Table 22) has probably not been seriously altered, the pyrite, on the contrary, has been oxidized to an increasing extent as the outcrop is approached, and is almost completely eliminated in the sample closest to the surface. The sulfate content of these samples, while unusually high for normal coals, is not sufficiently high to account for the sulfur from oxidized pyrite.

As the outcrop is approached, the sulfate sulfur content of the coal increases and then decreases. The ferrous sulfate which was formed by the oxidation of the pyrite has been leached or hydrolyzed to sulfuric acid and hydrated iron oxide. Since the high sulfur content of the cokes from these weathered coals would be unusual if the coals were normal, it was thought possible that the high sulfur retention was due to the iron oxides left in the coal from hydrolysis of the pyrite oxidation products. To test this, 2.5 per cent of ferric oxide (corresponding approximately to the iron associated with 2 per cent of pyritic sulfur) was added to each of two samples of coal, and the coke buttons from the mixture were carbonized. The sulfur contents of the cokes were materially increased by the iron oxide.

TABLE 21.—SULFUR DATA FOR COMPONENT BANDS OF ILLINOIS COALS[a]

1		2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
Sample No. coke	Sample No. coal	County and coal No.	Description	S_T per cent	S_{SO_4} per cent	S_P per cent	S_O per cent	F. C. per cent	Ash per cent	F. C. + Ash per cent	S-COKE per cent	S-COKE on coal basis per cent	Ratio S-coke/S-coal on coal basis	Ratio S-coke detd./S-coal	Ratio S_O/S_P	Ratio S_P/S_T	CO_2 per cent
847	48	Franklin 6	Face	1.21	0.05	0.66	0.50	56.0	9.7	65.7	1.02	0.67	0.553	0.843	0.758	0.620	....
910	66		F 1.3	0.88	0.03	0.23	0.62	61.4	1.6	63.0	0.96	0.60	0.682	1.091	2.696	0.261	....
852	508		Face	1.63	0.00	1.05	0.58	54.6	8.9	63.5	1.43	0.91	0.558	0.877	0.552	0.644	0.33
898	503		Clarain	0.75	0.00	0.23	0.52	53.5	7.3	60.8	0.49	0.30	0.400	0.653	2.261	0.307	0.12
899	506		Fusain	0.45	0.01	0.27	0.17	84.9	6.3	91.2	0.24	0.22	0.489	0.533	0.630	0.600	0.39
430	...	Jackson 6	Face	....	....	....	....	....	....	....	....	....	....	....	....	....	....
878	350		Fusain	2.34	0.06	1.75	0.53	62.8	15.2	78.0	2.04	1.59	0.679	0.872	0.303	0.748	....
879	354		Vitrain	1.92	0.01	0.49	1.42	56.2	2.7	58.9	1.33	0.78	0.406	0.693	2.878	0.255	....
880	355		Clarain	2.46	0.01	0.77	1.68	49.6	8.4	58.0	2.02	1.17	0.476	0.821	2.182	0.313	....
517	356–516		Vitrain	1.78	0.03	0.35	1.40	56.5	1.3	57.8	1.29	0.75	0.421	0.725	4.000	0.197	....
430	135	Knox 6	Face	3.80	0.04	2.35	1.41	48.4	12.2	60.6	3.46	2.10	0.553	0.911	0.600	0.618	1.06
429	134		Fusain	0.99	0.03	0.69	0.27	73.6	10.4	84.1	1.02	0.86	0.869	1.030	0.391	0.697	....
912	369	Washington 6	Face	4.50	0.02	2.59	1.89	44.3	14.8	59.1	3.99	2.36	0.524	0.887	0.730	0.576	0.73
905	385		F 1.28	3.02	0.01	0.71	2.30	49.2	2.8	52.0	2.06	1.07	0.354	0.682	3.239	0.235	....
907	387		F 1.35	3.62	0.02	1.53	2.07	47.1	8.6	55.7	2.68	1.49	0.412	0.740	1.353	0.423	....
892	372		Fusain	0.70	0.04	0.16	0.50	54.5	18.6	73.1	0.96	0.70	1.000	1.371	3.125	0.229	0.70
896	376		Fusain	1.42	0.05	0.96	0.41	64.0	17.5	81.5	1.70	1.39	0.979	1.197	0.427	0.676	8.41
897	377		Fusain	8.71	0.24	7.72	0.75	58.6	24.3	82.9	7.84	6.50	0.746	0.900	0.097	0.886	3.65
890	370		Clarain	4.02	0.01	1.51	2.50	44.9	5.0	49.9	3.30	1.65	0.410	0.821	1.656	0.376	0.22
891	373		Clarain	4.18	0.01	1.57	2.60	46.6	5.5	52.1	3.30	1.72	0.411	0.799	1.656	0.376	0.35
893	374		Clarain	4.67	0.03	2.10	2.54	43.7	12.1	55.8	3.76	2.10	0.450	0.805	1.210	0.450	0.70
894	378		Clarain	3.54	0.01	1.36	2.17	48.5	4.6	53.1	2.82	1.50	0.424	0.797	1.596	0.384	0.68
895	381		Clarain	3.15	0.02	1.11	2.02	46.1	12.4	58.5	2.36	1.38	0.438	0.749	1.820	0.352	0.76
807	803	Vermilion	Total	3.59	....	1.95[b]	1.64	48.0	6.6	54.6	3.37	1.84	0.513	0.939	0.841	0.543	....
808	804		Fusain	3.10	....	2.32[b]	0.78	57.6	17.6	75.2	3.31	2.49	0.803	1.068	0.336	0.748	....
809	805		Clarain	3.57	....	1.87[b]	1.70	46.4	6.3	52.7	2.55	1.34	0.375	0.741	0.909	0.524	....
810	806		Vitrain	2.33	....	0.87[b]	1.46	51.9	3.2	55.1	1.79	0.99	0.425	0.768	1.678	0.373	....

[a] Column designations, same as Table 22.

[b] Sulfate + pyritic sulfur.

Although coke would not ordinarily be made from coals as weathered as those studied here, these data show the serious influence which iron compounds in the coal can have on the sulfur content of the corresponding coke. Iron oxide in this respect is almost as bad as pyrite itself. These samples are designated on the graphs by squares with appropriate letters.

For the purposes of comparison the values found by McCallum[32] have also been plotted on the graphs. Considering the variations in the conditions under which the cokes were made, these values lie in good conformity to the present experimental points.

CONCLUSIONS REGARDING BEHAVIOR OF SULFUR DURING COAL CARBONIZATION.—The conclusions reached as the result of the study of the distribution of the forms of sulfur in the volatile matter are in conformity with reported general experience concerning the relationships between coal sulfurs and coke sulfurs in practical operation, as follows.

(1) The sulfur in coke is derived from both organic and pyritic sulfur in the coal; approximately 45 per cent of the organic sulfur and 60 per cent of the pyritic sulfur remain in the coke.

(2) Because the proportions of the organic and the pyritic sulfur which remain in the coke are of the same magnitude, the sulfur content of the coke is, for practical purposes, directly proportional to the total sulfur content of the coal. Approximately half of the sulfur in the coal remains in the coke. Because of the loss of volatile matter, the percentage of sulfur in the coke is about 80 per cent of the percentage of the sulfur in the coal from which the coke was made. Iron compounds other than pyrite in the coal, arising either from infiltration or from pyrite oxidation, increase the sulfur retention of the coke.

(3) The organic sulfur content of a coal and the lower limit to which its pyritic sulfur content may be reduced by cleaning procedures set the lower limit to the sulfur content of the coke made from that coal unless the coke is subjected to special sulfur removing procedures.

Sulfur behavior in the United States Steel Corporation carbonization.—On the basis of the results obtained in the coke button studies as noted above, certain conclusions were possible in regard to the probable behavior of sulfur during the United States Steel Corporation tests for coke and by-product yields described beginning on page 89 and in Appendix A. On the basis of the probable relationship between the amount of each of the varieties of sulfur in the coal and in the coke, the probable total sulfur content of the coke was calculated. This was then compared with the actual sulfur content of the coke as determined by analysis, in order to check the extent of agreement between theoretical and actual values. The experimental relationship between the per

---

[32] McCallum, A. L., The action of organic sulphur in coal during the coking process: The Chemical Engineer vol. 11, No. 1, pp. 27-28, Jan. 1910.

TABLE 22.—SULFUR DATA FOR SAMPLES OF WEATHERED ILLINOIS COALS[a]

1 Sample No. coke	2 Sample No. coal	3 County and coal No.	4 S_T per cent	5 S_{SO_4} per cent	6 S_P per cent	7 S_O per cent	8 F. C. per cent	9 Ash per cent	10 F. C. + Ash per cent	11 S_{COKE} per cent	12 S_{COKE} on coal basis per cent	13 Ratio S_{coke}/S_{coal} on coal basis	14 Ratio S_{coke} detd./S_{coal}	15 Ratio S_O/S_P	16 Ratio S_P/S_T	17 CO_2 per cent
864	542	Fulton 5	3.78	0.08 (1.87)[b]	1.79	1.91	48.0	10.3	58.3	3.07	1.78	0.471	0.812	1.067	0.474	0.75
865	543		3.53	0.38 (1.66)[b]	1.28	1.87	49.1	10.4	59.5	2.78	1.65	0.467	0.788	1.461	0.363	0.28
866	544		3.75	0.23 (1.77)[b]	1.54	1.98	50.1	9.9	60.0	2.80	1.68	0.448	0.747	1.286	0.411	0.14
867	545		2.37	0.18 (0.38)[b]	0.20	1.99	53.1	9.9	63.0	2.25	1.41	0.595	0.949	9.950	0.084	0.12
868	546		2.05	0.12 (0.27)[b]	0.15	1.78	50.3	12.3	62.6	2.24	1.40	0.683	1.093	11.867	0.073	0.14
905	385	Washington 6	3.02	0.01	0.71	2.30	49.2	2.8	52.0	2.06	1.07	0.354	0.682	3.239	0.235	....
906	385+Fe_2O_3		2.95	0.01	0.69	2.24	48.0	2.7	50.7	3.39	1.72	0.583	1.149	3.246	0.234	....
517	356[c]–516[d]	Jackson 6	1.78	0.03	0.35	1.40	56.5	1.3	57.8	1.29	0.75	0.421	0.725	4.000	0.197	....
911	356–516+Fe_2O_3		1.74	0.03	0.34	1.37	58.8	3.8	62.6	2.18	1.36	0.782	1.253	4.029	0.195	....

[a] The columns have the following designations:
4. Total sulfur content of coal.
5. Sulfate sulfur content of coal.
6. Pyritic sulfur content of coal.
7. Organic sulfur content of coal.
8. Fixed carbon content of coal.
9. Ash content of coal.
10. Fixed carbon plus ash content of coal.
11. Sulfur in coke.
12. Sulfur in coke calcd. to basis of original coal (column 10 times column 11).
13. Ratio of fixed to total sulfur in coal (12 divided by 4).
14. Ratio of sulfur in coke to sulfur in coal (11 divided by 4).
15. Ratio of organic to pyritic sulfur in coal.
16. Ratio of pyritic to total sulfur in coal.

[b] Pyritic + sulfate sulfur.

[c] For fixed carbon value.

[d] For sulfur values and ash.

cent of sulfur in the coke and coal was also determined. The coke sulfur content was calculated both neglecting the sulfate sulfur content using the expression

$$\text{Coke sulfur} = (0.60\ S_p + 0.45\ S_o)/\ \text{coke yield}$$

and considering the sulfate sulfur content using the expression

$$\text{Coke sulfur} = (0.60\ S_p + 0.45\ S_o + 1.00\ S_{so_4})/\text{coke yield}$$

The data and results of the calculation are presented in Table 23. Inspection of the table shows that the calculated values are low as compared with experimental determinations, the values obtained by including the sulfate values checking most closely with the experimental values. The fact that the coke sulfur content is high may be due to the great opportunity present in the apparatus for sulfur compounds which as hydrogen sulfide and mercaptans to crack and yield free sulfur which would combine with the very reactive low temperature coke first formed. The coals had also been dried in air in a finely divided state at a temperature of 105°C., conditions which are favorable to oxidation of pyritic sulfur. Oxidation of pyritic sulfur to sulfate sulfur would cause a much greater retention of sulfur by the coke. Sampling and analytical errors also can account for a considerable proportion of the differences.

Removal of sulfur from coke.—No practical method of removal of sulfur from coke has yet been discovered. Those proposed involve either (1) the reaction of the sulfur in the hot coke with steam, hydrogen, or coke oven gas to form gaseous sulfur compounds, mainly hydrogen sulfide, or (2) the addition of chemicals to the charge. The problem of the removal of sulfur from coke is complicated by the fact that the sulfur exists in coke in very intimate organic combination with or in solution in the carbon as well as in the form of sulfides. These intimately combined forms of sulfur are exceedingly difficult to remove, practically necessitating the destruction of the coke. These methods have been discussed and reviewed in the technical literature.[33]

GENERAL CONCLUSIONS

Illinois coals are of the common bright banded type of coal and are composed of jet black, vitreous appearing material derived from larger pieces of woody materials interspersed in a lustrous, striated mass derived from highly fragmented plant material, together with a minor amount of charcoal-like material. Because of the differing friabilities of these components their proportions in the sized fractions of naturally formed screenings differ from their proportions in the coal as it existed in the face. Such segregation is not especially

---

[33] Gluud, W. (Jacobson, D. L.), International handbook of the by-product coke industry: American Edition, New York, 1932, pp. 433-4.

Schellenberg, A., Über den Schwefel in der Steinkohle und die Entschwefelung des Kokses: Brenstoff-Chemie 2 (22), Nov. 15, 1921.

Powell, A. R., and Thompson, J. H., A study of the desulphurization of coke by steam: Carnegie Institute of Technology Cooperative Coal Mining Investigations Bull. 7, 1923.

TABLE 23.—RELATIONSHIP BETWEEN DISTRIBUTION OF SULFUR VARIETIES IN COAL AND SULFUR CONTENTS OF COKES MADE IN CARBONIZATION ASSAY TESTS

Carbonization No.	Sample No.	Sulfur varieties, per cent on dry basis				Calculated sulfur content of coke, per cent		Actual sulfur content of coke per cent	Difference per cent	Ratio S-coke to S-coal
		Sulfate	Pyritic	Organic	Total	Neglecting sulfate sulfur	Considering sulfate sulfur			
1	C–5	0.19	2.53	2.15	4.9	3.5	3.8	4.5	0.7	0.92
3	C–7	0.04	2.23	2.54	4.8	3.6	3.7	4.0	0.3	0.83
5	C–9	0.06	2.10	2.17	4.3	3.2	3.3	3.9	0.6	0.90
7	C–383	0.09	2.13	1.64	3.9	2.8	2.9	3.7	0.8	0.94
8	C–197	0.10	0.42	0.58	1.1	0.7	0.6	1.5	0.7	1.33
9	C–11	0.02	0.80	0.47	1.3	1.0	1.0	1.2	0.2	0.92
11	C–198	0.02	0.63	0.57	1.2	0.9	0.9	1.3	0.4	1.10
12	C–12	0.05	1.65	1.24	2.9	2.2	2.2	2.5	0.3	0.84
14	C–199	0.34	0.74	0.91	2.0	1.2	1.7	1.8	0.1	0.90
16	C–493	0.01	1.71	0.66	2.4	1.8	1.8	1.6	0.2	0.68
16[a]	C–497	0.04	0.16	0.60	0.8	0.5	0.6	0.91	0.3	1.14
16[b]	C–509	0.00	0.15	0.61	0.8	0.5	0.5	0.66	0.2	0.83

important for Illinois coals in the sizes in which coal is used for coke manufacture. Of greater importance is the concentration of the mineral matter in the smaller sizes of coal. It is highly desirable that the ash content of coal for coke making be as low as is practical. However caution must be taken to prevent the exposure during coal cleaning processes from destroying its caking power. This is especially important if the washed coal is dried by heat. The importance of sulfur as an impurity is not as great if the coke is not to be used for metallurgical purposes. It is a source of nuisance, however, and should be kept as low as possible. The minimum sulfur content attainable in a coke from a given coal is dependent upon the organic sulfur content and the minimum value to which the pyritic sulfur content of that coal can be reduced. No practical way has been found to eliminate sulfur from coke.

CHAPTER XV—COKE FROM ILLINOIS COALS AND BLENDS: SMALL SCALE TESTS

TESTS IN SIX-INCH RETORT

Very small scale laboratory tests do not usually yield much information about the physical quality of the coke produced from a given coal. To gain some idea of the effect of various factors on coke quality as judged by the shatter test and by observation, samples of coal weighing 5 to 7 pounds were carbonized in the six-inch retorts previously described. The tests were designed to determine the relative quality of the coke produced from coals originating in various parts of the state and the effects of high and medium (1000°C. and 800°C.) temperatures at completion of coking, of the addition of strongly caking low volatile coal, of the addition of inert carbonaceous materials, such as coke breeze and dedusting plant dust, and of the size of the coal as coked. The amount of coke remaining on the one-inch screen and the amount passing through a one-half inch screen have been used as criteria of coke quality. The percentage of coke remaining on the one-inch screen is an indication of the resistance of the coke to breakage by shattering; the amount passing the one-half inch screen is an indication of the resistance of the coke to abrasion and crumbling.[1]

COALS USED IN THE TESTS

Tests in this series were made on mine run samples of coal from the No. 6 bed from one mine in Franklin County and one mine in Randolph County; on a composite of face samples of coal from the No. 5 bed from one mine in Saline County; on a sample of commercially produced, minus ⅜-inch screenings from the No. 6 bed coal in Franklin County; on a sample of commercially produced, washed minus ⅜-inch screenings from the No. 6 bed in Perry County; and on mine run coal from the No. 2 bed from one mine in Woodford County. Low volatile matter, highly caking coal for blending was represented by a sample of Pocahontas coal from the Pocahontas No. 3 bed of West Virginia obtained from a local coal dealer. Coke breeze obtained by crushing and grinding commercial by-product coke was used as inert carbonaceous matter, and dedusting plant dust obtained from a commercial installation in Franklin County was used as a source of fusain. The composition of this dedusting plant dust is discussed in detail under the section of this report devoted to the influence of petrographic composition of the coal on coke quality. Analyses of these samples are presented in Table 24. In addition coal tar obtained from the local gas works and heavy petroleum oil were used in some tests.

---

[1] Mott, R. A., and Wheeler, R. V., Coke for blast furnaces: pp. 73-116. London, 1936.

TABLE 24.—ANALYSES OF COALS TESTED OR USED FOR BLENDS

Sample No.	Description	As Received Basis						Agglutinating value, Kg.	Softening temperature °C
		Moisture per cent	Ash per cent	Volatile matter per cent	Fixed carbon per cent	Sulfur per cent	Calorific value B.t.u./lb.		
C613	Franklin Co. No. 6	8.3	10.2	33.2	48.3	1.00	11632	3.2	376
C648	Randolph Co. No. 6	10.1	17.8	33.6	38.5	4.47	10041	4.3	352
C748–50	Saline Co. No. 5, (Average)	6.0	7.8	34.6	51.6	2.01	12914	3.7	378
C769	Franklin Co. No. 6 – 3/8 inch slack	9.1	10.4	31.0	49.5	1.19	11536	...	...
C770	Perry Co. No. 6 – 3/8 inch, washed	12.7	6.9	35.5	44.9	2.58	11454	...	...
C612	Woodford Co. No. 2	12.7	8.3	34.9	44.1	1.65	11433	4.0	374
C491	Dedusting plant dust	9.6	9.8	27.1	53.5	1.19	11746	...	...
C610	Coke breeze	0.2	9.3	1.2	89.3	0.6	12855	...	...
C611	Pocahontas No. 3, West Virginia	2.7	2.7	16.7	77.9	0.5	14834	9.9	406

RESULTS OF TESTS IN SIX-INCH RETORTS

The results of these tests are most conveniently studied in tabular and graphic form. Table 25 records the samples and blends which were carbonized, the final temperatures in the center of the charge, the average rate of temperature rise, the quantities of coke remaining on the one-inch and passing through the one-half inch screen after the fourth drop in the shatter test, and the number of the corresponding illustration picturing the coke as removed from the retort. Table 26 presents screen analyses of the cokes as removed from the retort and detailed results of the shatter tests. The cokes as removed from the retort are shown photographed against a grid with 3-inch squares in figures 47 to 53 inclusive.

It is difficult to draw definite and uncontrovertible conclusions from these tests. The number of tests involved is too small to furnish sufficient data to determine accurately the effects of the factors which were studied. The number of tests which could be made were limited by time and by the available assistance. Much experience and considerable work by previous investigators can, however, be drawn upon in interpreting the results.

Effect of coal used.—The geographic or geologic location of the coal, other factors being similar, seemed to be a minor factor. Such factors as elapsed time between removal from the mine face and carbonization, size of coal, and conditions of carbonization were more important. Cokes made under similar conditions from different coals can be compared as follows:

Origin of Coal	Figure	Test	Conditions	
No. 6 Bed, Franklin Co.	47a	3	20-mesh	1000 °C.
No. 6 Bed, Randolph Co.	49c	15	20-mesh	1000 °C.
No. 5 Bed, Saline Co.	52a	35	20-mesh	1000 °C.
No. 2 Bed, Woodford Co.	53c	2	20-mesh	1000 °C.
Pocahontas	53d	24	40-mesh	1000 °C.
No. 6 Bed, Randolph Co.	49e	22	4 -mesh	1000 °C.
No. 6 Bed, Perry Co.	53b	33	−⅜ inch	825 °C.
No. 6 Bed, Franklin Co.	53a	31	−⅜ inch	1000 °C.
No. 6 Bed, Randolph Co.	49d	23	20-mesh	800 °C.
No. 5 Bed, Saline Co.	52b	36	20-mesh	850 °C.

Effect of the size of coal as coked.—The use of fine coal represented by the 20-mesh samples resulted in the formation of coke with very small cell structure and cross fractures when the coal formed a completely fused mass, as compared with the 4-mesh or large coal. In the cylindrical retort used, the cross fracturing showed up as concentric rings or onion-peel structure in the cokes. Compare figures 49c and 49d with figures 49e, 50a and 50b. The effect is also apparent when figures 53a and 53b are compared with 53c and 53d, although the coals involved are different. The onion-peel structure was not very evident in the granular cokes made from mixtures rich in non-caking material, figures 48a to 48e and figure 50e. The larger sized coal gave the better coke.

TABLE 25.—SUMMARY OF COKE QUALITY TESTS

Test[a] No.	Composition of charge	Final temperature °C	Average rate of temperature rise, °C per minute	"Stability" per cent retained on 1 inch screen after 4 drops	"Friability" per cent passing through ½ inch screen after 4 drops	Figure No.
	Mine Run No. 6 Bed Coal from Franklin County					
3	100% No. 6 coal, 20-mesh	1000	11.1	64	2.3	47a
11	100% No. 6 coal, 20-mesh	900	4.6	89	3.0	47b
4	80% No. 6 coal, 20% Pocahontas	1100	10.3	75	2.2	47c
6	80% No. 6 coal, 20% Pocahontas, wet	1100	12.3	42	3.9	47d
8	60% No. 6 coal, 40% Pocahontas, wet	1100	11.8	65	3.1	47e
12	80% No. 6 coal, 20% coke breeze, wet	....	....	89	7.8	47f
5	80% No. 6 coal, 20% dust	1100	10.0	71	3.5	48a
7	80% No. 6 coal, 20% dust, wet	1100	10.0	60	3.0	48b
13	75% No. 6 coal, 20% coke breeze, 5% dust	1100	9.9	64	22.8	48c
10	70% No. 6 coal, 10% Pocahontas, 20% coke, breeze wet	1100	10.8	95	3.5	48d
9	73% No. 6 coal, 20% Pocahontas, 7% dust, wet	1100	5.8	91	3.7	48e
26	95% No. 6 coal, 5% tar	1025	10.2	70	3.1	48f
27	90% No. 6 coal, 10% tar	1050	9.6	61	3.1	49a
25	95% No. 6 coal, 5% petroleum oil	1000	8.7	80	5.0	49b
	Mine Run No. 6 Bed Coal from Randolph County					
15	100% No. 6 coal, 20-mesh	1100	11.1	85.5	3.0	49c
23	100% No. 6 coal, 20-mesh	800	10.1	77.8	5.9	49d
17	100% No. 6 coal, 4-mesh	1000	4.8	86.4	5.8	49e
22	100% No. 6 coal, 4-mesh	800	8.1	....	....	50a
14	100% No. 6 coal, 4-mesh	1100	6.9	87.1	5.2	50b
18	80% No. 6 coal, 20% Pocahontas	975	10.2	92.2	5.0	50c
21	80% No. 6 coal, 20% Pocahontas	1000	8.4	81.0	3.6	50d
19	80% No. 6 coal, 20% coke breeze	1000	7.9	85.7	8.1	50e
20	75% No. 6 coal, 20% Pocahontas, 5% dust	1025	13.7	91.5	3.7	50f
28	95% No. 6 coal, 20-mesh, 5% tar	1025	11.3	80.2	4.1	51a
29	95% No. 6 coal, 4-mesh, 5% tar	1025	8.9	82.2	8.3	51b
16	100% No. 6 coal, wet	1000	11.0	94.0	3.4	51c
40	100% No. 6 coal, cleaned at sp. gr. 1.5	1000	6.1	84.6	2.5	51d

Number 5 Bed Coal from Saline County

35	100% No. 5 coal	1000	12.7	69.7	4.5	52a
36	100% No. 5 coal	850	8.4	84.4	4.5	52b
37	85% No. 5 coal, 15% Pocahontas	1000	5.4	93.3	2.4	52c
38	85% No. 5 coal, 15% Pocahontas	850	9.2	81.4	3.6	52d

Number 6 Bed Coal from Franklin County

31	100% washed – 3/8 inch No. 6 coal	1000	7.2	88.1	4.3	53a

Number 6 Bed Coal from Perry County

33	100% washed – 3/8 inch No. 6 coal	825	5.6	73.8	6.9	53b

Number 2 Bed Coal from Woodford County

2	100% No. 2 bed coal, 20-mesh	1100	11.1	62.5	3.3	53c

Pocahontas No. 3 Bed Coal from West Virginia

24	100% Pocahontas, 40-mesh	1100	9.3	68.2	2.4	53d

[a] Corresponds to coke number in text.

TABLE 26.—SIZE DISTRIBUTION OF COKES AS RECOVERED FROM THE RETORTS AND RESULTS OF SHATTER TESTS

Coke	Size in inches	Percentage of each size as recovered from retort	Percentage based on + 1 inch as 100	Size distribution (per cent) after shatter test drop No.			
				1	2	3	4
1	+2	28.1	30.3	5.4	0	0	0
	−2+1	64.6	69.7	75.0	70.0	61.2	49.0
	−1+½	3.3		17.2	26.1	33.7	44.3
	−½	4.0		2.4	3.9	5.1	6.7
2	+2	3.9	4.5	0	0	0	0
	−2 +1	84.0	95.5	86.7	76.5	68.5	62.5
	−1 +½	8.4		12.2	21.4	29.2	34.2
	−½	2.7		1.1	2.1	2.3	3.3
3	+2	3.8	4.0	0	0	0	0
	−2 +1	90.5	96.0	78.8	76.8	67.8	63.8
	−1 +½	5.3		20.6	21.7	30.1	33.9
	−½	0.4		0.6	1.5	2.1	2.3
4	+2	34.5	35.7	3.1	0	0	0
	−2 +1	63.0	64.3	84.8	80.0	80.0	74.7
	−1 +½	1.3		11.3	18.5	18.1	23.1
	−½	1.2		0.8	1.5	1.9	2.2
5	+2	36.1	39.3	3.2	0	0	0
	−2 +1	55.7	60.7	85.7	82.7	76.6	70.5
	−1 +½	5.2		9.9	15.8	21.8	26.0
	−½	3.0		1.2	1.5	2.6	3.5
6	+2	21.4	22.9	0	0	0	0
	−2 +1	72.5	77.1	84.5	70.5	50.3	41.8
	−1 +½	2.3		14.6	27.4	46.5	54.3
	−½	3.8		0.9	2.1	3.2	3.9
7	+2	20.2	26.7	0	0	0	0
	−2 +1	74.0	78.3	79.8	70.5	60.7	60.2
	−1 +½	2.5		18.3	27.6	36.9	36.8
	−½	3.3		1.9	1.9	2.4	3.0
8	+2	26.3	28.2	0	0	0	0
	−2 +1	66.8	71.8	84.2	75.7	73.3	64.7
	−1 +½	4.1		14.6	22.1	23.7	32.2
	−½	2.8		1.2	2.2	3.0	3.1
9	+2	56.1	57.2	7.4	0	0	0
	−2 +1	42.0	42.8	88.0	93.5	92.3	90.7
	−1 +½	0.7		3.2	4.5	5.3	5.6
	−½	1.2		1.4	2.0	3.4	3.7
10	+2	95.3	98.1	63.5	46.3	45.5	45.2
	−2 +1	1.9	1.9	34.7	50.1	50.3	49.7
	−1 +½	0.5		0.8	1.8	1.5	1.6
	−½	2.3		1.0	1.8	2.7	3.5

TABLE 26.—CONTINUED

Coke	Size in inches	Percentage of each size as recovered from retort	Percentage based on +1 inch as 100	Size distribution (per cent) after shatter test drop No.			
				1	2	3	4
11	+2	65.0	66.6	8.4	4.6	4.6	0
	−2 +1	32.7	33.4	86.3	88.6	86.0	88.8
	−1 +½	1.0		4.0	4.9	6.7	8.2
	−½	1.3		1.3	1.9	2.7	3.0
12	+2	89.0	98.2	81.2	45.2	44.3	31.8
	−2 +1	1.7	1.8	16.3	48.8	46.7	57.8
	−1 +½	1.1		0.6	1.5	2.5	2.6
	−½	8.2		1.9	4.5	6.5	7.8
13	+2	49.5	74.5	50.8	31.2	31.2	30.0
	−2 +1	16.9	25.5	36.7	44.2	37.4	33.7
	−1 +½	5.0		5.2	10.2	11.0	13.5
	−½	28.6		7.3	14.4	20.4	22.8
14	+2	100.0	100.0	48.3	49.2	37.2	31.1
	−2 +1	1 piece		45.1	43.7	52.7	56.0
	−1 +½			3.5	4.9	5.9	7.7
	−½			3.1	2.2	4.2	5.2
15	+2	54.6	57.9	0	0	0	0
	−2 +1	39.6	42.1	94.5	90.2	86.2	85.5
	−1 +½	3.8		4.5	8.0	11.4	11.5
	−½	2.0		1.0	1.8	2.4	3.0
16	+2	34.3	36.1	4.6	4.6	0	0
	−2 +1	60.8	63.9	92.5	91.0	94.5	94.0
	−1 +½	1.4		1.4	2.2	2.8	2.6
	−½	3.5		1.5	2.2	2.7	3.4
17	+2	93.5	94.8	77.5	46.8	29.1	20.6
	−2 +1	5.2	5.2	18.0	44.5	60.4	65.8
	−1 +½	0.3		2.8	5.4	6.4	7.8
	−½	1.0		1.7	3.3	4.1	5.8
18	+2	96.7	100.0	55.4	45.5	36.3	30.8
	−2 +1	0		41.8	50.0	58.1	61.4
	−1 +½	0.3		0.8	1.4	1.4	2.8
	−½	3.0		2.0	3.1	4.2	5.0
19	+2	94.5	97.0	79.3	62.7	64.0	54.4
	−2 +1	2.9	3.0	13.0	26.3	21.6	31.3
	−1 +½	0.3		4.7	5.7	7.3	6.2
	−½	2.3		3.0	5.3	7.1	8.1
20	+2	56.5	57.5	15.0	4.8	0	0
	−2 +1	41.7	42.5	83.0	90.5	94.6	91.5
	−1 +½	0.2		0.8	2.4	2.4	4.8
	−½	1.6		1.2	2.3	3.0	3.7

TABLE 26.—CONTINUED

Coke	Size in inches	Percentage of each size as recovered from retort	Percentage based on +1 inch as 100	Size distribution (per cent) after shatter test drop No.			
				1	2	3	4
21	+2	23.2	25.4	0	0	0	0
	−2 +1	68.3	74.6	93.5	86.5	85.7	81.0
	−1 +½	3.9		5.3	11.1	11.3	15.4
	−½	4.6		1.2	2.4	3.0	3.6
23	+2	69.0	72.9	17.2	0	0	0
	−2 +1	25.7	27.1	76.8	89.0	82.2	77.8
	−1 +½	1.9		4.0	7.1	12.7	16.3
	−½	3.4		2.0	3.9	5.1	5.9
24	+2	2.7	3.2	0	0	0	0
	−2 +1	82.0	96.8	86.8	79.7	71.3	68.2
	−1 +½	11.9		12.5	18.9	26.7	29.4
	−½	3.4		0.7	1.4	2.0	2.4
25	+2	42.7	29.6	0	0	0	0
	−2 +1	64.3	70.4	92.7	87.0	82.8	79.5
	−1 +½	3.0		5.5	10.5	13.2	15.5
	−½	5.7		1.8	2.5	4.0	5.0
26	+2	42.7	45.4	0	0	0	0
	−2 +1	51.4	54.6	84.3	76.6	71.0	70.0
	−1 +½	1.2		14.6	21.2	26.5	26.9
	−½	4.7		1.1	2.2	2.5	3.1
27	+2	3.7	4.0	0	0	0	0
	−2 +1	89.7	96.0	87.0	71.6	68.2	61.3
	−1 +½	4.1		12.1	26.2	29.2	35.6
	−½	2.5		0.9	2.2	2.6	3.1
28	+2	45.6	49.6	0	0	0	0
	−2 +1	46.3	50.4	90.7	87.5	82.0	80.2
	−1 +½	3.3		8.1	10.3	14.7	15.7
	−½	4.8		1.2	2.2	3.3	4.1
29	+2	90.7	93.5	65.0	26.7	36.4	19.2
	−2 +1	6.3	6.5	29.2	63.3	51.0	63.0
	−1 +½	0.7		3.5	5.2	6.3	9.5
	−½	2.3		2.3	4.8	6.3	8.3
30	+2	80.5	82.8	56.4	27.0	23.2	18.0
	−2 +1	16.7	17.2	40.7	65.2	65.3	67.1
	−1 +½	.6		2.1	5.2	8.1	10.6
	−½	2.2		.8	2.6	3.4	4.3
31	+2	93.8	96.5	63.8	57.0	47.0	34.9
	−2 +1	3.4	3.5	31.7	35.8	42.5	53.2
	−1 +½	.5		3.3	4.5	7.2	7.8
	−½	2.0		1.5	2.7	3.3	4.1

TABLE 26.—CONCLUDED

Coke	Size in inches	Percentage of each size as recovered from retort	Percentage based on +1 inch as 100	Size distribution (per cent) after shatter test drop No.			
				1	2	3	4
32	+2	56.4	57.5	11.1	0	0	0
	−2 +1	41.7	42.5	73.7	65.8	60.5	50.5
	−1 +½	1.3		13.1	29.3	33.1	41.1
	−½	.6		2.1	4.9	6.4	8.4
33	+2	85.0	86.7	30.1	6.9	0	0
	−2 +1	13.0	13.3	64.5	79.2	78.4	73.8
	−1 +½	.6		3.4	9.9	15.8	19.3
	−½	1.4		2.0	4.0	5.8	6.9
34	+2	85.9	87.8	42.9	16.9	11.7	
	−2 +1	11.9	12.2	48.5	69.9	68.2	72.0
	−1 +½	1.3		6.3	9.3	14.4	19.9
	−½	.9		2.3	3.9	5.7	7.2
35	+2	40.1	41.9	0	0	0	0
	−2 +1	55.6	58.1	89.1	77.4	70.0	69.7
	−1 +½	2.2		9.4	20.1	26.5	25.8
	−½	2.1		1.5	2.5	3.5	4.5
36	+2	73.9	76.2	31.8	16.3	11.5	9.4
	−2 +1	23.0	23.76	64.6	74.3	76.6	75.0
	−1 +½	1.5		2.2	7.2	8.3	11.1
	−½	1.6		1.4	2.2	3.6	4.5
37	+2	83.3	84.0	31.2	15.9	7.7	7.7
	−2 +1	15.9	16.0	66.2	80.9	85.6	85.6
	−1 +½	.2		2.0	1.9	4.7	4.3
	−½	.6		.6	1.3	2.0	2.4
38	+2	63.9	64.7	16.3	4.3	4.3	0
	−2 +1	34.8	35.3	79.2	86.6	82.9	81.4
	−1 +½	.1		3.1	6.8	9.8	15.0
	−½	1.2		1.4	2.3	3.0	3.6
39	+2	23.9	25.3	0	0	0	0
	−2 +1	70.4	74.7	89.4	82.5	73.7	64.8
	−1 +½	3.8		10.5	15.8	23.7	31.9
	−½	1.9		.1	1.7	2.6	3.3
40	+2	58.9	59.6	4.7	4.7	0	0
	−2 +1	40.0	40.4	90.2	88.0	86.3	84.6
	−1 +½	.7		4.5	6.1	11.7	12.9
	−½	.4		.6	1.2	2.0	2.5

Effect of the addition of non-caking carbonaceous material.—Cokes from Illinois coals shrink excessively when devolatilized due to the high volatile matter content of the coal. This shrinkage results in the formation of fingery and much fractured coke which yields an excessive amount of breeze. The addition of low volatile content, non-caking carbonaceous material has been found an effective remedy in certain cases. The present tests show that the addition of coke or dedusting plant dust to Illinois coals tends to give more blocky and less fractured coke with a higher resistance to shattering; but that, because of the relatively poor caking power of Illinois coals, the coke is more friable and may even crumble quite readily, when appreciable quantities of the non-caking materials are added. Compare figure 47a (coke 3) with figures 47f, 48a, 48b, 48c (cokes 12, 5, 7, and 13) and figure 49c (coke 15) with figure 50e (coke 19). The addition of a strongly caking, low volatile coal to the mixture of coal and non-caking material did not provide any considerable improvement, probably because the Illinois coal and the low-volatile coal became plastic during different temperature ranges. See figures 48d, 48e, and 50f (cokes 10, 9, and 20). The addition of non-caking material such as coke dust or dedusting plant dust (fusain) is recommended only if the coal has sufficient caking power to permit complete incorporation of the inert material in the coke.

Effect of low volatile coal.—The addition of low volatile content coal is another way in which excessive shrinkage of coke is sometimes minimized. Brewer and Atkinson[2] have pointed out, however, that the difference in softening point of low and high rank coals may make them non-compatible and that coke from a mixture of two coals of widely different plastic temperature ranges may be weaker than cokes made from either of the coals alone. This was found to be the case with the coals studied in these tests, although there was indication of improvement in some cases. Compare figure 47a (coke 3) with figures 47c, 47d and 47e (cokes 4, 6, 8); figure 49c (coke 15) with figure 50d (coke 21); figure 49d (coke 23) with figure 50c (coke 18); figures 52a and b (cokes 35 and 36) with figures 52c and d (cokes 37 and 38). It is possible that a more compatible low volatile coal can be found, but no attempt to do so was made during the course of this work.

Effect of the addition of tar or petroleum oil.—The addition of coal tar or petroleum oil to the coal in an attempt to increase the quantity of cementing materials was not encouraging. The addition of petroleum oil gave a poorly structured, abradable coke with much fines, figure 49b (coke 25), probably because of the well known incompatibility between petroleum oils and coal tars. The addition of coal tar did not improve the structure of the cokes probably because the greater part of the tar was volatilized before the coal became plastic

---

[2] Brewer, R. E., and Atkinson, R. G., Plasticity of coals: Ind. Eng. Chem. Anal. Ed. vol. 8, No. 6, pp. 433-9, Nov. 1936.

and coked. Compare figure 47a (coke 3) with figures 48f and 49a (cokes 26 and 27; figure 49c (coke 15) with figure 51a (coke 28; and figure 49e (coke 17) with figure 51b (coke 29).

Influence of the rate of heating and final temperature.—The use of lower rates of heating may cause better coke structure by preventing overcoking and fingering of the coke already formed, but this is not definitely established. A low final temperature does not tend to prevent much of the fracturing. Compare figures 47a (coke 3) with figure 47b (coke 11); figure 52a (coke 35) with figure 52b (coke 36); figure 52c (coke 37) with figure 52d (coke 38); and figure 49c (coke 15) with figure 49d (coke 23).

Effect of added moisture.—The effect of moisture added to prevent segregation of certain of the mixtures was apparently deleterious, unless the effect noted was actually caused by the added materials which were prevented from segregating or being blown out of the charge by the evolved gases. Compare figure 47d (coke 4) with figure 47e (coke 6); figure 48a (coke 5) with figure 48b (coke 7); and figure 49c (coke 15) with figure 51e (coke 16).

Effect of removal of mineral matter.—The effects of reduction of mineral matter on the coke structure did not show up sufficiently in these tests to warrant conclusions. One reason for this was the few tests in which this factor could be directly evaluated; another was the small amount of coal used in most of the tests. See figure 51d (coke 40), figure 53a (coke 31), and figure 53b (coke 33).

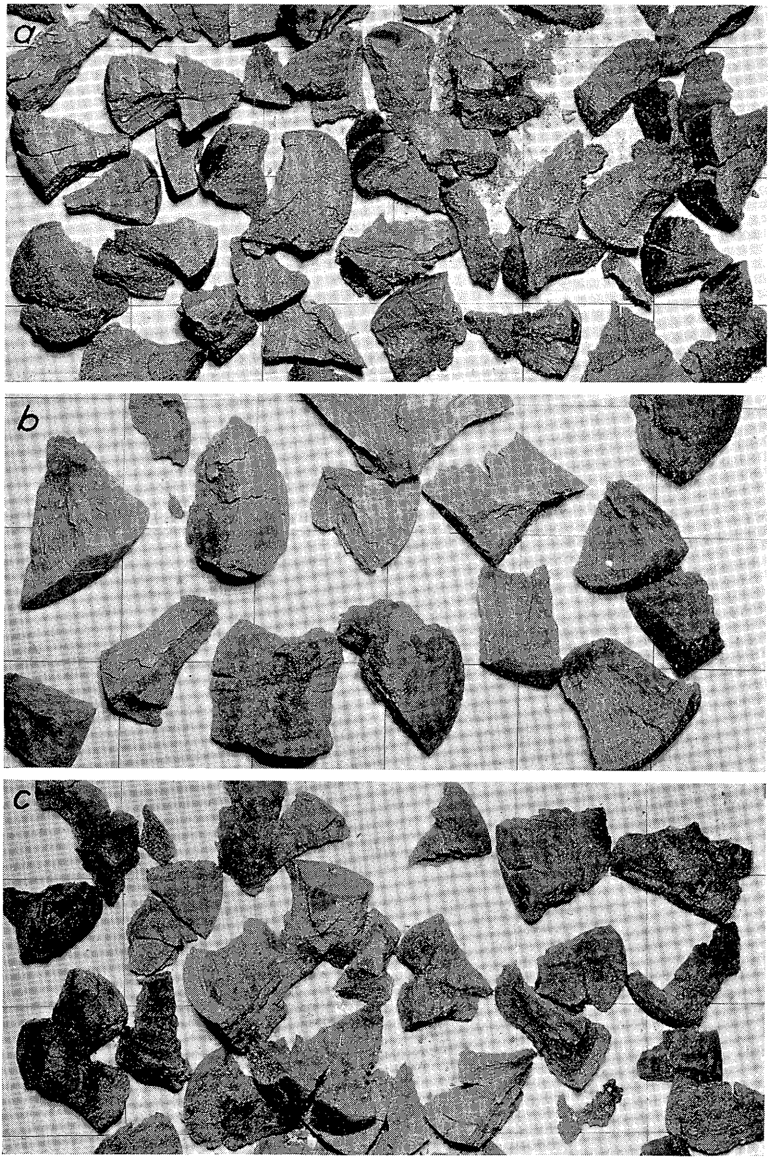


Fig. 47.—Cokes from Franklin County, Illinois, No. 6 Coal, Made in Six-inch Retort (3-inch grid).

a. 100% No. 6 coal, 20-mesh; 11.1°C. per minute to 1000°C. (Coke No. 3).
b. 100% No. 6 coal, 20-mesh; 4.6°C. per minute to 900°C. (coke No. 11).
c. 80% No. 6 coal, 20% Pocahontas; 10.3°C. per minute to 1100°C. (coke No. 4).

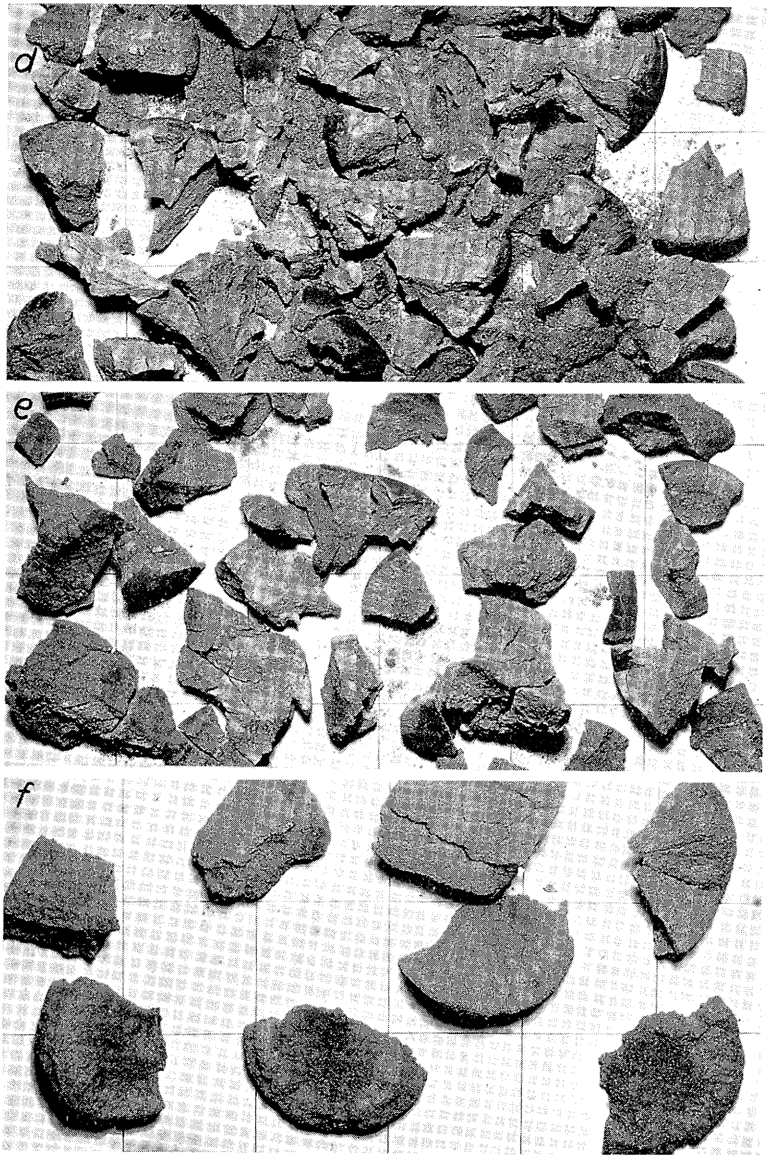


Fig. 47 (*con't.*).—COKES FROM FRANKLIN COUNTY, ILLINOIS, NO. 6 COAL, MADE IN SIX-INCH RETORT (3-INCH GRID).

d. 80% No. 6 coal, 20% Pocahontas, wetted; 12.3° C. per minute to 1100° C. (coke No. 6).
e. 60% No. 6 coal, 40% Pocahontas, wetted; 11.8° C. per minute to 1100° C. (coke No. 8).
f. 80% No. 6 coal, 20% coke breeze, wetted; (no temp. data) (coke No. 12).

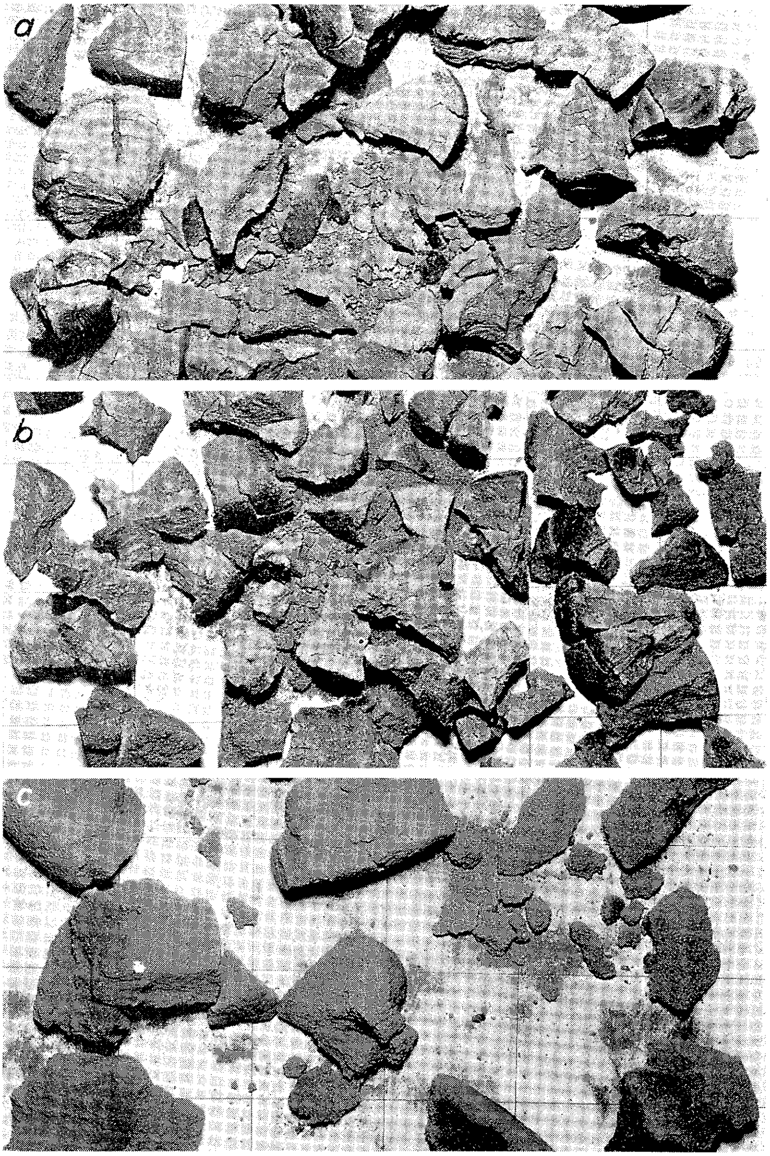


Fig. 48.—Cokes from Franklin County, Illinois, No. 6 coal, Made in Six-inch Retort (3-inch grid).

a. 80% No. 6 coal, 20% dust; 10.0° C. per minute to 1100° C. (coke No. 5).
b. 80% No. 6 coal, 20% dust, wetted; 10.0° C. per minute to 1100° C. (coke No. 7).
c. 75% No. 6 coal, 20% coke breeze, 5% dust; 9.9° C. per minute to 1100° C. (coke No. 13).

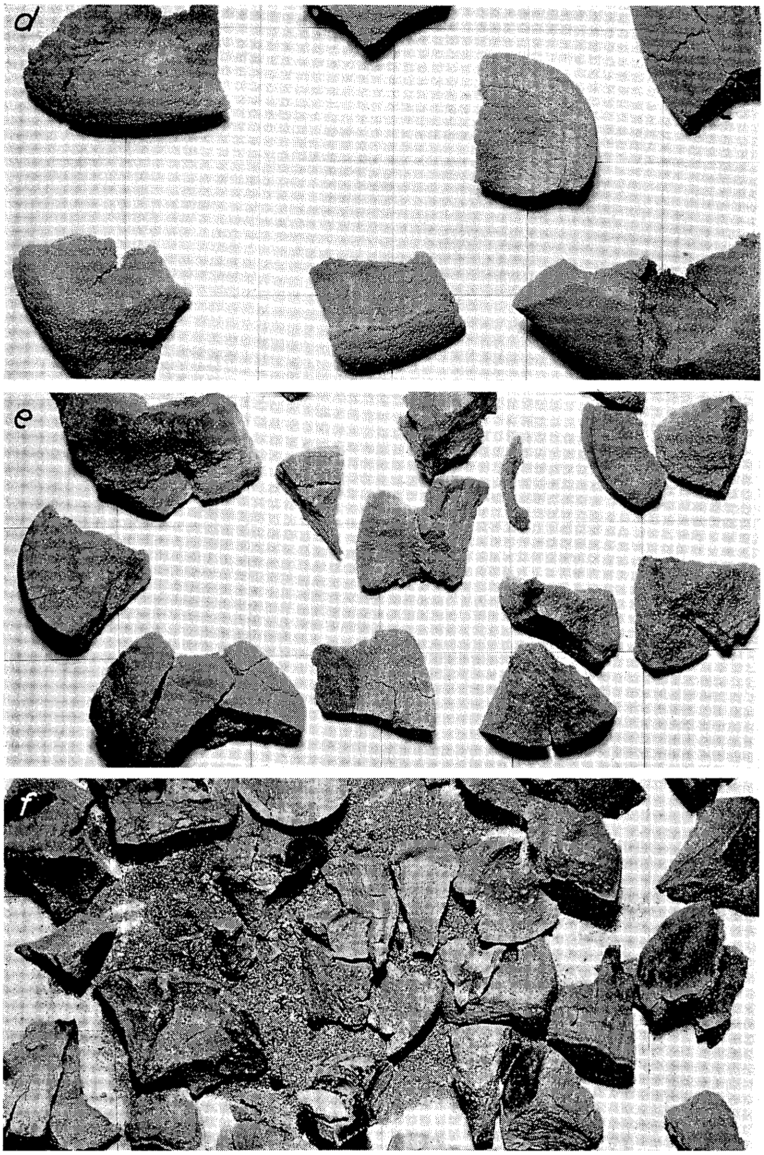


Fig. 48 (*con't.*).—COKES FROM FRANKLIN COUNTY, ILLINOIS, NO. 6 COAL, MADE IN SIX-INCH RETORT (3-INCH GRID).

d. 70% No. 6 coal, 10% Pocahontas, 20% coke breeze, wetted; 10.8°C. per minute to 1100°C. (coke No. 10).
e. 73% No. 6 coal, 20% Pocahontas, 7% dust, wetted; 5.8°C. per minute to 1100°C. (coke No. 9).
f. 95% No. 6 coal, 5% tar; 10.2°C. per minute to 1025°C. (coke No. 26).

Fig. 49.—Cokes from Franklin and Randolph counties, Illinois, No. 6 coal, Made in Six-inch Retort (3-inch grid).

a. 90% Franklin Co. No. 6 coal, 10% tar; 9.6° C. per minute to 1050° C. (coke No. 27).

b. 95% Franklin Co. No. 6 coal, 5% petroleum oil; 8.7° C. per minute to 1000° C. (coke No. 25).

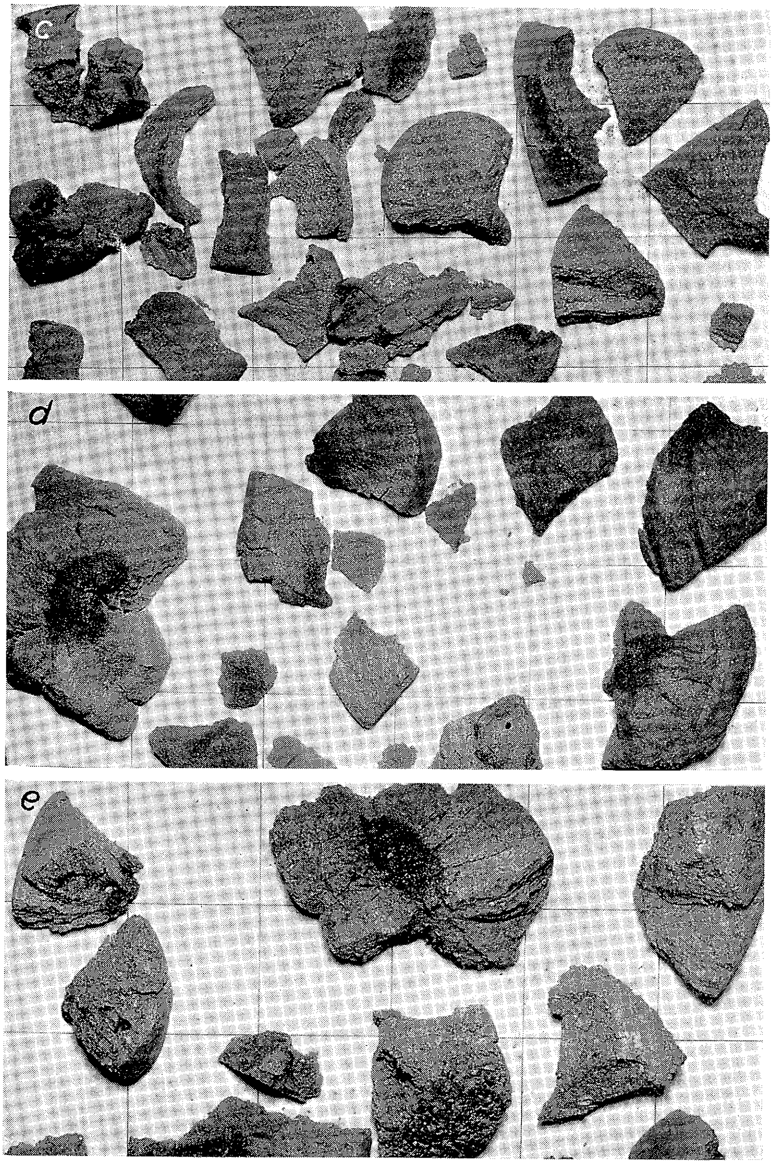


Fig. 49 (*con't.*).—Cokes from Franklin and Randolph counties, Illinois, No. 6 coal, Made in Six-inch Retort (3-inch grid).

c. 100% Randolph Co. No. 6 coal, 20-mesh; 11.1° C. per minute to 1100° C. (coke No. 15).
d. 100% Randolph Co. No. 6 coal, 20-mesh; 10.1° C. per minute to 800° C. (coke No. 23).
e. 100% Randolph Co. No. 6 coal, 4-mesh; 4.8° C. per minute to 1000° C. (coke No. 17).

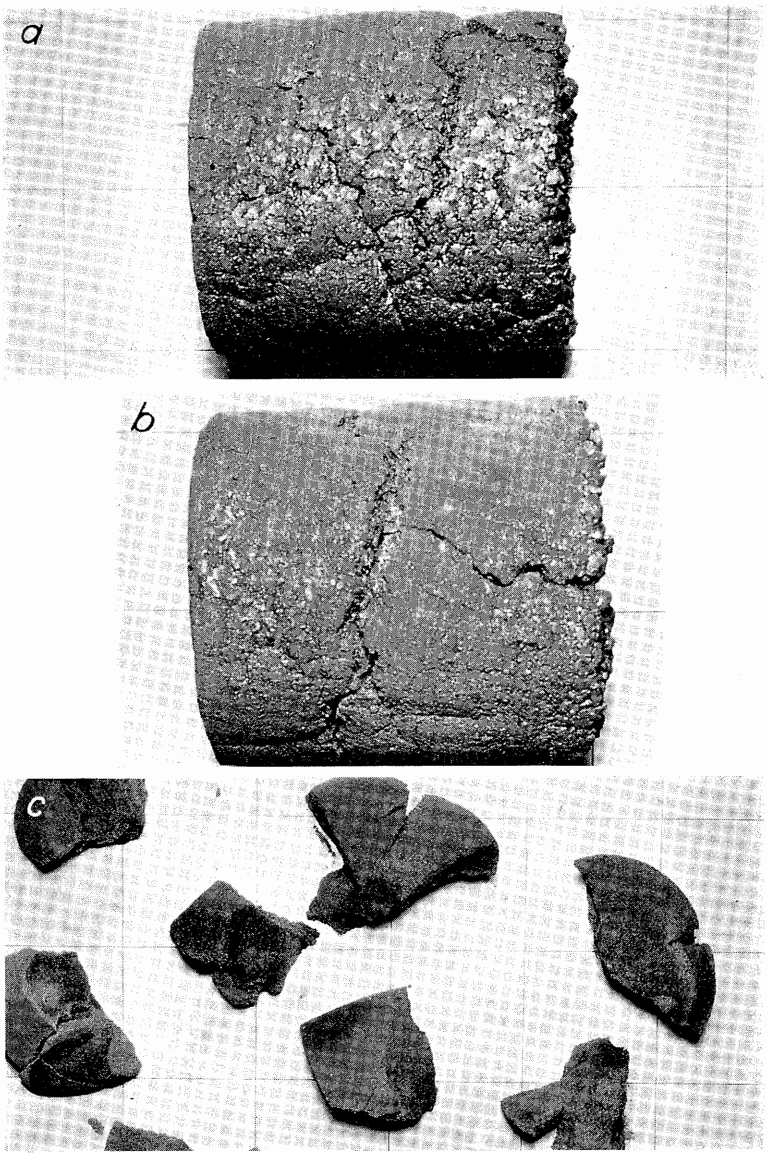


Fig. 50.—Cokes from Randolph County, Illinois, No. 6 coal, Made in Six-inch Retort (3-inch grid).

a. 100% No. 6 coal, 4-mesh; 8.1° C. per minute to 800° C. (coke No. 22).
b. 100% No. 6 coal, 4-mesh; 6.0° C. per minute to 1100° C. (coke No. 14).
c. 80% No. 6 coal, 20% Pocahontas; 10.2° C. per minute to 975° C. (coke No. 18).

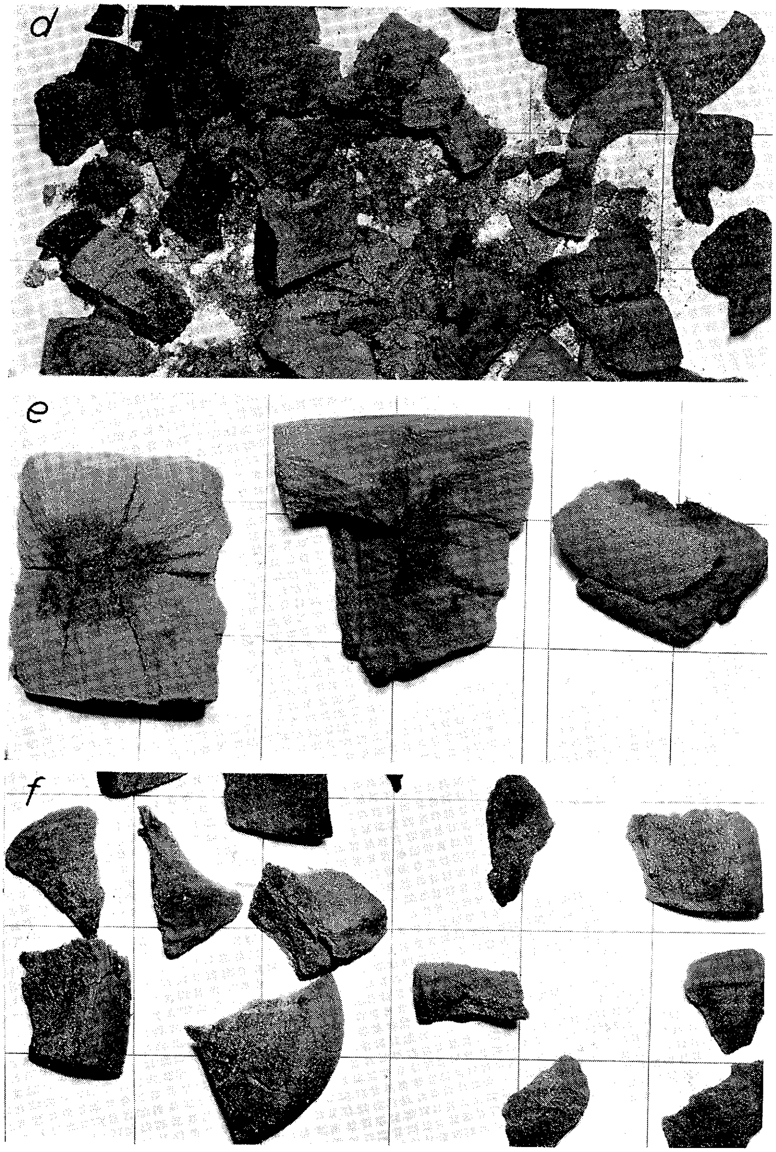


Fig. 50 (*con't.*).—COKES FROM RANDOLPH COUNTY, ILLINOIS, NO. 6 COAL, MADE IN SIX-INCH RETORT (3-INCH GRID).

d. 80% No. 6 coal, 20% Pocahontas; 8.4° C. per minute to 1000° C. (coke No. 21).
e. 80% No. 6 coal, 20% coke breeze; 7.9° C. per minute to 1000° C. (coke No. 19).
f. 75% No. 6 coal, 20% Pocahontas, 5% dust; 13.7° C. per minute to 1025° C. (coke No. 20).

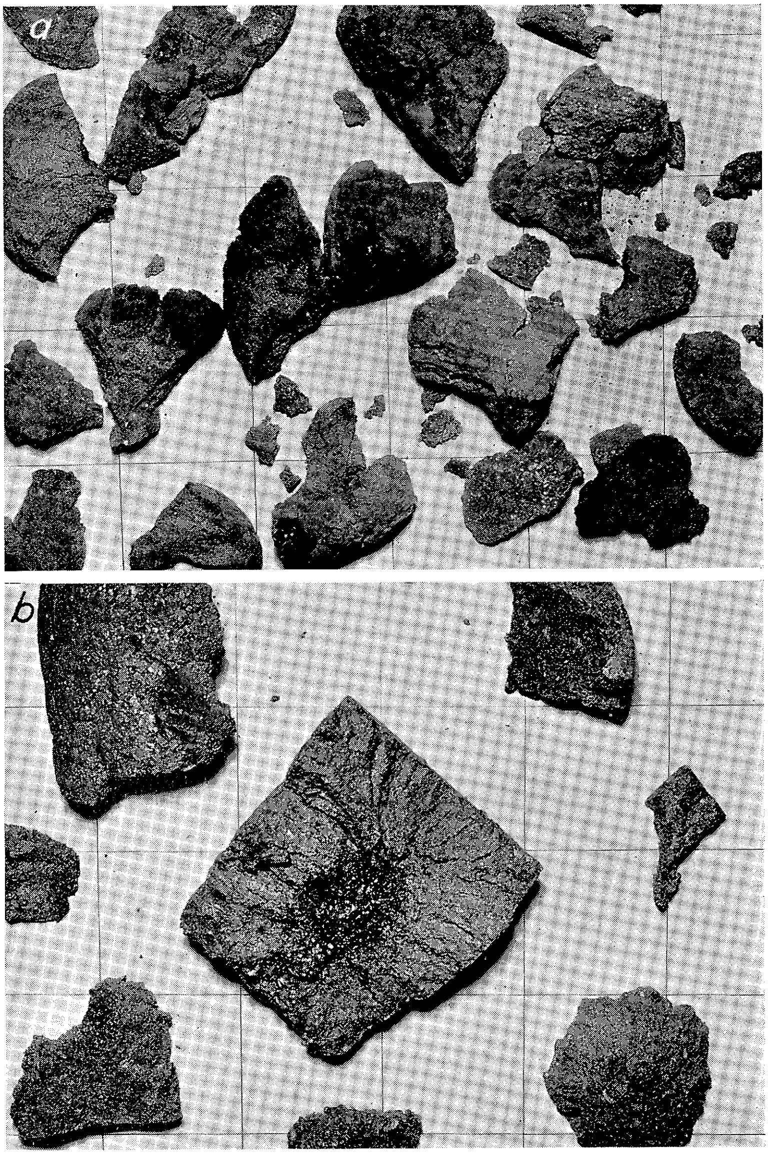


Fig. 51.—Cokes from Randolph County, Illinois No. 6 coal, Made in Six-inch Retort (3-inch grid).

a. 95% No. 6 coal, 20-mesh, 5% tar; 11.3°C. per minute to 1025°C. (coke No. 28).
b. 95% No. 6 coal, 4-mesh, 5% tar; 8.9°C. per minute to 1025°C. (coke No. 29).

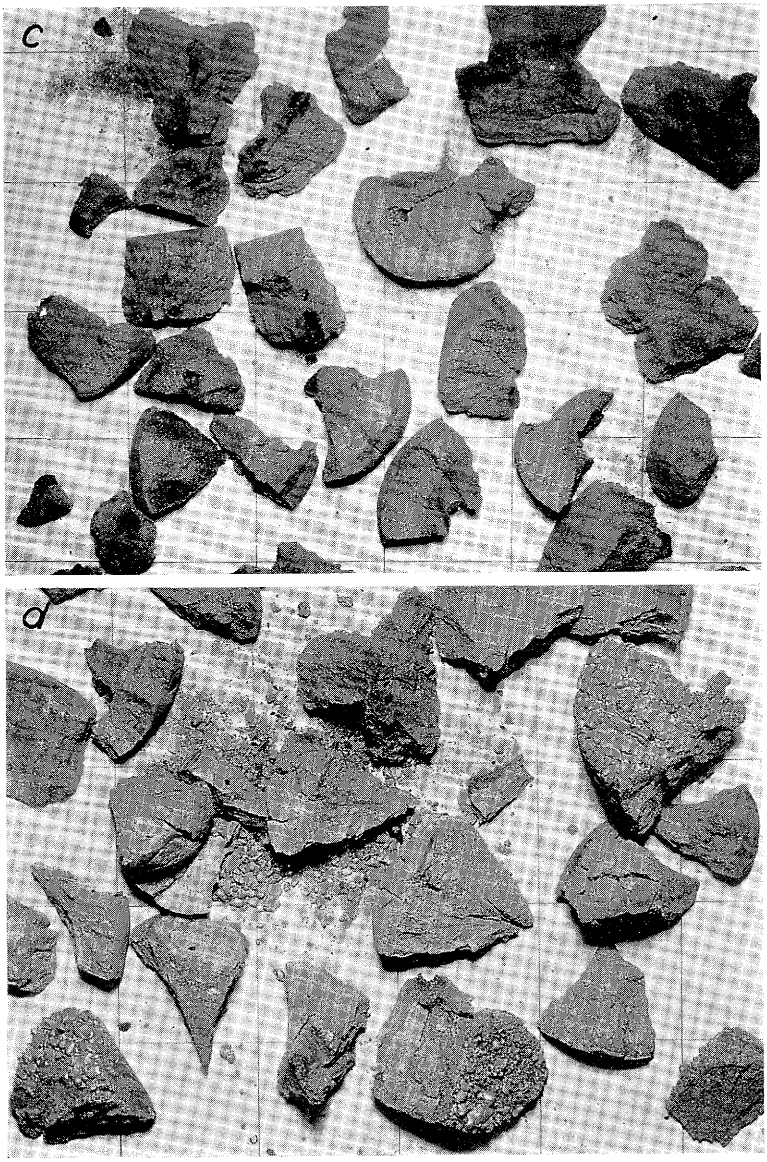


Fig. 51 (*con't.*).—Cokes from Randolph County, Illinois, No. 6 coal, Made in Six-inch Retort (3-inch grid).

c. 100% No. 6 coal, wetted: 11.0° C. per minute to 1000° C. (coke No. 16).
d. 100% No. 6 coal cleaned by floating on liquid of 1.5 sp. gr.; 6.1° C. per minute to 1000° C. (coke No. 40).

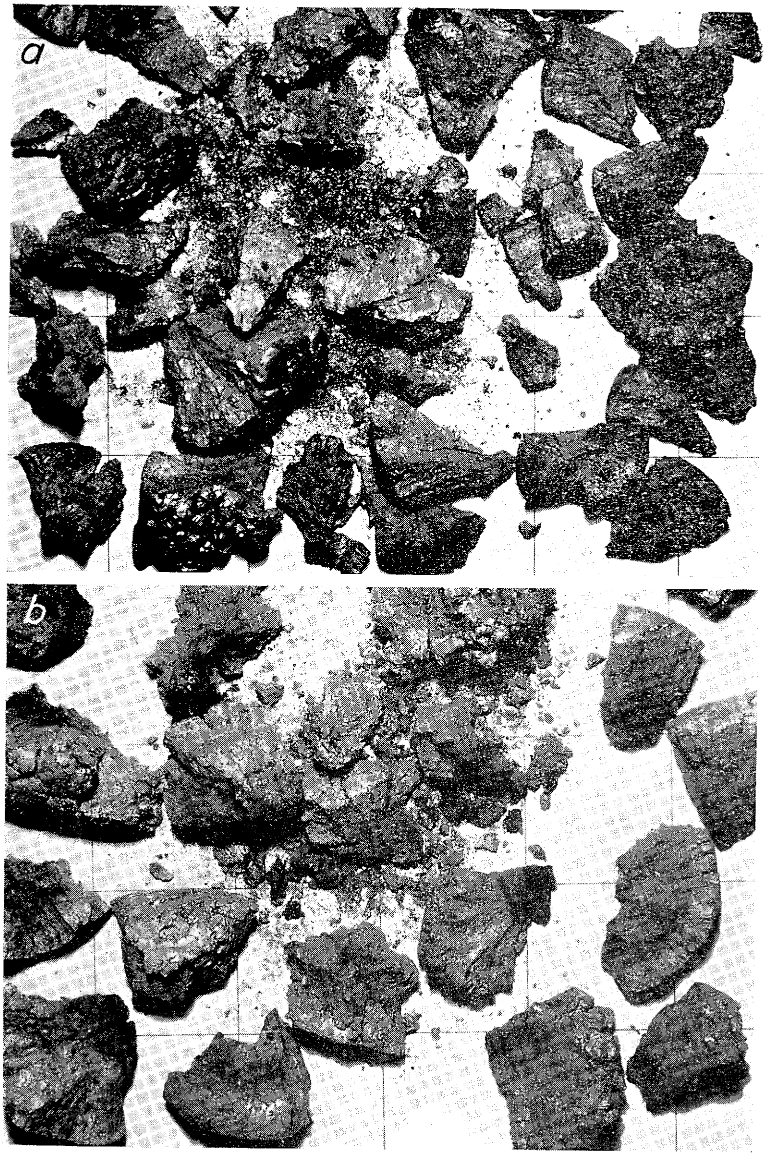


Fig. 52.—Cokes from Saline County, Illinois, No. 5 coal, Made in Six-inch Retort (3-inch grid).

a. 100% No. 5 coal; 12.7° C. per minute to 1000° C. (coke No. 35).
b. 100% No. 5 coal; 8.4° C. per minute to 850° C. (coke No. 36).

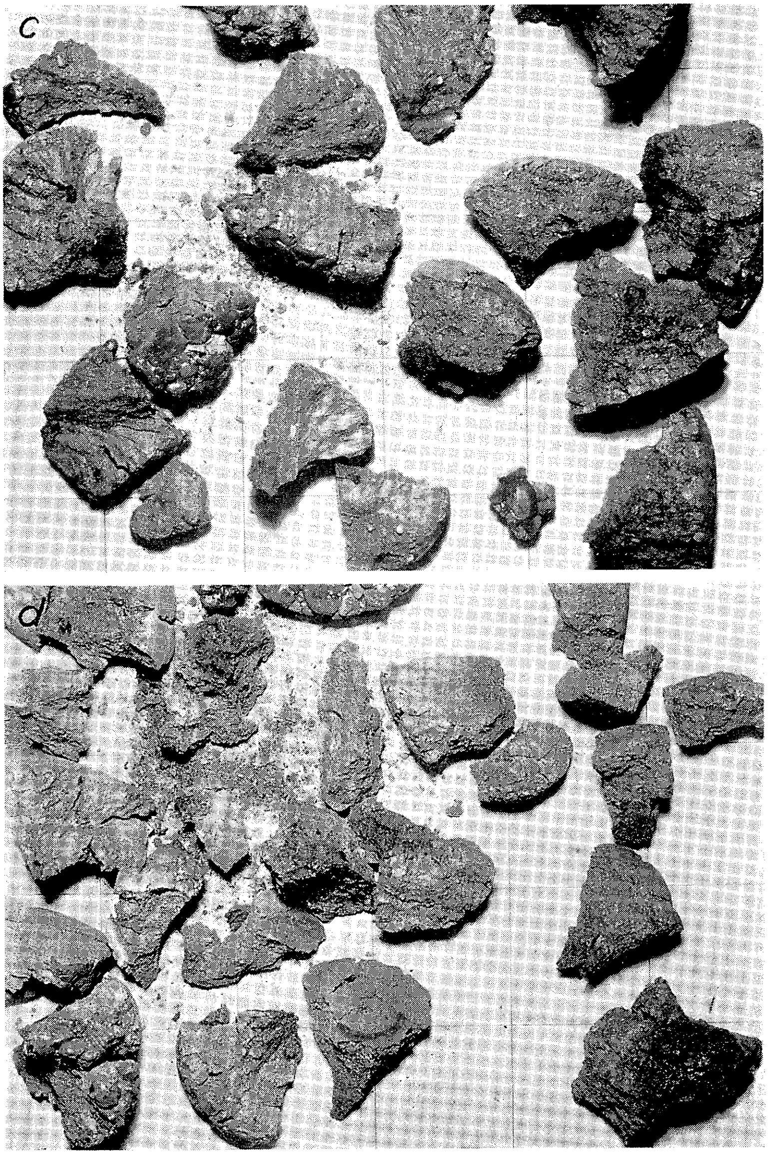


Fig. 52 (*con't.*).—COKES FROM SALINE COUNTY, ILLINOIS, NO. 5 COAL, MADE IN SIX-INCH RETORT (3-INCH GRID).

c. 85% No. 5 coal, 15% Pocahontas; 5.4° C. per minute to 1000° C. (coke No. 37).

d. 85% No. 5 coal, 15% Pocahontas; 9.2° C. per minute to 850° C. (coke No. 38).

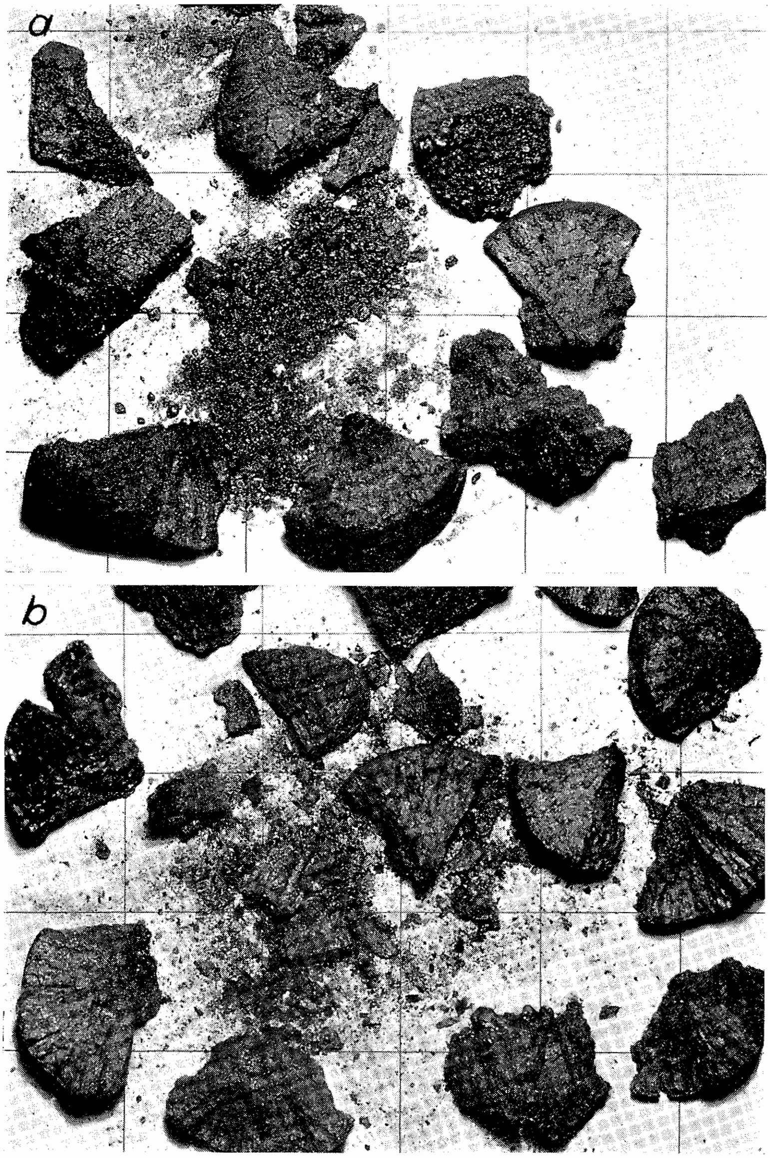


Fig. 53.—Cokes from Various Coals, Made in Six-inch Retort (3-inch grid).

a. 100% Franklin Co. Illinois No. 6 coal, minus ⅜-inch size, washed; 7.2° C. per minute to 1000° C. (coke No. 31).

b. 100% Perry Co. Illinois No. 6 coal, minus ⅜-inch size, washed; 5.6° C. per minute to 825° C. (coke No. 33).

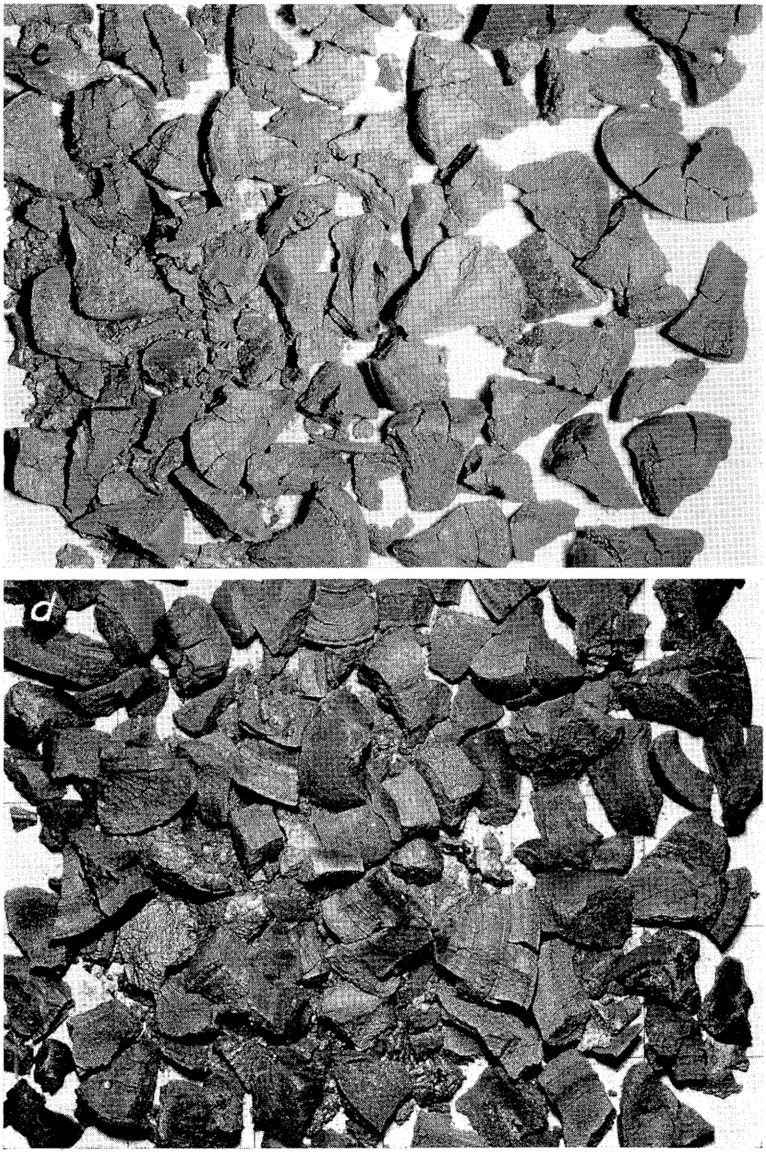


Fig. 53 (*con't.*).—COKES FROM VARIOUS COALS, MADE IN SIX-INCH RETORT (3-INCH GRID).

c. 100% Woodford Co. Illinois No. 2 coal, 20-mesh; 11.1°C. per minute to 1100°C. (coke No. 2).

d. 100% West Virginia Pocahontas coal, 40-mesh; 9.3°C. per minute to 1100°C. (coke No. 24).

BOX TESTS IN KNOWLES SOLE-FLUE OVENS

While making other tests in connection with the operation of sole-flue ovens, advantage was taken of the opportunity to make basket tests on prepared screenings from Franklin County No. 6 coal. In order to show the effect of different methods of treating or preparing coal screenings the baskets used to hold the coal samples were made of angle iron welded to form a frame 18 inches long, 12 inches wide and 10 inches high, covered with expanded metal lath (fig. 54). The filled baskets were placed on the floor of the oven in a space cleared of the regular oven charge and the oven charge then filled in around them. The experimental charges were thus coked under conditions as close as possible to those to which the normal oven charge was subjected. Description of the coals, analyses of the coals and cokes, and results of shatter tests on the coke are presented in Table 27. Photographs of specimens of the cokes obtained are shown in figures 55 and 56. Photographs of cross-sections prepared according to the method of Rose are shown natural size in figure 57 and at a magnification of seven times in figures 58, 59 and 60.

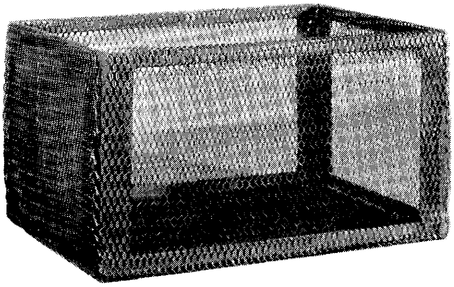


Fig. 54.—Metal Basket Used in Coking Tests.
(Size 18 inches long, 12 inches wide, and 10 inches high)

Results of box tests in Knowles ovens.—In comparing the coke made in the baskets with that made at the same time outside of the baskets as the usual oven charge, the apparent effect of including the coal in the basket must be taken into consideration. The cokes made in the baskets had a much greater tendency to form fingery pieces, as is evident from inspection of the photographs of coke 71 (figure 55b), made in a basket, and coke 72 (figure 55c) made in the oven as part of the regular charge from the same coal. These fingers radiated from the central part of the top of the charge to the lower corners and edges. This tendency of the coke made in the baskets to acquire a fingery structure as a result of its isolation from the rest of the oven charge must be taken into consideration in judging the experimental results on coals varying in character from the usual charge.

TABLE 27.—ANALYSES OF COALS USED IN BASKET TESTS IN KNOWLES OVENS, WITH ANALYSES OF COKES, COKE BREEZE, AND RESULTS OF SHATTER TESTS

Serial No.	Description of samples from No. 6 bed Franklin County, Ill.	Analyses of coal samples							Analyses of cokes remaining on ½″ screen after shatter test				
		Dry basis, per cent							Dry basis, per cent				
		Ash	Volatile matter	Fixed carbon	Total sulfur	Pyritic sulfur	Organic sulfur	Calorific value B.t.u./lb.	Ash	Volatile matter	Fixed carbon	Sulfur	Calorific value B.t.u./lb.
F-66	2×¾ inch screenings cleaned at 1.5 sp. gr. and crushed to ¼ inch......	9.8	36.1	54.1	1.14	0.58	0.55	13026	14.4	2.1	83.5	0.88	12264
F-67	¾×0 inch raw screenings...........	10.2	34.4	55.4	1.90	1.29	0.60	12966	13.9	3.7	82.2	2.10	12217
F-70	¾×0 inch raw screenings washed at 1.5 sp. gr..........................	6.8	35.8	57.4	1.32	0.69	0.61	13480	10.7	2.2	87.1	1.28	12906
F-68	⅜ inch × 10-mesh screenings washed at 1.5 sp. gr.....................	6.2	35.7	58.1	1.30	0.69	0.60	13555	9.7	1.6	88.7	1.06	11881
F-69	10-mesh screenings, air dried.........	12.3	31.7	56.0	1.83	1.29	0.52	12619	17.7	1.8	80.5	1.99	12727
F-71	Regular oven feed coked in basket....	12.1	34.0	53.9	1.43	0.96	0.46	12647	16.5	3.0	80.5	1.49	12055
F-72	Regular oven coke.................	12.1	34.0	53.9	1.43	0.96	0.46	12647	16.1	3.0	80.9	1.36	12103

TABLE 27.—CONCLUDED

Serial No.	Description of samples from No. 6 bed Franklin County, Ill.	Analyses of cokes passing ½″ screen after shatter test					Size classification of coke after shatter test, per cent of original + 1 inch coke			Figure No.
		Dry basis, per cent								
		Ash	Volatile matter	Fixed Carbon	Sulfur	Calorific Value B.t.u./lb.	Remaining on ½ inch screen	Remaining on 1 inch screen "Stability"	Passing through ½ inch screen "Friability"	
F-66	2×¾ inch screenings cleaned at 1.5 sp. gr. and crushed to ¼ inch	14.8	2.4	82.8	0.96	12258	95.6	87.7	4.4	55a
F-67	¾×0 inch raw screenings	22.7	6.9	70.4	2.42	11105	87.4	68.3	12.6	56a
F-70	¾×0 inch raw screenings washed at 1.5 sp. gr.	14.3	2.3	83.4	1.90	12307	91.6	82.0	8.4	56b
F-68	⅜ inch × 10-mesh screenings washed at 1.5 sp. gr.	11.6	2.4	86.0	1.30	12614	94.6	81.7	5.4	56c
F-69	10-mesh screenings, air dried	19.5	2.7	77.8	2.48	11662	93.2	90.6	6.8	56d
F-71	Regular oven feed coked in basket	24.8	3.4	71.8	2.23	10892	92.0	80.3	8.0	55b
F-72	Regular oven coke	20.4	6.6	73.0	1.44	11539	93.1	82.1	6.9	55c

The coal used in the box tests was very similar except for size to the coal regularly charged into the oven at the time the tests were made. It was taken from one of the mines which supplied coal to the coke ovens.

The influence of cleaning on the strength of the coke is shown by the results of the shatter tests on cokes 67 and 70, Table 27. The amount of coke remaining on the one-inch screen at the end of the test increased from 68 per cent to 82 per cent while the amount passing the one-half inch screen dropped from 12.6 per cent to 8.4 per cent. That much of this minus half-inch material is composed of shale or other high-ash material which is only loosely held by the coke mass is indicated both by the decreased amount in the coke from the washed coal and also by the lower ash content of the minus-half-inch material from the washed coal and by the smaller difference between the ash content of the larger and smaller coke from the washed coal. Comparison of the magnified Rose section of cokes 67 and 70 (figs. 59a and 59b) shows the effect of the reduction of mineral matter by coal cleaning on the amount of non-fusing material in the coke. Many non-cellular dark areas may be noticed in figure 59a which are absent in 59b. These dark, non-cellular areas represent pieces of shale or high-ash material. The washed coal showed a greater tendency to produce a fingery coke (coke 70, fig. 56b) than did the raw coal (coke 67, fig. 56a), probably because of the greater amount of moisture in the washed coal. These effects of reducing the mineral matter content of coal on the resulting coke are also evident from a comparison of cokes 68 and 71 which, while not from the same coal, are made from material of similar size. The coke from the washed coal, No. 68, had 90 per cent remaining on the one-inch screen compared with 80 per cent for the coke from the raw coal, No. 71. The ash content of the minus one-half inch material was 8 per cent higher than the ash content of the plus half-inch material in coke from raw coal and only 1.8 per cent higher in coke from washed coal.

The coke made from the minus 10-mesh material (No. 69, fig. 56d) is typical of coke made from fine coal containing much inert matter, such as fusain. It has good resistance to breakage by shattering but is soft and friable. Its friability was more apparent on handling than is indicated by the amount of minus half-inch material formed on dropping. Its blocky structure is striking when compared with the other cokes in this series. The large amount of fine fusain in this coke shows up in the Rose sections of it in figures 57 and 60b as small dark non-cellular fragments.

The coke No. 66 from the 2 × ¾-inch washed screenings which had been crushed in a hammer mill so that the entire sample passed a screen with ¼-inch openings gave a small celled, rather uniform coke with a tendency toward cross fracturing, figures 55a, 57 and 58a.

The coke from the ⅜-inch by 10-mesh coal (No. 68) showed a greater tendency to fracture than did the other cokes, probably because of the removal of the minus 10-mesh material which contained a high proportion of non-shrinking fusain, figures 56c, 57 and 60a.

GENERAL CONCLUSIONS ON THE USE OF ILLINOIS COALS FOR MAKING COKE

Because of the relatively high volatile matter content of Illinois coals, cokes made from them tend to form slender fingery pieces. The formation of the fingery pieces is caused by shrinkage due to loss of volatile matter after the plastic coal mass has become solid. The formation of shrinkage cracks can be greatly decreased by adding low volatile matter content, non-fusing carbonaceous material to the coal to be coked. However, Illinois coals have too low caking powers to hold this non-fusing material firmly. Consequently cokes from such mixtures are soft and abradable even though they may resist shattering. Because of the tendency of ash forming material to concentrate in the naturally formed fine sizes of coal commonly used for coke making and because this ash forming material decreases the value of the coke, it is usually desirable that the cokes be made from properly cleaned coals. The coking process should be so regulated that the coke already formed in the oven is not overcoked before the remainder of the charge is coked. Illinois coals should be coked as soon as possible after removal from the mine face. The coal should not be ground too fine and the oven charge should have as great a bulk density as possible. The coal charge should not be too wet when charged into the oven. With reasonable care, coke which is satisfactory for domestic fuel, many industrial and some metallurgical uses, can be made from most of the coals found in Illinois. Exceptions would be those coals which have lost their caking properties due to weathering, either because they are now, or have been at some time past, too close to the surface.

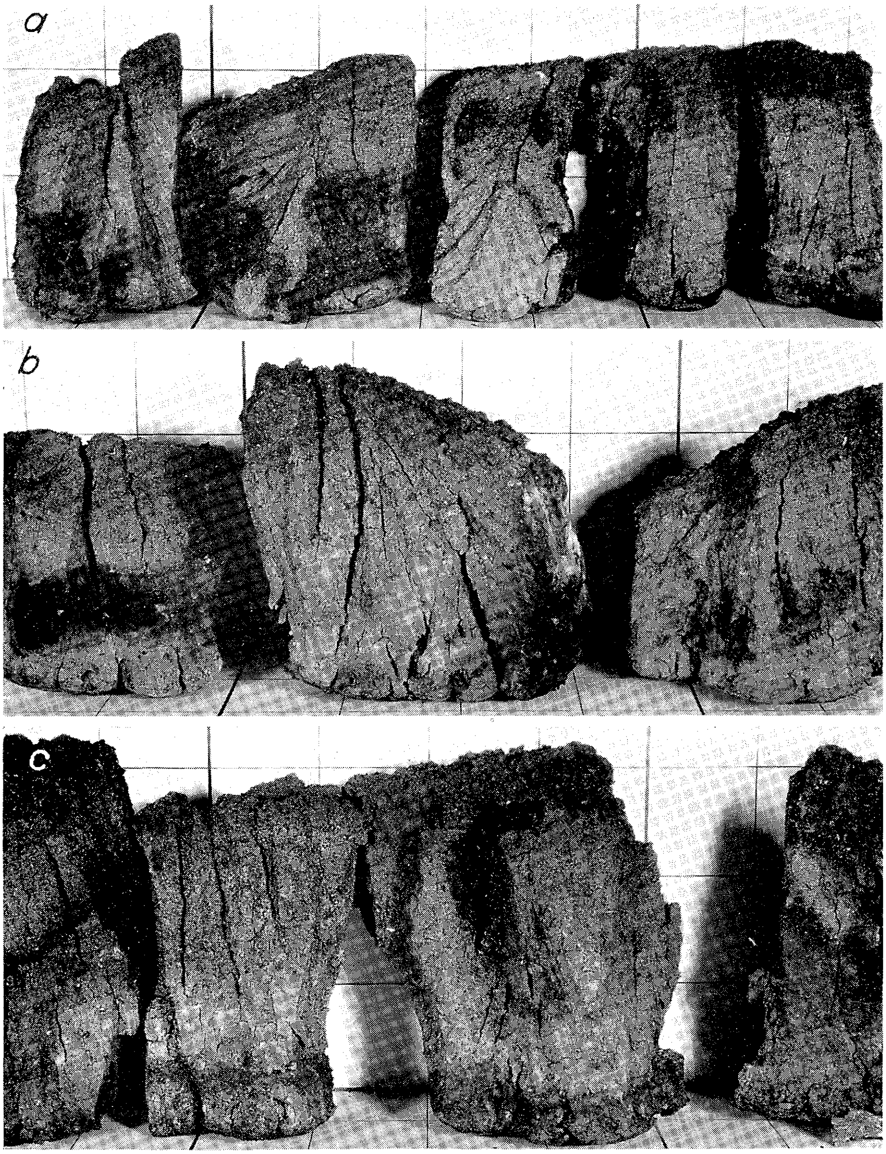


Fig. 55.—Basket Test Coke and Plant-run Coke (3-inch grid).

a. Coke No. 66, made in basket test from 2 × ¾ inch washed slack, crushed.
b. Coke No. 71, made in basket test from regular plant feed, minus 5/16-inch screenings.
c. Plant-run coke No. 72.

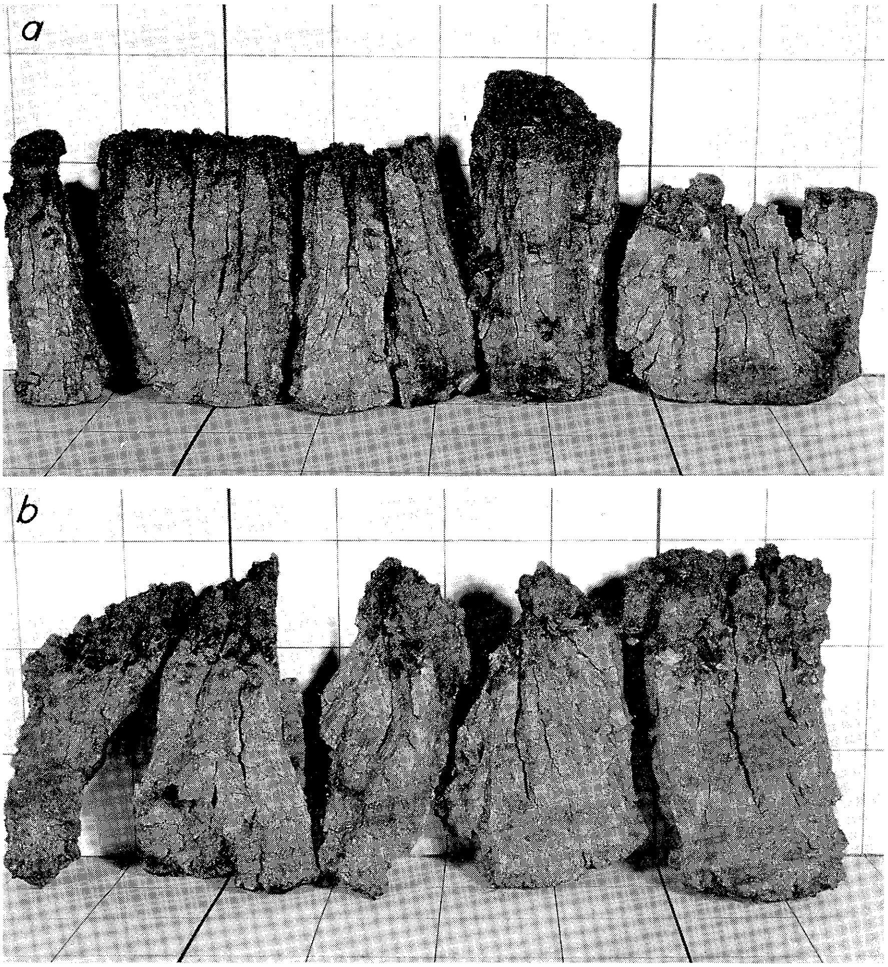


Fig. 56.—COKES MADE IN BASKET TESTS (3-INCH GRID).

a. Coke No. 67 made from ¾ × 0 inch raw screenings.
b. Coke No. 70 made from ¾ × 0 inch washed screenings.

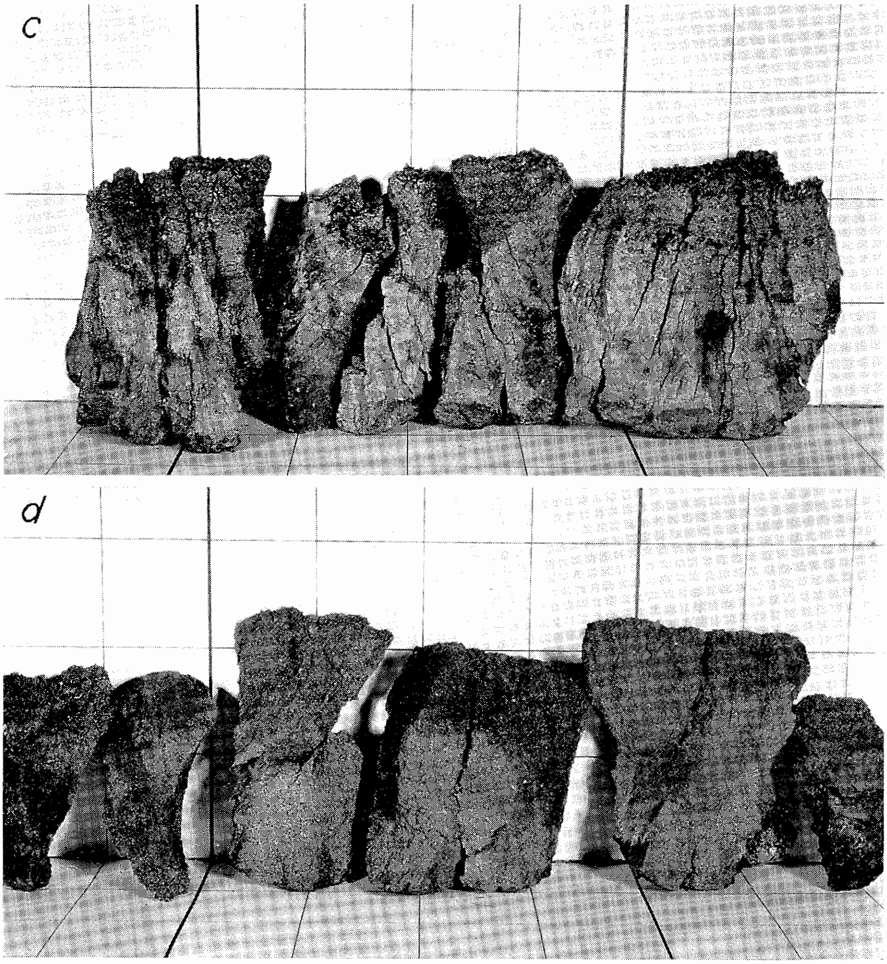


Fig. 56 (*con't.*).—Cokes Made in Basket Tests (3-inch grid).

c. Coke No. 68 made from ⅜ inch × 10-mesh washed screenings.
d. Coke No. 69 made from dried minus 10-mesh screenings.

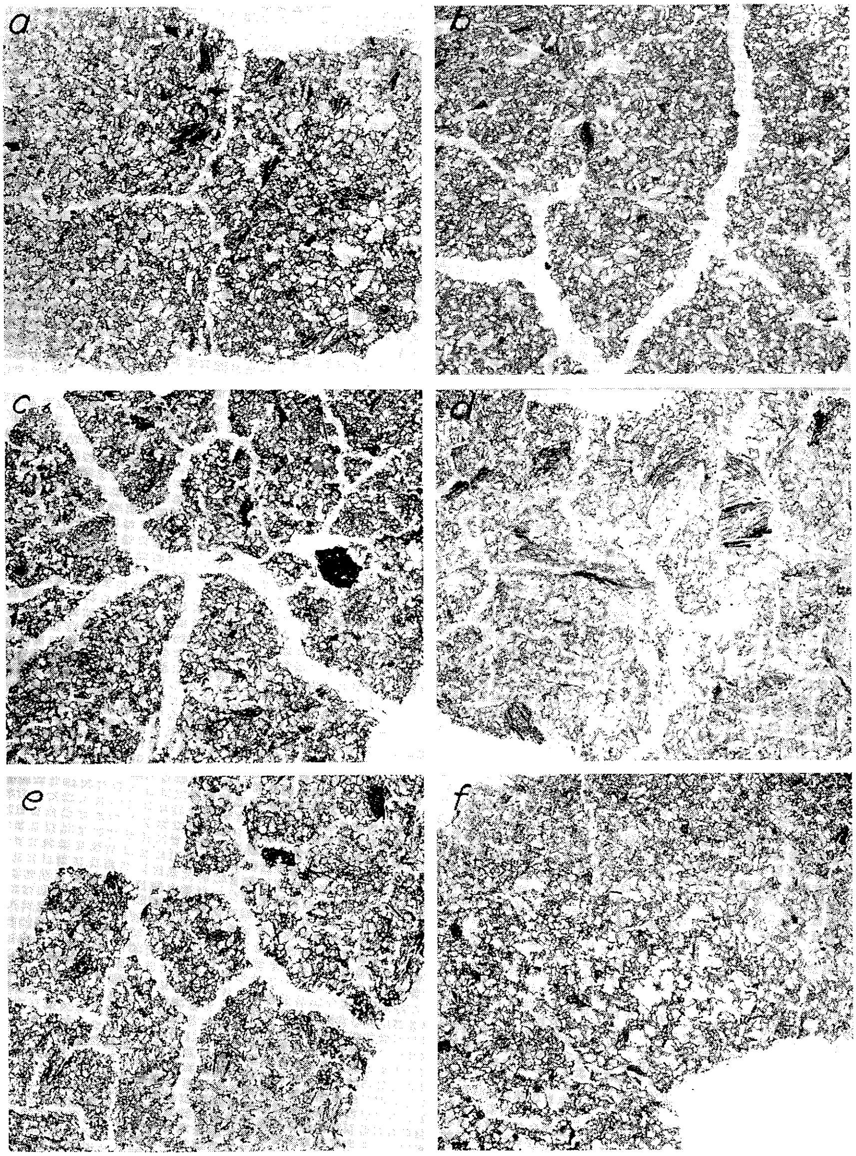


Fig. 57.—Rose Cross-sections, Natural size, of Cokes Made in Basket Tests.

a. Coke No. 66 (fig. 55a), from 2 × ¾ inch washed slack; crushed.
b. Coke No. 71 (fig. 55b), from minus 5/16-inch screenings.
c. Coke No. 67 (fig. 56a), from ¾ × 0 inch raw screenings.
d. Coke No. 70 (fig. 56b), from ¾ × 0 inch washed screenings.
e. Coke No. 68 (fig. 56c), from ⅜ inch × 10-mesh washed screenings.
f. Coke No. 69 (fig. 56d) from dried minus 10-mesh screenings.

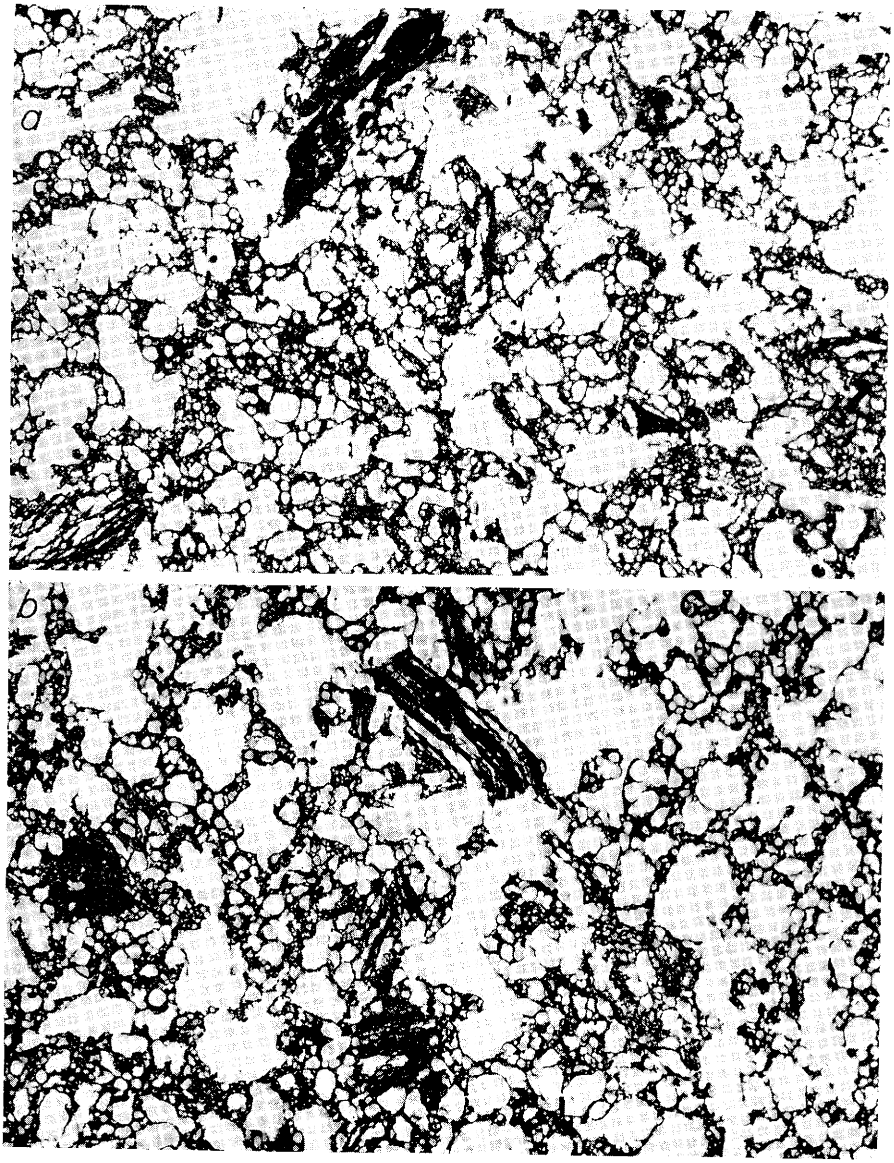


Fig. 58.—Rose Cross-sections at a Magnification of Seven Diameters.

a. Coke No. 66 (figs. 55a and 57a), from 2 × ¾ inch washed slack, crushed.
b. Coke No. 71 (figs. 55b and 57b), from minus 5/16-inch screenings.

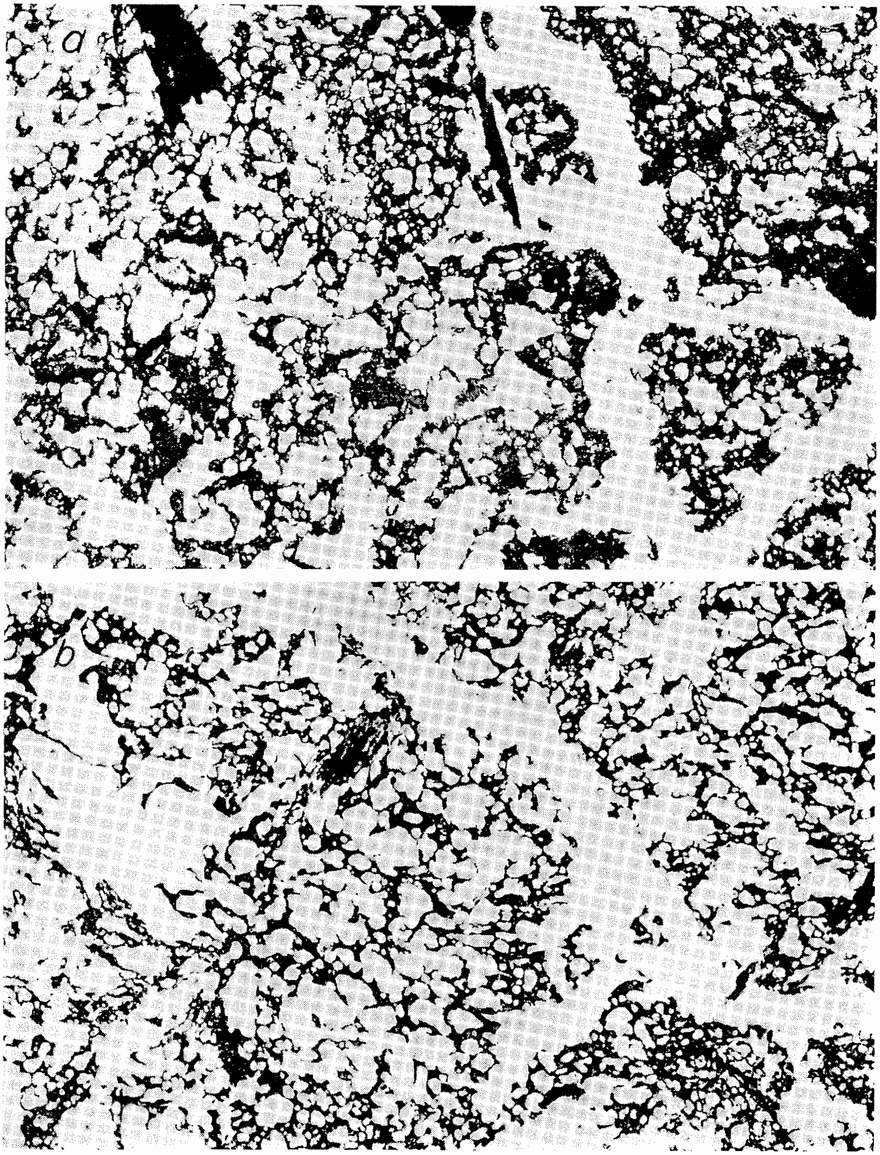


Fig. 59.—ROSE CROSS-SECTIONS AT A MAGNIFICATION OF SEVEN DIAMETERS.

a. Coke No. 67 (figs. 56a and 57c), from ¾ × 0 inch raw screenings.
b. Coke No. 70 (figs. 56b and 57d), from ¾ × 0 washed screenings.

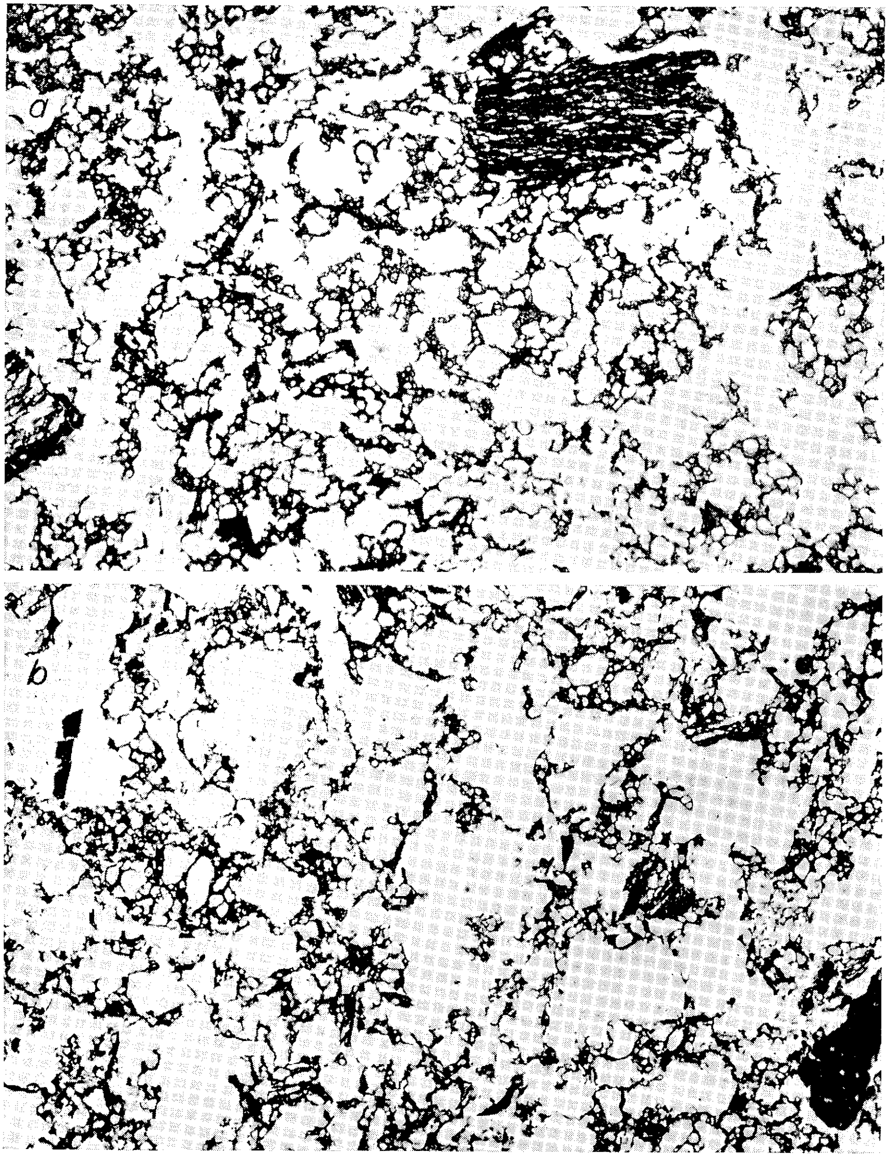


Fig. 60.—Rose Cross-sections at a Magnification of Seven Diameters.

a. Coke No. 68 (figs. 56c and 57e), from ⅜ × 10-mesh washed screenings.
b. Coke No. 69 (figs. 56d and 57f), from dried minus 10-mesh screenings.

CHAPTER XVI—AN INDUSTRIAL COKING PLANT UTILIZING ILLINOIS COAL

Coke is produced commercially at the present time from Illinois coal only at an installation of 26 Knowles type, sole-flue ovens at West Frankfort, Franklin County. Because the Knowles coke oven is a relatively new type of carbonization equipment of which descriptions are not commonly available and because it is now being used with apparent success in the production of a domestic smokeless fuel (coke), a description of its construction and operation seems pertinent.

For satisfactory operation of the slot-type coke oven the coal charge must have certain definite properties. The charge must not swell excessively or develop any great pressure against the side walls or serious damage to the oven results; the coke must shrink away from the walls in the final stages of coking so that the charge may be pushed from the oven without damaging the walls and, finally, the coke must have a coherent structure so that it will move as a single block under the influence of the pusher ram, and not crush and jam against the walls. This last requirement offers the most serious difficulty encountered when coking coals of the Eastern Interior basin in ovens of this type. Because of their high volatile matter and moisture content, these coals have a tendency to form slender, fingery pieces during the final devolatilization and shrinkage period. These fingery pieces tend to cause the charge of coke to crumble in front of the pusher ram and to jam in the oven, making it necessary to maintain very close control over the coking operation. The difficulties are further aggravated by the fact that these coals rapidly lose their coking properties upon storage.

It was to overcome these difficulties that the Knowles oven in which the coal is heated from below in a wide, flat layer was applied to Illinois coals (fig. 9). The charge is more easily removed from the oven even if it should crush in front of the pusher ram. A coke more suited to domestic use in which the coke grades from a low-volatile-matter content, high-temperature coke at the bottom to a high-volatile-matter content, low-temperature coke at the top can be made if desired and pushed from the oven. The present installation consists of three batteries, one of ten and two of eight ovens each,[1] shown in panoramic view in figure 61.

---

[1] Anon., Knowles oven widens market for No. 5 Illinois screenings by turning fines into domestic coke: Coal Age vol. 39, pp. 421-423, Nov. 1934.

McBride, R. S., Processing coal in Knowles coke oven. Chem. and Met. vol. 42, No. 6, pp. 300-3, June 1935.

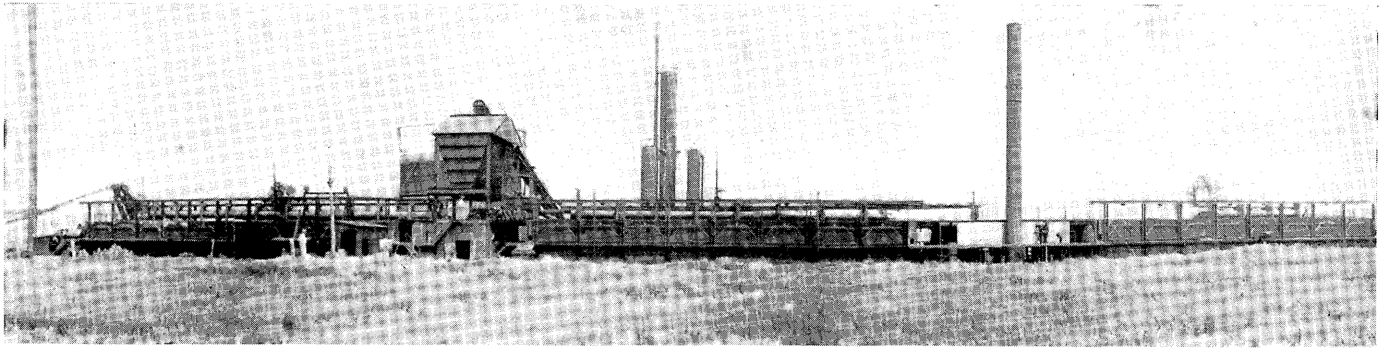


Fig. 61.—Knowles Ovens at West Frankfort, Illinois, in 1936.

These ovens are built in the form of an arched rectangular chamber, approximately 30 feet long, 7½ feet wide, and 4 feet high at the top of the arch and 2 feet high at the sides. Heat is applied through flues which form the floor or sole of the oven. These flues are connected to regenerators built into the oven foundations. Figure 62 shows construction details of the ovens in cross and horizontal section. Figure 63 shows a view of the coke side of the ovens, while figure 64 shows another view of the coke side with coke quenching car in position as an oven is being discharged. The ovens are charged with 5 tons of "No. 5 carbon" (5/16-inch) screenings making a layer 10 to 12 inches thick. Coking time has varied from 8½ to 10½ hours. Temperatures in the flues are about 1360°C. (2480°F.) with the floor temperature at the completion of a coking cycle at about 1100°C. (2000°F.)

Historically, this type of oven is derived from the recuperative beehive oven in which the products of combustion of the volatile part of the coal are brought under the oven floor in a flue system.[2]

The sole-flue oven has been developed both as a recuperative[3] and as a regenerative[4] by-product recovery oven. The ovens which have found commercial use have been regenerative, although some non-heat recovery ovens of this type are in use in the petroleum industry for the production of a dense, low volatile-matter content petroleum coke from petroleum residues.[5]

OPERATION OF KNOWLES SOLE-FLUE OVENS

In the investigation of the operation of these ovens the temperature conditions in the body of the charge of coal being coked were considered especially important in view of differences in the manner of applying the heat to the coal in these ovens compared to other coal carbonizing equipment. The importance of the rate of coking to the production of good coke from high-volatile content, poorly caking coals is mentioned in the coke literature.[6] Temperature conditions inside of coal charges being coked in slot-type by-product ovens and in vertical continuous gas retorts have been studied by various investigators and are available in the liter-

---

[2] Bone, W. A., Coal and its scientific uses: Longmans, London, 1910, p. 302. Sole-flue beehive oven patented by Breckon and Dixon, 1858. Rectangular ovens mentioned.

Fulton, J., Coke: International Textbook Co., Scranton, Pa., 1906, p. 173-7. The Ramsay Patent Beehive Coke Oven.

Ibid., p. 177-8, Daube's Economic Down-Draft Coke Oven.

Moss, R. S., Improved Heminway Process: Mines and Minerals, vol. 21, No. 6, pp. 412-4, April 1901.

[3] Zwillinger, B. U. S. Patent 1656617, Jan. 17, 1928. Zwillinger, B. U. S. Patent 1428621, Sept. 12, 1922. von Bauer and Zwillinger, B., U. S. Patent 1478410, Dec. 25, 1923.

[4] Knowles, A. S., and McIntire, C. V., U. S. Patent 1635280, July 12, 1927. Knowles, A. S., U. S. Patent 1745996, Feb. 4, 1930.

[5] Campbell, O. F., Petroleum coke makes ideal household fuel when its properties are understood: Oil and Gas Journ. vol. 33, No. 45, pp. 68-69, March 28, 1935.

Knowles, A. S., U. S. Patent 1717884. June 18, 1929.

Ziegenhain, W. T., Tidal refining company making high quality coke in Knowles unit: Oil and Gas Jour. vol. 30, No. 17, pp. 16-17, 100, Sept. 10, 1931.

[6] Ovitz, F. K., Coking of Illinois coals: U. S. Bureau of Mines Bull. 138, p. 17, 1917.

Roberts, A., Art of coking coal: U. S. Patent 1352696, Sept. 14, 1920.

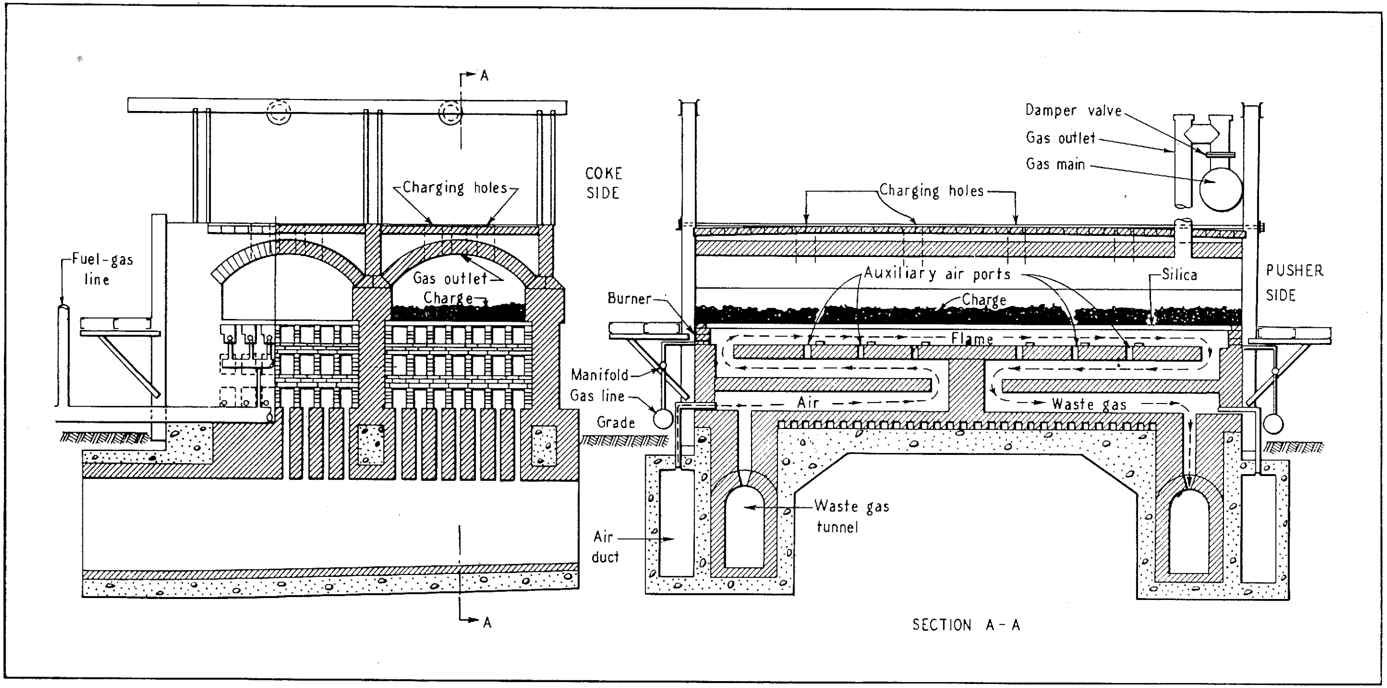


Fig. 62.—Construction Details of Knowles Sole-flue Oven.
Scale omitted. *(From "Coke from Illinois Coals," by G. Thiessen. Ind. and Eng. Chem. vol. 29, May, 1937.)*

ature for purposes of comparison.[7] Through the courtesy of the Radiant Fuel Corporation, owners of the Knowles ovens at West Frankfort, it was possible to study the temperature conditions inside a charge being coked in this type of oven.

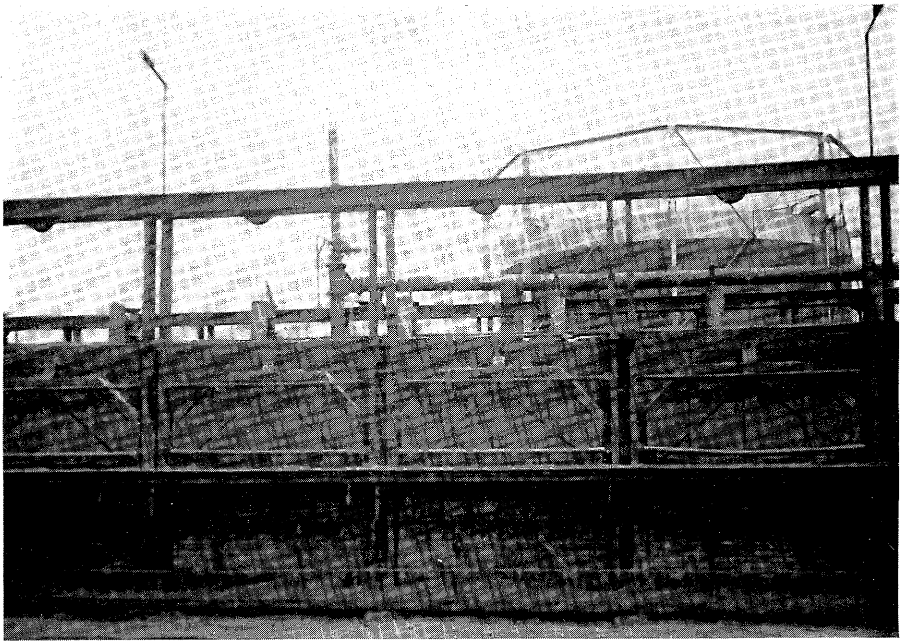


Fig. 63.—COKE SIDE OF KNOWLES SOLE-FLUE OVENS
(From "Coke from Illinois Coal," by G. Thiessen, Ind. and Eng. Chem. vol. 29, May 1937.)

OBJECTIVES OF THE INVESTIGATIONS

The objectives of these investigations were to determine the temperature conditions inside a charge of Illinois coal being coked in the sole-flue type of coke oven, including a determination of the rate of travel of the plastic zone, the temperature gradients, and the final temperature achieved in the charge at selected points; to measure regenerator and flue temperatures; to determine the volatile-matter gradient from bottom to top in the finished coke; and to determine average gas composition.

---

[7] Cooper, G. S., The by-product coking industry and its relation to the manufacture of iron and steel. Appendix II. Temperature Measurements in a Koppers Oven: Jour. Iron and Steel Inst. vol. 90, pt. II, pp. 17-47, 1914.

Hilgenstock, R. W., Über Destillations-Cokerei: Journal für Gas Beleuchtung 45, pp. 617-21, 1902.

McBride, R. S., and Selvig, W. A., Coking of Illinois coal in Koppers type oven: U. S. Bureau of Standards Tech. Paper 137, 1919.

Simmersbach, O., Berg- und Hüttenmannische Rundschau 157, 1913.

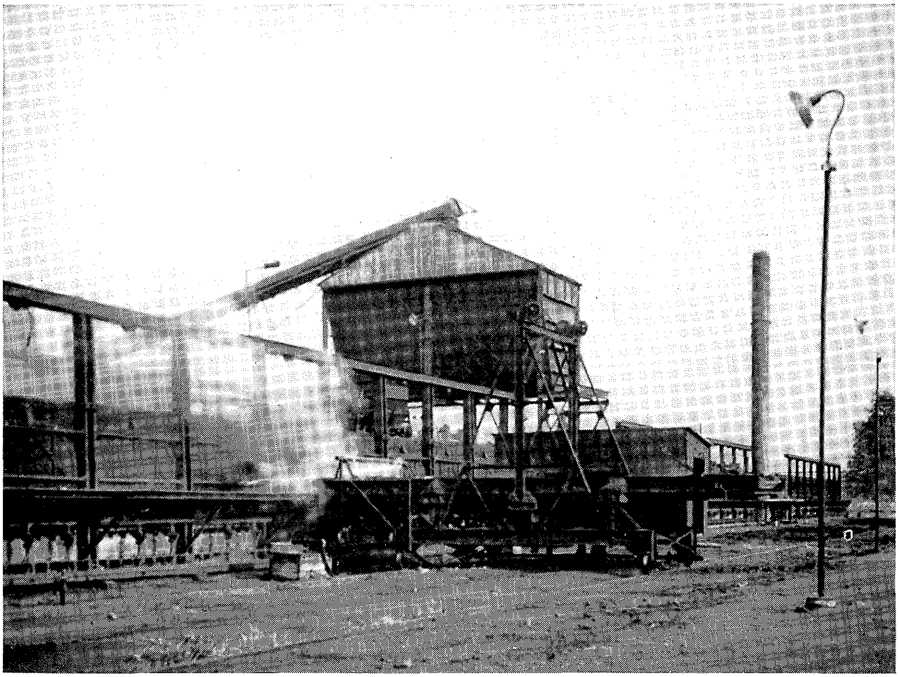


Fig. 64.—Coke Side of the Knowles Ovens with the Quenching Car in Position. *(From "Coke from Illinois Coal," by G. Thiessen. Ind. and Eng. Chem. vol. 29, May, 1937.)*

TEMPERATURE MEASURING EQUIPMENT

Temperatures in the charge being coked in the oven selected for the investigation were measured at the center line of the oven 6½ feet from the door, directly below the outer charging hole line at seven points spaced above the floor by means of the following arrangement: Seven chromel-alumel thermocouples, made of number 22-gauge wire, 12 feet long, were insulated with 2-hole sillimanite insulators and enclosed in ¼-inch steel pipes welded shut at the end for protection. These protecting pipes were welded to steel strips in such a way that they lay flat on the floor of the oven as they passed under the door and were then directed upwards toward the center line of the oven where they were supported in a vertical sequence by a supporting strip with the lowest couple on the oven floor and the others successively 1, 3, 5, 7, 9, and 10½ inches above the floor (fig. 65), the last about at the surface of the charge. The pipes containing the wires extended about a foot in a horizontal line beyond the supporting strip in order to minimize possible errors caused by the conduction of heat along the pipes. In figure 65 the location of the couples with respect to the ovens is as follows: The end of the pipes extend 8 inches beyond the edge of the 4-foot walkway extending along the ends of the ovens, pass over the walkway and under the 10-inch thick door, and then gradually curve upward and toward the center line of the oven.

Oven number nine was selected for this investigation because its operation was typical of those in the battery; it was not an end oven and yet was near to the end of the battery for convenient location of test equipment out of the way of operating equipment. The temperatures measured by the couples located 1, 3, 5, and 7 inches from the floor were automatically recorded by a Brown Instrument Co. four-point recording potentiometer pyrometer, whereas the temperatures measured by the remaining couples were manually read every five minutes with a Leeds and Northrup portable potentiometer. Temperatures in the regenerator outlet pass and in the stack were measured with chromel-alumel couples and the portable potentiometer. Flue temperatures were measured with a Leeds and Northrup optical pyrometer.

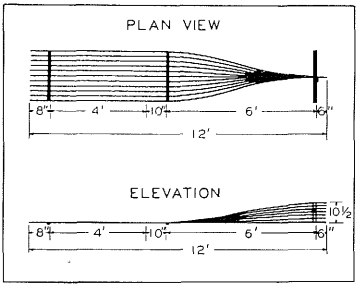


Fig. 65.—THERMOCOUPLE WELL ASSEMBLY.

(From "Coke from Illinois Coal," by G. Thiessen. Ind. and Eng. Chem. vol. 29, May, 1937.)

In addition to the thermocouple protection tubes, four similar open-ended tubes were included in the assembly for measurement of gas pressure in the charge.

The pyrometers and other test equipment were mounted on a framework at the end of the battery and leads were so placed that connections could be made quickly after the thermocouples were in place.

PROCEDURE FOR TEMPERATURE MEASUREMENTS

The thermocouple assembly was lifted into position on the floor of the empty oven, from which the charge had just been pushed in such a position as to clear the base of the charge-leveller shoes. The oven was then charged and levelled with care so as not to disturb the thermocouples. The doors of the oven were then closed and luted and the connections to the measuring instruments made. The automatically recording potentiometer connected to the couples at 1, 3, 5, and 7 inches from the floor was started, recording temperatures about ten minutes after the oven was charged. Manual readings of the temperatures indicated by the thermocouples on the floor, at the top of the charge and 9 inches from the floor were not started until almost 45 minutes after the oven was charged, due to activities of all observers in connection with the leveling and door closing procedure. Thereafter these temperatures were read every five minutes. Upon return to the laboratory, temperatures were read off the recorder chart at corresponding five-minute intervals and combined with the manual readings in the form of a table and were plotted.

SAMPLING AND TESTING OF COAL, COKE, AND GAS

Samples of the coal being coked at the time the tests were made were taken from the belt conveyor carrying the coal from the railroad cars to the storage hopper on the oven. Laboratory samples were quartered from the bulk samples, the remainder being used in one of the box coking tests conducted. A sample of coke representing the oven charge made during the temperature measurements was taken from the coke conveyor belt. Individual pieces representing the coke at the end of the thermocouples were carefully removed as the coke quenching car was being emptied. The piece of coke directly at the ends of the thermocouples was recovered in one large piece. The analyses were made in the laboratories of the geochemical section of the Illinois State Geological Survey using A.S.T.M. standard methods.

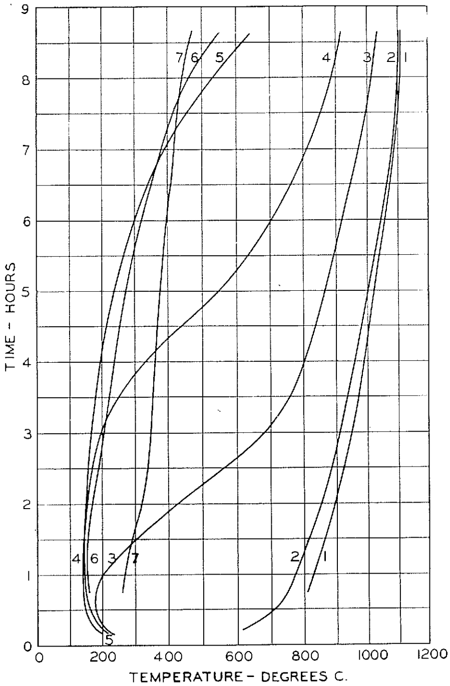


Fig. 66.—Temperature-Time Relations at Various Points in the Sole-flue Oven Charge. *(From "Coke from Illinois Coal," by G. Thiessen. Ind. and Eng. Chem. vol. 29, May, 1937.)*

1. On oven floor.
2. One inch from oven floor.
3. Three inches from oven floor.
4. Five inches from oven floor.
5. Seven inches from oven floor.
6. Nine inches from oven floor.
7. Ten and a half inches from oven floor.

Gas samples were taken over a period of 40 minutes, from the main gas line before and after sulfur removal, by displacement of water in 5-gallon glass bottles. Gas analyses were made both with the Orsat-type gas analysis equipment and with the Podbielniak gas fractionating equipment.

RESULTS OF TEMPERATURE MEASUREMENTS

Temperatures in charge.—Table 28 presents the temperatures measured inside the charge at five-minute intervals from a time shortly after the coal was introduced into the oven to a time just a few minutes before the coke was pushed out of the oven. The temperatures at the seven places in the charge are shown plotted against time in figure 66 as smooth curves through average values. The actual curves for points 1 and 2 and the later part of the curve for point 3 show a regular wave form caused by temperature changes in the combustion flues brought about by the half-hourly reversal of regenerators and burners. Being closest to the flues, the thermocouple resting on the oven floor very definitely showed this effect. In figure 67 the isothermal lines for each 50°C. temperature interval have been plotted and in figure 68 the isochronal lines for the same data have been plotted for hourly intervals, to assist in making the significance of the results apparent.

TABLE 28.—TEMPERATURES IN CHARGE OF ILLINOIS COAL COKED IN OVEN 9, KNOWLES OVEN INSTALLATION OF RADIANT FUEL CORPORATION, WEST FRANKFORT, ILLINOIS
Noon to 8:45 P. M., Oct. 10, 1935

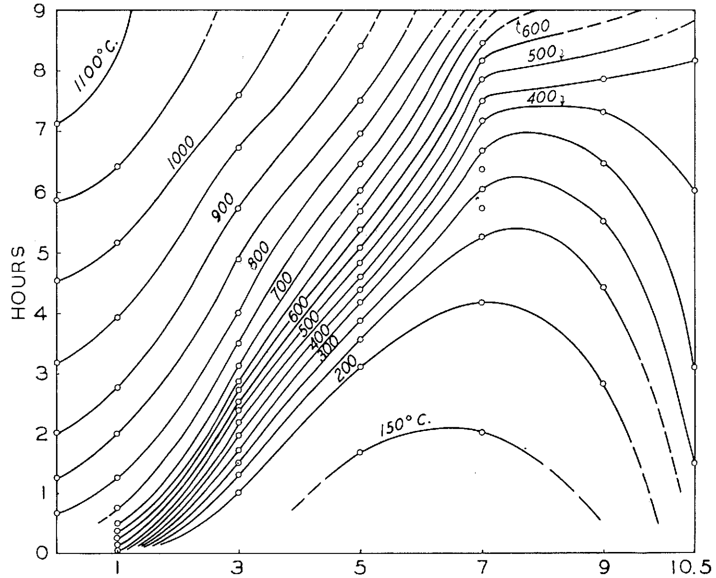
Time	Thermocouple[a] 1	2	3	4	5	6	7
12:12	. . .°C	575°C	240°C	200°C	220°C	. . .°C	. . .°C
12:15	. . .	600	220	187	207	. . .	. . .
12:20	. . .	642	198	170	190	. . .	. . .
12:25	. . .	770	186	159	175	. . .	. . .
12:30	. . .	700	180	152	167	. . .	. . .
12:35	. . .	717	178	149	161	. . .	. . .
12:40	. . .	735	178	146	157	. . .	. . .
12:45	(12:44)807	750	180	145	155	(12:44)162	(12:44)264
12:50	. . .	762	182	144	153	. . .	. . .
12:55	(12:56)827	770	190	144	151	(12:56)157	(12:56)266
1:00	(1:01)827	774	198	144	150	(1:01)157	(1:01)264
1:05	832	785	210	144	148	157	268
1:10	839	793	223	144	148	162	268
1:15	849	797	240	144	150	156	283
1:20	856	805	256	147	147	157	286
1:25	866	813	276	147	147	162	292
1:30	876	822	297	146	146	159	300
1:35	886	827	315	147	147	164	307
1:40	891	833	340	150	148	169	310
1:45	896	837	360	151	149	167	314
1:50	891	838	380	152	149	172	314
1:55	891	840	396	154	150	172	319
2:00	891	841	411	156	150	174	324
2:05	891	843	427	157	151	177	326
2:10	894	846	446	159	152	179	326
2:15	898	850	466	161	153	184	331
2:20	904	858	491	163	155	184	334
2:25	911	867	514	165	156	187	336
2:30	928	876	543	167	157	192	338
2:35	934	886	568	170	158	194	341
2:40	938	895	593	174	160	194	341
2:45	941	900	620	177	161	197	341
2:50	944	905	645	182	162	202	343
2:55	944	907	664	187	164	202	343
3:00	. . .	910	680	193	166	. . .	. . .
3:05	944	912	695	200	167	209	350
3:10	946	915	708	207	171	212	353
3:15	951	918	717	216	174	214	350
3:20	956	922	729	225	176	217	353
3:25	964	927	738	235	178	219	355
3:30	974	935	750	247	180	224	358
3:35	979	942	760	257	182	227	355
3:40	987	950	770	270	184	229	360
3:45	987	952	780	280	186	232	360
3:50	987	954	788	292	190	236	362
3:55	987	953	793	308	192	239	364
4:00	984	953	800	323	195	244	367
4:05	976	955	805	339	198	242	362
4:10	979	956	808	355	200	242	362
4:15	984	961	812	373	204	246	364
4:20	992	966	817	388	207	246	364
4:25	997	971	824	408	211	249	367
4:30	1002	977	830	430	214	254	367
4:35	1002	980	835	450	215	259	369

TABLE 28.—CONCLUDED

Time	Thermocouple[a] 1	2	3	4	5	6	7
4:40	1002°C	980°C	840°C	467°C	220°C	261°C	369°C
4:45	(4:46)1002	980	845	485	222	(4:46) 266	(4:46)374
4:50	1000	979	848	503	225	268	376
4:55	1000	980	852	522	228	273	376
5:00	1005	983	855	540	234	276	376
5:05	1007	990	860	554	240	280	379
5:10	1020	993	865	567	245	286	379
5:15	1023	1003	868	580	250	288	381
5:20	1034	1007	875	595	254	292	384
5:25	1034	1016	882	609	258	295	386
5:30	1044	1020	890	624	263	300	388
5:35	1046	1023	895	637	267	305	391
5:40	1046	1025	898	650	273	310	391
5:45	1044	1024	903	663	278	312	393
5:50	1041	1022	908	675	285	317	396
5:55	....	1017	910	686	291	322	...
6:00	1041	1016	913	696	297	324	400
6:05	1041	1020	915	708	303	331	403
6:10	1038	1022	917	719	310	334	405
6:15	1052	1027	920	728	317	338	405
6:20	1062	1033	924	736	324	343	407
6:25	1065	1041	928	745	330	348	410
6:30	1075	1046	935	753	336	353	410
6:35	1078	1052	942	762	343	358	412
6:40	(6:42)1083	1060	948	770	351	(6:42)364	(6:42)417
6:45	1086	1065	955	778	359	369	419
6:50	(6:51)1094	1070	962	787	367	374	419
6:55	(6:56)1102	1076	968	796	375	379	419
7:00	1099	1082	974	805	385	381	419
7:05	....	1083	979	813	393	...	...
7:10	1096	1082	984	820	400	391	422
7:15	1094	1083	986	827	410	396	424
7:20	(7:21)1099	1085	988	835	422	405	426
7:25	1099	1087	991	842	436	410	426
7:30	1107	1090	995	849	450	417	428
7:35	1110	1092	999	855	462	426	431
7:40	1115	1098	1004	860	475	431	433
7:45	1115	1102	1009	865	486	438	436
7:50	1115	1105	1013	872	497	448	438
7:55	(7:56)1112	1100	1012	875	512	455	440
8:00	1107	1095	1012	878	523	462	443
8:05	1110	1093	1012	882	535	471	445
8:10	1107	1092	1014	886	553	466	450
8:15	1107	1091	1014	892	565	492	452
8:20	1110	1091	1015	896	580	504	455
8:25	1110	1093	1018	902	595	516	457
8:30	1112	1096	1021	907	608	527	462
8:35	1115	1100	1026	910	624	539	466
8:40	1112	1100	1029	914	637	555	471
8:45	....	1100	1032	917	650	...	...

Position of thermocouples

[a] No. 1—On oven floor.
No. 2—1 inch from oven floor.
No. 3—3 inches from oven floor.
No. 4—5 inches from oven floor.
No. 5—7 inches from oven floor.
No. 6—9 inches from oven floor.
No. 7—10½ inches from oven floor, on top of charge.



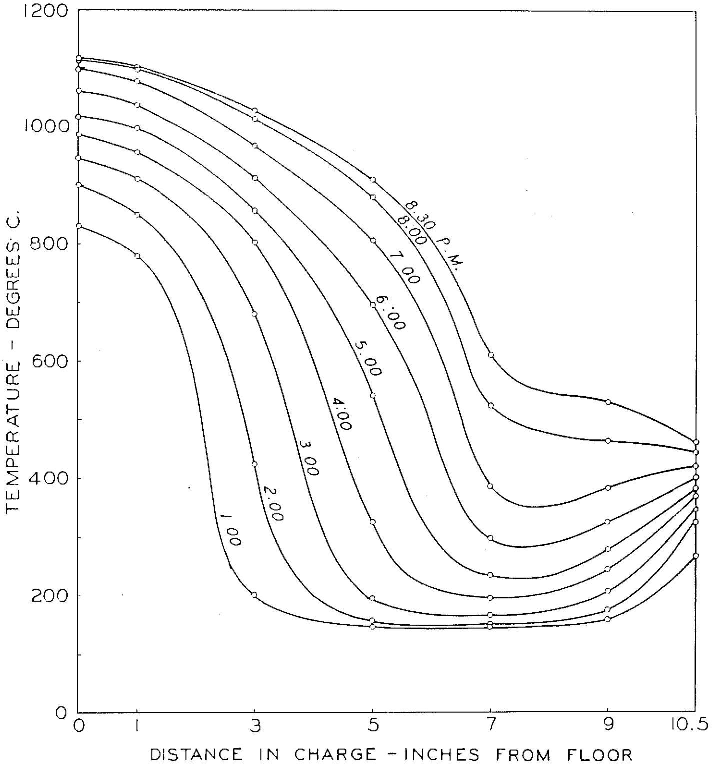


Fig. 67 (*Above*).—Isothermal Relations, and Fig. 68 (*Below*).—Isochronal Curves of Temperature Distribution Through Sole-Flue Oven Charge. (*Both from Ind. and Eng. Chem. vol. 29, May, 1937.*)

Relation of time and temperature.—The isochronal chart (fig. 68) shows graphically the temperature in any part of the charge, at the position of the thermocouple column, at any time during the coking period. It demonstrates the relative effect on the charge of the heat received from the floor and that received from the roof and gases above the charge. The similarity of the curves on the left hand side of the diagram up to the line of 7 inches indicates that the temperature conditions in this part of the charge are similar and that the transfer of heat is from the floor. The upper 3½ inches of the charge received heat mainly from above during the early part of the coking period, then gradually more from below until after eight hours the rise in temperature of the top of the charge due to heat from below completely overshadowed the effect due to heat from above, and it is evident that the charge lost heat to the space above it in the last stages of the coking cycle.

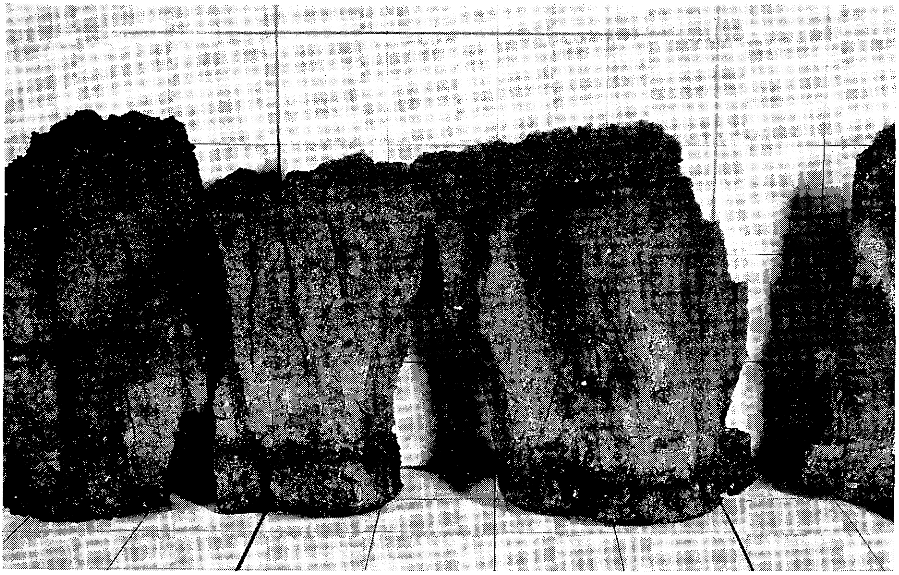


Fig. 69.—Coke Taken from End of the Thermocouple Assembly (3-inch grid).
(From "Coke from Illinois Coal," by G. Thiessen. Ind. and Eng. Chem. vol. 29, May, 1937.)

The heat absorbed from above caused no apparent temperature effects below 3½ inches from the upper thermocouple. The charge may, therefore, conveniently be regarded as consisting of two parts on the basis of its thermal behavior, a lower part coked at relatively high temperature by heat transfer from below, and an upper part coked at relatively low temperature mainly by downward heating. In the lower zone the plastic zone travels regularly upward and the charge is heated relatively rapidly; the upper part of the charge is heated gradually and more or less uniformly but to a lower temperature, first by gases in the oven chamber and eventually by heat transmitted through the lower part of the charge. In the upper zone the heat gradients are lower, the final temperature is lower, and the mass cokes throughout within a relatively short time interval after the plastic temperature of the coal is attained. The gases are probably released throughout the mass rather than at a plastic zone of limited extent as in the lower part of the charge. The difference in the progress of heating in the upper and lower portions of the charge is equally well shown by plotting the determinations on a time-distance (isothermal) chart (fig. 67). This shows clearly the relatively higher temperature of the upper 3 to 4 inches of the charge as compared with the middle part, at least during the first 6 to 7 hours. It also shows that this part of the charge never attains the temperature of the lower part.

The effect of this manner of heating the charge upon the appearance of the coke is demonstrated by the accompanying photographs (fig. 69) of pieces of coke taken from the oven from near the end of the thermocouples after the observations were made.

Travel of the plastic zone.—A charge of coal being coked in a high temperature coke oven consists of (1) a zone of coke extending from the hot wall or floor to (2) a plastic zone which is quite narrow and which separates the coke from (3) the unchanged part of the coal charge. It is common knowledge that there is a large temperature gradient across the plastic zone, on one side of which there is hot coke and on the other side coal at a temperature a little above that of boiling water. The rate of coking is determined by the rate of travel of this plastic zone.

The movement of the plastic zone can be estimated (1) from measurements of gas pressure at various points along the line of travel of the plastic zone, since a high gas pressure occurs at the plastic zone due to its impermeability, (2) from the movement of the point of maximum temperature gradient in the charge, since there is a high heat gradient across the plastic zone due to its low heat conductivity, or (3) from the movement of the temperature zone corresponding to the plastic range of the coal.[8] Our attempt to follow the movement of the plastic zone through pressure measurements was unsuccessful due to plugging by tarry material of the tubes placed in the oven for pressure measurements.

In order to determine the movement of the plastic zone by following the point of maximum heat gradient in the charge, this point was determined graphically using values read from a large scale plot of the isochronal lines (fig. 68). Temperature differences over one-half inch intervals were plotted against the position of the mid-point of the interval to obtain the approximate position of the point of maximum gradient. Temperature differences across smaller intervals in this region were then taken to determine the position of the point more

---

[8] Ryan, W. P., Rate of travel of fusion zone in coke oven: Proc. A.G.A. vol. 7, pp. 861-878, 1925.

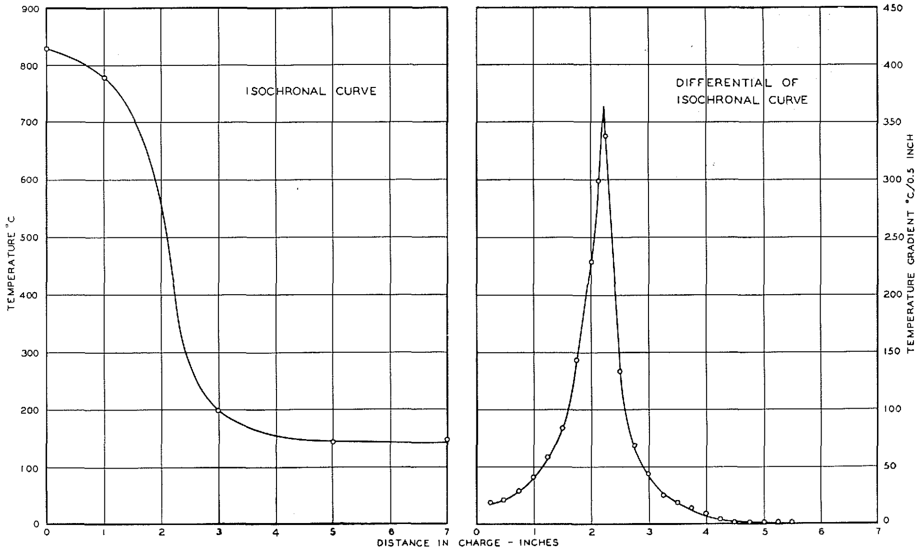


Fig. 70.—Method of Determining Position and Magnitude of Maximum Heat Gradient from an Isochronal Curve.

accurately. Figure 70 shows the curve obtained for the one-hour point, plotted together with the isochronal curve for one hour to illustrate how the values were obtained. The values obtained for the position of the point of maximum gradient, the temperature gradient, and the temperature at that point are represented in Table 29; the movement of the point and the temperature at the point are shown graphically in figure 71, the broken line indicating the movement of the point through the charge. The relatively large heat gradient compared to the temperature and the relatively constant temperature at the point of maximum gradient should be observed.

Table 29.—Location of Points of Maximum Heat Gradient at Hourly Intervals after Charging, and Heat Gradient over Distance One-fourth Inch Each Side of that Point

Time, hours after charging	Location of point of maximum gradient inches from floor[a]	Heat gradient degrees C. per half inch at point of maximum gradient	Temperature at point of maximum gradient
1	2.125	290	480
2	2.875	210	475
3	3.688	170	470
4	4.375	150	490
5	5.250	140	470
6	6.125	140	450
7	6.375	150	545
8	6.500	140	630

[a] to nearest eighth inch

Determination of the position of the plastic zone by determining the position of the temperature interval during which the coal was plastic was accomplished by first determining the plastic temperature range of the coal by means of the Agde-Damm apparatus.[9] It was found that the coal started to soften at 350°C. and that the decomposing plastic mass set to coke at 450°C.

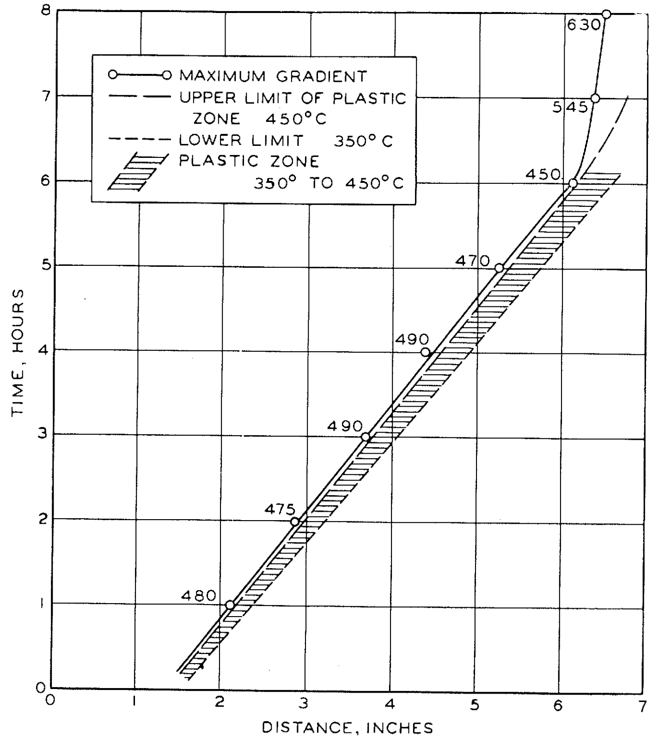


Fig. 71.—Travel of Plastic Zone, 350°C. to 450°C., and Position of Point at Maximum Gradient Temperature. *(Modified after "Coke from Illinois Coal," by G. Thiessen. Ind. and Eng. Chem. vol. 29, May, 1937.)*

The position of these temperature (350-450°C.) limits have been included in figure 71. The close parallelism of the line of maximum heat gradient and the upper limit of the plastic temperature range (450°C.) leaves no doubt that the maximum heat gradient occurs at the plastic zone.

Due to the influence of heat absorbed from above, the conditions in the upper 3 or 4 inches of the charge are not the same as those in the lower part. The heat gradients are greatly different in these two parts.

---

[9] Fieldner, A. C. and others, Methods and apparatus used in determining the gas, coke and by-product making properties of American coals: U. S. Bureau of Mines Bull. 344, p. 15, 1931.

The rate of travel of the position of maximum gradient, which may also be considered the rate of travel of the plastic zone, was found to be 0.78 inch per hour over the lower six inches of the charge. This is somewhat greater than the rates reported as occurring in standard slot-type coke ovens. It must not be overlooked that the coking time in inches per hour as frequently given for slot-type ovens is found by dividing the oven width by the coking time. The rate so obtained is twice the rate of coking from each wall to the center which would correspond to the rate considered here.

The combustion flue temperatures were 1360°C. (2480°F.) during the test.

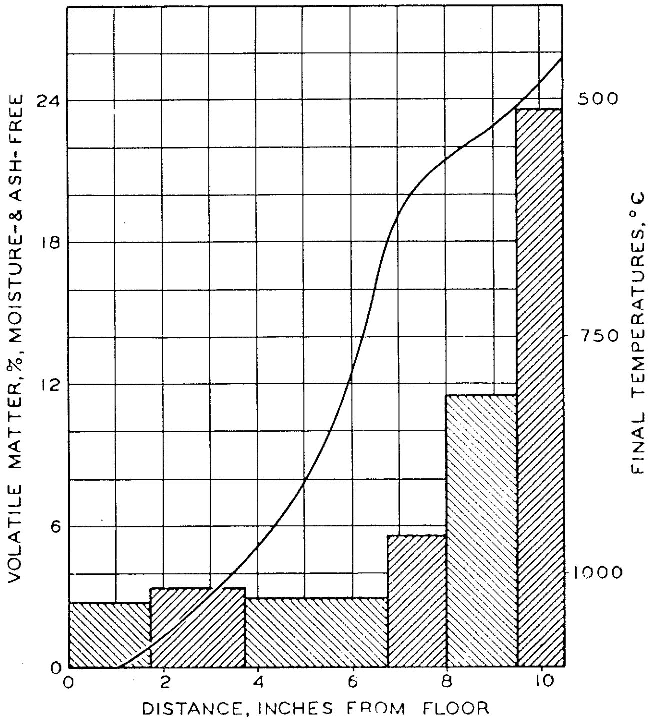


Fig. 72.—Relation of Heat Gradient and Volatile Matter Gradient at Completion of Coking. *(After "Coke from Illinois Coal," by G. Thiessen. Ind. and Eng. Chem. vol. 29, May, 1397.)*

Temperature in flues, regenerators, and stack.—Temperatures of the side walls of the sole flues in various ovens in the battery as measured with the optical pyrometer ranged from 1300°C. (2372°F.) to 1420°C. (2588°F.), with most of the flues at around 1350°C. (2462°F.). The side walls of the flues in oven No. 9 at the time of the test were at temperatures averaging 1360°C. (2480°F.).

The temperature of the flue gases leaving the last regenerator pass and entering the stack flue ranged between 230°C. (446°F.) and 590°C. (1094°F.) for ovens in normal operation. The temperature regularly increased during a regeneration cycle. Stack temperature ranged between 300°C. (572°F.) and 340°C. (644°F.) also reflecting the regeneration cycle but to a lesser extent.

ANALYSES OF COAL COKED AND OF COKE PRODUCED

The proximate analysis of the minus 5/16-inch screenings which were being coked in the ovens at the time the temperature measurements were being made and of the coke produced are given in Table 30. The coke analyzed is material remaining on a one-half inch screen after the original sample had been subjected to four drops in the standard shatter test. The minus one-half inch material removed is composed largely of uncoked material and of mineral matter which came away when the coke broke. The plus one-half inch coke corresponds roughly to the coke as sold after being screened to remove the fine materials.

TABLE 30.—ANALYSES OF COAL COKED AND OF COKE PRODUCED DURING TEST
(Dry Basis)

Sample of Sample No. Date sampled	Coal C–1545 10/10/35	Coke+½″ C–1563 10/11/35	Coke−½″ C–1556 10/11/35
Ash, per cent	12.1	16.1	20.4
Volatile matter, per cent	34.0	3.0	6.6
Fixed carbon, per cent	53.9	80.9	73.0
Total sulfur, per cent	1.43	1.36	1.44
Sulfate sulfur, per cent	0.01	....	....
Pyritic sulfur, per cent	0.96	....	....
Organic sulfur, per cent	0.46	....	....
Heating value, B.t.u./lb	12,650	12,100	11,540
Proportion of total sample, per cent	....	93.6	6.4

TABLE 31.—ANALYSES OF COALS COKED AND OF COKE PRODUCED AT VARIOUS TIMES IN THE SOLE-FLUE OVENS
(Dry Basis)

Sample of Sample No. Date sampled	Coke C–811 3/10/34	Coal C–1439 7/29/35	Coke C–1453 7/29/35	Coke C–1454 7/29/35
Ash, per cent	17.9	12.0	17.6	15.5
Volatile matter, per cent	5.3	33.8	5.1	4.2
Fixed carbon, per cent	76.8	54.2	77.3	80.3
Total sulfur, per cent	1.39	1.73	1.44	1.21
Sulfate sulfur, per cent	....	0.04	....	....
Pyritic sulfur, per cent	....	1.20	....	....
Organic sulfur, per cent	....	0.49	....	....
Heating value, B.t.u./lb	11,615	12,680	11,870	12,180
Ash softening temperature °F	....	....	2063	2087
Ash softening temperature °C	....	....	1128	1142

This procedure was adopted since in the sampling of this particular sample special care was taken to keep the sample in as large pieces as possible, resulting in the inclusion of the unconsolidated top layer. The analysis of this minus one-half inch material is included in Table 30.

In Table 31 are presented the analyses of samples of coke taken at other times from the main coke conveyor.

INFLUENCE OF OVEN CONDITIONS ON COKE CHARACTER

At the completion of coking about two-thirds of the charge had the nature of high temperature coke while the top one-third being coked under conditions of low temperature gradient and being finished at a low final temperature was more like low temperature coke. The 8:30 line on figure 68 shows the distribution of temperature in the charge at the time the charge was removed from the oven. To investigate the influence of these conditions pieces of coke representing the entire thickness of the charge were taken from a region close to the ends of the thermocouples (fig. 69). One of these was sectioned horizontally at approximately 1.75 inch intervals and the individual pieces were analyzed (Table 32); and cross sections at approximately 1.25 inch intervals and a longitudinal section through an entire thickness of other pieces also taken from near the thermocouples were made for a study of cell structure, according to the method of H. J. Rose.[10] The analyses of the sections are presented in Table 32. The volatile matter content of the sections has been plotted in figure 72 on which also appears the final temperature distribution in the oven.

TABLE 32.—ANALYSES OF CROSS SECTIONS OF COKE SPECIMENS MADE DURING TEST

Section No.	1	2	3	4	5	6	Weighted Ave.	Ave. Sample
Lab. Sample Number.	C–1564	C–1565	C–1566	C–1567	C–1568	C–1569		c-1563
Location, inches from floor	10.5–9.5	9.5–8.0	8.0–6.75	6.75–3.75	3.75–1.75	1.75–0		0-10.5
Thickness of section, inches	1.0	1.5	1.25	3.0	2.0	1.75	10.5	10.5
Ash,[a] (%)	12.9	15.4	15.7	15.5	17.0	15.5	15.5	16.1
Volatile matter,[a] (%)	20.5	9.7	4.7	2.4	2.9	2.3	5.5	3.0
Fixed carbon,[a] (%)	66.6	74.9	79.6	82.1	80.1	82.2	79.0	80.9
Sulfur,[a] (%)	1.23	1.18	1.31	1.34	1.41	1.29	1.31	1.36
Volatile matter,[b] (%)	23.5	11.5	5.6	2.9	3.4	2.8	6.5	3.5
Fixed carbon,[b] (%)	76.5	88.5	94.4	97.1	96.6	97.2	93.5	96.5
Final temp. at mid-point of section °C.	490	530	590	890	1040	1100	....	....

[a] dry basis
[b] moisture- and ash-free

---

[10] Rose, H. J., The study of coke macrostructure: Ind. Eng. Chem. vol. 17, No. 9, pp. 895 ff., 1925.

Fig. 73.—Rose Longitudinal Section, Natural Size, of Specimen of Sole-flue Oven Coke (No. 72, figs. 55c and 69).

Bottom of photograph illustrates structure near the end of the specimen resting on the oven floor.

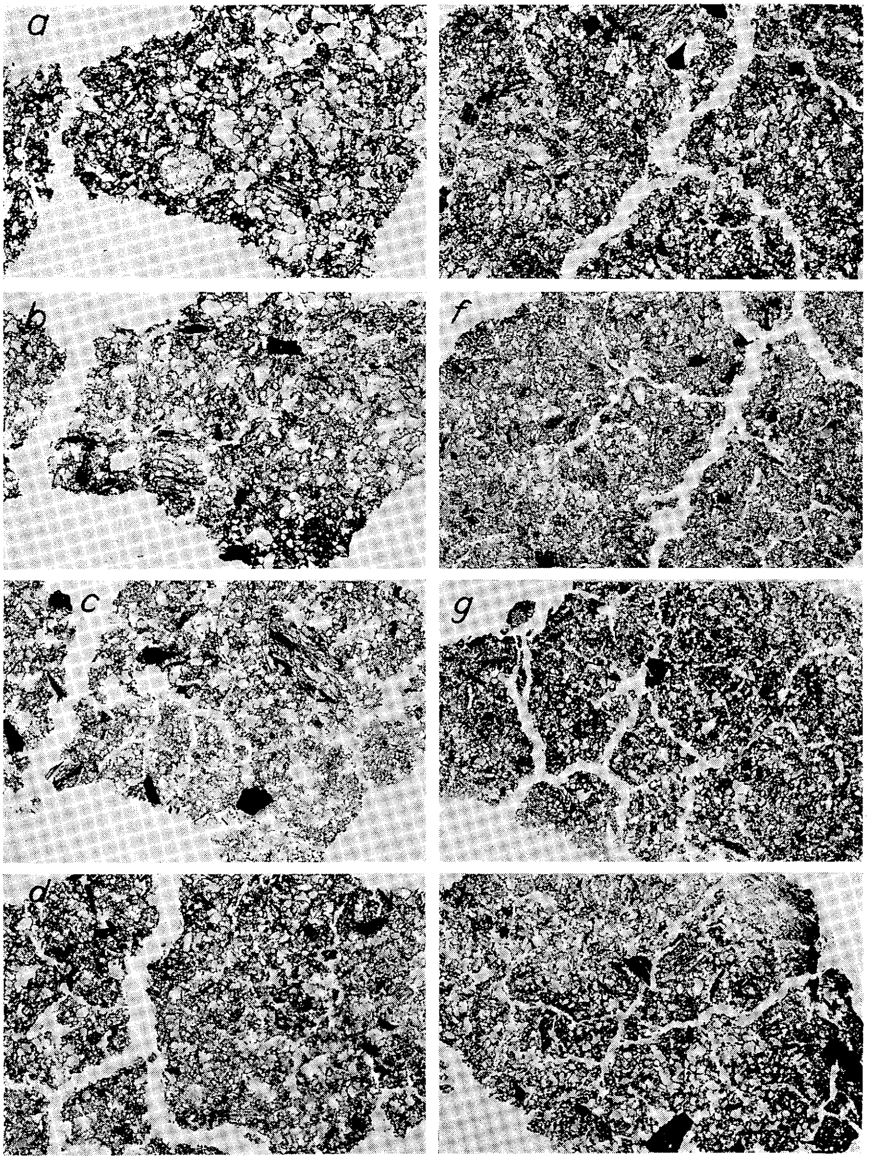


Fig. 74.—Rose Cross-sections, Natural Size, of Sole-flue Oven Coke (No. 72) Representing Horizontal Cuts in the Same Specimen (Fig. 73).

a. Section through top of specimen.
b through g. Sections at successive 1¼ inch levels from top toward bottom of specimen.
h. Section through bottom of specimen which rested on the floor of the oven.

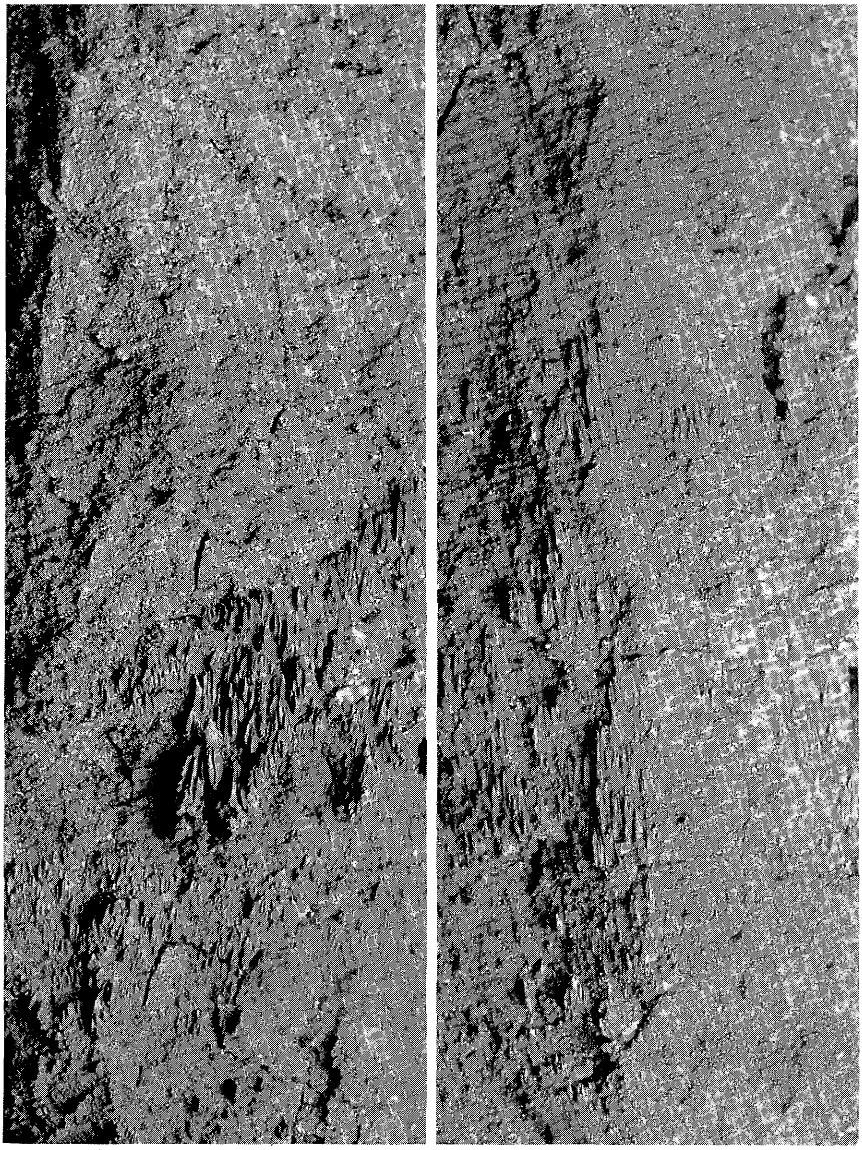


Fig. 75.—Stalagmite Carbon on Surface of Coke Made in Sole-flue Oven.

Longitudinal and cross sections of one of the blocks shown in figure 69, made according to the method of H. J. Rose, are shown in figures 73 and 74, respectively. The regular gradation of coke structure from a small celled, much shrunken, highly devolatilized, high temperature coke on the bottom (fig. 74*h*) to a large celled, very thin walled coke on the top (fig. 74*a*) is plainly visible.

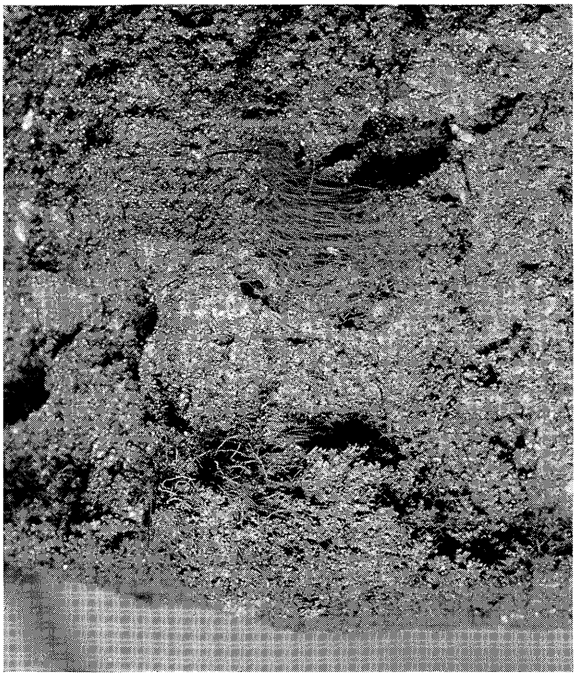


Fig. 76.—HAIR CARBON ON SURFACE OF COKE MADE IN SOLE-FLUE OVEN.

Carbon deposited in the form of stalagmites (fig. 75) and hair carbon (fig. 76) may be seen by close inspection of the surfaces formed during coking in the lower part of the charge. Such deposits are characteristic of the upper portion of the coke formed in beehive ovens and have been discussed at length by Roberts and Jenkner,[11] who believe that they are formed by the cracking of volatile matter as it passes from the downward travelling plastic zone up through the hot coke. The presence of such deposits in the lower part of the coke made in sole-flue ovens indicates that shrinkage cracks had formed in the coke in the lower part of the charge while the upper parts of the charge were still evolving volatile matter and may also indicate a too rapid coking rate.

---

[11] Roberts, J., and Jenkner, A., International coal carbonization: Pitman, London 1934. 453 pp. See Chapter V, especially pp. 103-108.

Volatile matter gradient in coke made during 10½ hours coking time.—Since the time the temperature measurements were made two new batteries of eight ovens each have been put into operation. All ovens are now operating on a ten and one-half hour coking cycle. Believing that it may be of interest to show the influence of a longer coking time upon the analysis of the coke, pieces of coke from a charge coked in one of the new ovens were sctioned horizontally at one-inch intervals and analyzed for moisture, ash, and volatile matter. (Table 33). The volatile matter content of the upper sections of this coke is much lower than those of corresponding sections from the eight-hour coke (Tables 30 and 31).

TABLE 33.—ANALYSES OF CROSS SECTIONS OF COKE SPECIMENS TAKEN FROM NEW OVENS 10.5 Hours Coking Time, Sampled July 22, 1936

Section No.	1 (top)	2	3	4	5	6	7	8 (bottom)
Sample No.	C1865	C1866	C1867	C1868	C1869	C1870	C1871	C1872
Location, inches from floor.	7–8	6–7	5–6	4–5	3–4	2–3	1–2	0–1
Ash,[a] per cent.	14.2	15.3	14.4	15.5	14.9	15.4	16.3	16.2
Volatile matter,[a] per cent.	5.0	4.4	3.4	2.8	2.4	1.6	2.3	2.5
Fixed carbon,[a] per cent.	80.8	80.3	82.2	81.7	82.7	83.0	81.4	81.3
Volatile matter,[b] per cent.	5.8	5.2	3.9	3.4	2.8	2.0	2.7	2.9

[a] Dry Basis.
[b] Moisture-and-ash-free basis.

GAS ANALYSES

Five-gallon gas samples were taken at three different times and analyzed. The samples consisted of: (1) a pair of samples taken July 29, 1935 from the main gas line, one just before the gas entered, the other just after the gas left the sulfur removal equipment; (2) a sample taken Oct. 11, 1935 just before the gas entered the sulfur removal equipment; and (3) two samples taken July 22, 1936 from the suction mains coming from the two new batteries and from the old battery, both just before the gas entered the suction pump. The samples taken at all times were analyzed in a modified Shepherd-Orsat apparatus[12] which had been equipped for the determination of hydrogen by combustion over copper oxide,[13] and for the determination of illuminants by absorption in fuming sulfuric acid. The sample taken at the second time was also analyzed by fractional distillation in a Podbielniak Model A precision fractioning unit.[14]

---

[12] Shepherd, M., An improved apparatus and method for the analysis of gas mixtures by combustion and absorption: Bureau of Standards Jour. of Research vol. 6, No. 1, pp. 121-167, Jan. 1931. Research Paper No. 266.

[13] Burrell, G. L., Seibert, F. M., rev. by Jones, G .W., Sampling and examination of mines gases and natural gas: U. S. Bureau of Mines Bull. 197, pp. 46-8, 1926.

[14] Podbielniak, W. J., Apparatus and methods for precise fractional-distillation analysis: Ind. Eng. Chem., Anal. Edition, vol. 3, pp. 177-188, April 15, 1931.

The results of these analyses together with published analyses of coke oven gas[15, 16] are presented in Table 34. The heats of combustion are calculated from the analyses of the gas as taken from the sample bottles in the laboratory. At the time the samples were taken in the plant an automatic recording calorimeter indicated a heat of combustion for the gas of 480 B.t.u. per cubic foot. The lower temperatures in the oven are reflected in a lower content of methane and probably a lower hydrogen content, greatly increased by cracking.

TABLE 34.—ANALYSES OF COKE OVEN GAS PRODUCED IN SOLE-FLUE OVENS

Orsat Analysis

Description	Sole-flue oven gas					Standard coke oven gas		
	a	a	a	b	b			
Sample No.	G–111	G–112	G–113	G–128	G–129	(14)	(15)	
Date of sampling	7/29/35	7/29/35	10/11/35	7/22/36	7/22/36			
Sampling position	Before H_2S removal	After H_2S removal	Before H_2S removal	Main from new batteries	Main from old battery			
CO_2, per cent....	3.7	4.4	3.4	5.4	4.1	2.2	2.6	1.4
O_2, per cent......	1.3	1.2	0.7	0.6	1.3	0.8	0.6	0.5
Illuminants, per cent..........	2.6	3.3	2.2	2.8	1.9	4.0	5.2	2.9
CO, per cent.....	14.2	14.9	15.7	12.2	14.4	6.3	6.1	5.1
H_2, per cent.....	44.0	40.0	46.5	45.7	44.3	46.5	47.9	57.4
N_2, per cent.....	17.0	17.3	15.2	11.7	17.7	8.1	3.7	4.2
CH_4, per cent....	15.1	17.6	16.1	20.0	15.1	32.1	33.9	28.5
C_2H_6, per cent...	1.7	1.3	0.2	1.6	1.2	....	....	....
H_2S, per cent....	0.4	negative to lead acetate paper	not determined	not determined	not determined	....	....	....
B.t.u./cu. ft. gross, calc.....	423	441	407	471	400	574	600	536

[a] Eight and one-half hours coking time.
[b] Ten and one-half hours coking time.

Fractionation Analysis G-113

Methane and lighter H_2, CH_4, CO, O_2, N_2, per cent..............	96.3[c]
Ethane and ethylene, per cent.....	1.8
Pentanes and higher, per cent......	1.9

[c] Too much methane and lighter constituents for satisfactory fractionation of constituents higher than methane fraction.

---

[15] Combustion, a reference book on theory and practice: American Gas Association, Ed. 3, 1932, p. 95.

[16] Haslam, R. T., and Russell, R. P., Fuels and their combustion: New York, McGraw-Hill, p. 282, 1926.

Part V — Conclusions and Recommendations

CHAPTER XVII

The present report must be considered largely as a progress report; it by no means provides all of the information necessary or desirable for the commercial carbonization of Illinois and similar coals. Certain conclusions may be drawn from the experimental work reported here and from the review of the previous available information and some recommendations for additional desirable work may be made.

SCOPE OF THE REPORT

This report includes a study of the economic factors involved in the production of coke from Illinois coal, a consideration of the possible market for such a coke, a review of the fuel requirements for the Illinois coal market area and a review of the production and sale of coke in that area. The history of the production of coke from Illinois coals is briefly sketched and the difficulties which were encountered are reviewed.

Considering the question as to whether Illinois coals are coking coals, various criteria which have been proposed for the identification of coking coals have been reviewed and applied to the coals of Illinois. These criteria are based largely upon the behavior of coals from which satisfactory metallurgical cokes have been made and are probably too stringent in identifying coals from which satisfactory domestic coke can be made.

The section of the report covering the experimental work carried on in an investigation of the possibilities of making coke, particularly domestic coke, from Illinois coals includes the determinations of the coke, gas, and by-product yields of various Illinois coals, the temperature range during which they are plastic, their agglutinating or caking values, the influence of geographical location, size, admixtures, preparation, and the temperature of completion of coking on coke quality. The influence of impurities such as moisture, ash and sulfur on coke quality are discussed and an experimental study of the influence of the organic and pyritic forms of sulfur in the coal on the sulfur content of the coke is reported. The petrographic composition of Illinois coals is briefly described and the minor significance of petrographic composition on coke quality in the case of the majority of Illinois coals is discussed.

An installation of Knowles sole-flue coke ovens at West Frankfort, the only plant at which coke is at present being made from Illinois coal, is described and some details of its operation, especially of the temperature conditions in the oven, are reported.

CONCLUSIONS

As a result of the experimental work and review of previous information, presented in this report, the following general conclusions may be made.

(1) Under favorable conditions coke suitable for domestic and some industrial fuel uses can be made from most of the coals occurring in Illinois.

(2) The domestic fuel market appears to be the best and to be a growing outlet for coke made from coals from the Eastern Interior coal basin, of which most come from Illinois.

(3) Previous attempts to produce coke commercially from Illinois coals have failed, probably because its sale was attempted in the metallurgical coke market, the market for domestic coke having not yet been foreseen or developed.

(4) Illinois coals should be coked with as little delay as possible after having been removed from the mine face, say within two weeks at the most.

(5) Mineral impurities in the coal should be kept at a minimum, not only to obtain a coke with a low content of impurities but in order to obtain a stronger and better structured coke.

(6) The organic sulfur content of the coal sets the lower limit to the sulfur content which can be attained in the coke by processes which treat only the coal.

(7) Because of the high volatile matter content of Illinois coals, cokes made from them shrink excessively when completely devolatilized and develop a highly fractured, fingery structure. The addition of non-coking, non-shrinking carbonaceous material to the coal before it is coked as a means of minimizing this shrinkage is not practical in the case of Illinois coals because of their small caking power or ability to cement non-coking material into a homogeneous mass.

(8) Compared with the range of variation in carbonization yields and behavior under the influence of heat possible among coking coals, Illinois coals are relatively uniform, with any variations which do occur taking place gradually as the coal fields are traversed and may in general be correlated with changes in rank as indicated by the moist mineral-matter-free calorific value.

(9) Coke is being made commercially from Illinois coal screenings in ovens of a type which have not previously been used commercially. In these ovens the coal is heated in a wide flat layer, mainly from below. No conclusions are to be made concerning the commercial success of this plant from the data presented.

(10) Before commercial production of coke from a given coal by a given process is attempted, tests should be made to determine the effects of the various factors which may be encountered, such as the time elapsed after the coal was

removed from the face, the influence of the coal preparation, a cleaning process to which the coal might be subjected, and the effects of segregation of components of the coal if screenings are to be used.

RECOMMENDATIONS FOR ADDITIONAL WORK

(1) The addition of minor quantities of strongly caking low volatile coals to Illinois coals in order to improve the coke made from them has not been particularly successful. Some of the results presented in this report indicate that this may be due to the differences in the temperature range over which the two coals become plastic. Additional study of this point seems to be desirable, both to either establish or disprove this theory, and to find, if possible, a suitable coal for blending which will materially improve the quality of the coke without prohibitively increasing the cost.

(2) Additional coals, particularly from Northern and Central Illinois, should be investigated as to the quality of coke which can be made from them under the most favorable conditions.

(3) The influence of coal cleaning as now carried out in the various types of coal cleaning plants on the coke-making properties of the coal, especially any tendency towards increasing the rate at which the coking property is lost during the period between cleaning and coking, should be studied.

Appendices

By Gilbert Thiessen and Paul E. Grotts

APPENDIX A

DESCRIPTION OF THE BY-PRODUCT ANALYSIS METHOD AS APPLIED TO ILLINOIS COALS

The test adopted for the by-product analysis method was the dry distillation test described by the chemists of the United States Steel Corporation.[1] A fused quartz tube was used in these experiments in place of the Jena glass distillation tube specified by the Steel Corporation method. To distinguish between the two methods, which are identical in other respects, the test employed by this laboratory has been designated as the coal carbonization assay in the following discussion.

Method of the Coal Carbonization Assay

A 20-gram sample of coal is progressively carbonized at 900° C. and the volatile products are passed through crushed silica brick, maintained at 720° C., to absorption bulbs, a freezing tube, and a gasometer. The apparatus and procedure provide for the separation and subsequent estimation of the gross fractions of the distillation products which are coke, tar, ammonia, liquor (water), hydrogen sulfide, carbon dioxide, light oils, and fixed gas.

Character of the Assay

The test is carried out in laboratory scale with the 20-gram portion of coal being subjected to controlled heat effects and cracking surfaces which approximate the corresponding effects existing under slot-type coking plant operation. The results obtained by the analysis are empirical and the yields do not conform absolutely to yields from any one plant. However, an estimate is obtained, by means of the test, of the practical coke and by-product yields from a coal. The non-conformity is due to the impossibility of accurately reproducing the action of a coking plant in small laboratory scale. To arrive at a quantitative prediction of the behavior of a coal in commercial carbonization relationships between the assay and the plant yields must be evaluated. A correlation may be established which may be used in interpreting the assay analysis. Inasmuch as the yields indicated by the assay are relative it is not of immediate importance to establish the correlation in a study of the coking possibilities in a series of coals. Thus a variation in the amounts of tar obtained in two specific coals would be approximately the same in both the laboratory test and in plant yield.

In the assay the coal charge is heated progressively. Heat is first applied to the discharge end of the test charge corresponding to coal next to the oven wall in a freshly

---

[1] Methods of the Chemists of the United States Steel Corporation for the Sampling and Analysis of Coal, Coke and By-Products, 3rd Ed. pp. 130-143, 1929.

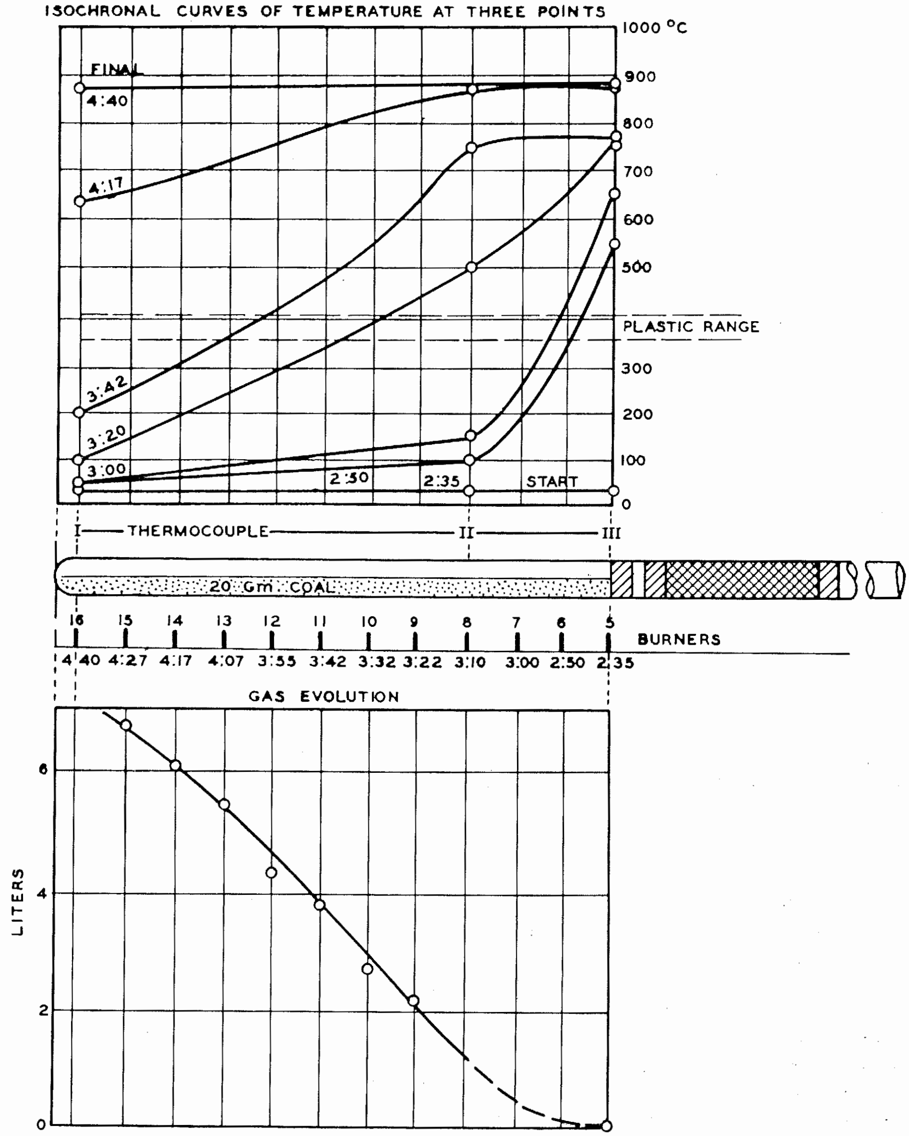


Fig. I.—Travel of Heat and Evolution of Fixed Gas in a Carbonization Assay Test. (Carbonization No. 44.)

charged coke oven. The evolved gases pass outward through a section of coarse silica particles which is held constantly at a temperature of 720°C. This provision is made to expose the coal gases to the same kind of cracking surface found in the coke oven and to such an extent that equivalent cracking takes place. The first heating of the test charge occurs at the end corresponding to the oven wall face and when that portion has coked, an additional section of uncoked coal is heated. Continuing in this way the whole test charge is carbonized. Although the manipulation of the burners heating the charge proceeds in a step-wise fashion the temperatures in the coal charge grade continuously, but not linearly, from the hot end to the cold. At the end of the test all of the coal charge has been carbonized and the coke has reached 900°C. Throughout the heating, however, the silica portion of the tube packing has been held at 720°C.

TABLE I—CARBONIZATION ASSAY TEST NO. 44
Sample: R-24 (Franklin Co. No. 6 Coal)
(Plastic Range: 360-408°C.)

Time	Thermocouple		
	I 11″ from open end of charge	II 3″ from open end of charge	III Open end of charge
2:35	35°C	35°C	35°C
2:50	50	100	550
3:00	50	160	650
3:20	100	500	750
3:42	200	750	775
4:17	630	870	875
Final 4:40	875	875	880

The temperature experience of the coal and coke in the laboratory assay may be graphically represented. Data from one of the tests (Table I) has been used to fix the temperature curves in figure I which represents temperatures along the coal charge at

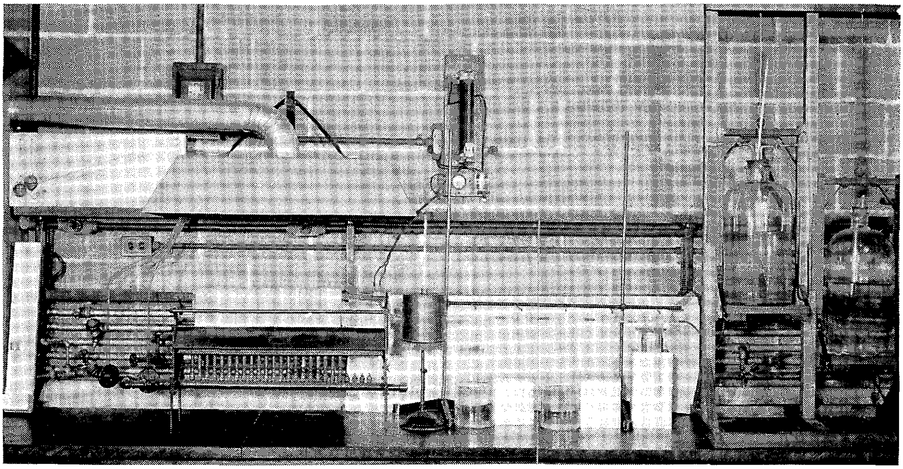


Fig. II.—CARBONIZATION ASSAY EQUIPMENT.

specific times. A separate measurement by the use of the Agde-Damm plastic range test has shown the plastic range of this sample of coal to have been from 360° to 408°C The curves show that the face end of the test charge had softened and solidified within a few minutes after heating was started. However, this position in the charge did not reach a temperature of 900°C. until late in the coking period. This was due to the cooling effect of the evolved gases which had to pass through and over the carbonized coal. The cooling effects of the volatile products of the coal when passed over the portions of the charge already carbonized would be due in part to the absorption of heat in the cracking of the gaseous materials. The impossibility of raising these parts of the charge to 900° early in the test was found to be typical of the test.

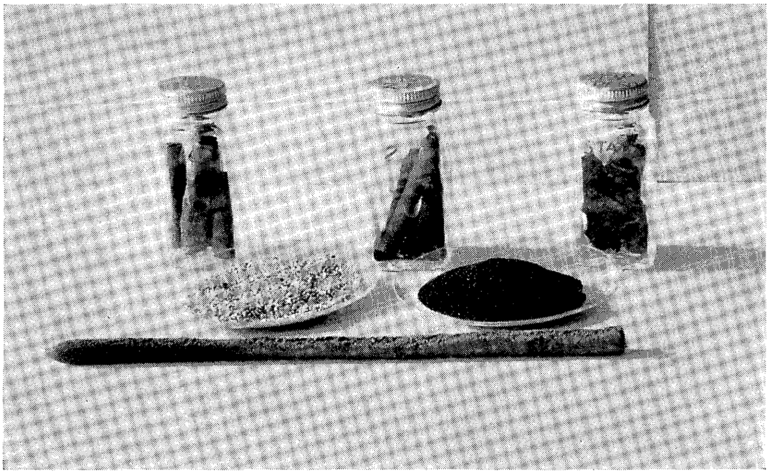


Fig. III.—SPECIMENS OF SILICA, COAL, AND COKE.

The plastic range traveled the length of the charge and then disappeared before the burners had been lighted under the last ¼ of the charge. These effects are at variance with the conditions found in a coke oven. First the coking period is much shorter, but this is dependent on the second effect: that the uncoked coal is heated by radiation and convection arising both outside and in the tube. In the coke oven, heat must invariably pass through the fused plastic zone before the uncoked coal may be raised to its plastic temperature. The plastic zone is a most efficient heat insulator. Thus the coking period in the coke oven depends on the travel of the plastic zone through the charge, which in turn depends on the heat applied and the nature of the coal. The situation is different in the assay since the plastic zone does not exist in the same form as in the coke oven. The manner of applying the heat to the test charge precludes the existence of the thin layer of plastic coal with a high temperature gradient through it. Instead heat reaches the uncoked coal from almost all directions.

Preparation of the Sample

Samples of coal were prepared by the standard method of the American Society for Testing Materials for the preparation of 60-mesh samples of coal for analysis except that the samples were ground to pass a 40-mesh U. S. Standard Series wire mesh sieve (35-mesh Tyler Standard series).

The 40-mesh coal is dried at 105°C. for 1 hour and cooled in a desiccator over calcium chloride.

Apparatus and Procedure

The assembled test equipment consists of the carbonization tube and a gas-fired combustion type of furnace exactly as described in "The Method of the Chemists of the U. S. Steel Corporation for the Sampling and Analysis of Coal, Coke and By-Products," the tar piece, the tar filter enclosed by a steam jacket, a Geissler bulb to contain sulfuric acid and which is immersed in an ice-water bath, a drying U-tube, a second Geissler bulb to contain potassium hydroxide solution and which is also immersed in an ice-water bath, another drying tube, the freezing tube and finally the gasometer.

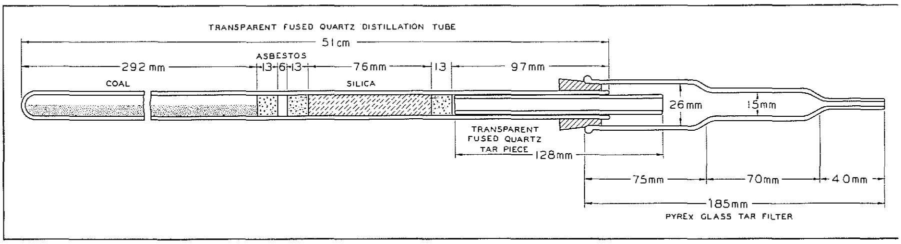


Fig. IV.—DISTILLATION TUBE, TAR PIECE, AND TAR FILTER FOR CARBONIZATION ASSAY.

Equipment:

(1) Gas-fired combustion furnace
(2) Steam jacket
(3) Battery jars (two) for ice-water baths
(4) Wide mouth quart size Dewar flask
(5) Gasometer (see figure II)
(6) Fused silica distillation tubes (three)
(7) Fused silica tar pieces (three)
(8) Pyrex tar filters (three)
} see specifications figure IV

(9) Geissler bulbs (four)
(10) Glass stoppered U-tubes (four)
(11) Freezing tube (two) (see figure V)

(12) Auxiliary equipment included the automatic four-point temperature recorder, and sampling and analytical equipment.

The furnace.—The special gas-fired combustion furnace used in the test is equipped with 25 individually controlled Bunsen type burners. The furnace supplied by several scientific supply houses does not meet the specifications as set forth in the description referred to above. The method described as used in the laboratories of the U. S. Bureau of Mines in the Bureau of Mines, American Gas Association cooperative studies of the coal, coke, and by-product making properties of Illinois coals also does not meet these specifications, the furnace used being one supplied by a number of apparatus supply houses with a smaller number of Meeker type burners. The method specifies a type of flame and rate of heating which is obtainable only when artificial gas having a rapid rate of combustion and a short hot flame is used. The laboratories of the Illinois State Geological Survey were supplied with such a gas when this equipment was first put into use. Shortly thereafter the gas supply was changed at various times to natural gas or to a mixture of natural and manufactured or reformed natural gases. Even with burners of different design it was impossible to obtain the required type of flame and rate of heating when gas alone was supplied to the burners. This problem was solved by supplying a mixture of gas and air to the gas inlet of the burners. Both gas and air lines were equipped with flow meters and the gas line with a manometer preceding the flow meter in order that suitable flames could easily be reproduced. One specified condition of the test is that the portion of the distillation tube containing the crushed silica is to be maintained at 720°C. To facilitate the control of that part of the tube the first four gas burners were replaced by a small electric tube furnace fitted with a thermocouple connected to the recorder. Three other thermocouples were installed to measure the temperatures of the tube at 0, 3, and 11 inches from the "face", or open end of the leveled coal charge. Temperatures were recorded at four points in the tube throughout the test.

The distillation tube and its preparation.—The tube is fused silica, 51 cm. in length and 18.5 mm. in outer diameter. In the test it contains a 20-gram coal sample, asbestos packing, and sized silica particles.

The cleaned, dried distillation tube is weighed and a weighed sample of coal is placed in it. Previously ignited asbestos, rolled into a plug 13 mm. long is inserted to a position 292 mm. from the closed end of the tube. Another 13 mm. plug of asbestos is pushed into the tube to a position 6 mm. from the first plug. Crushed silica brick sized to pass the 10-mesh and be retained by the 20-mesh screen is poured into the tube until the column of silica particles is 76 mm. in length, held in place by another asbestos plug 13 mm. in length which completes the packing of the tube. The tube and its contents in place ready for a test is shown in figure IV.

After weighing the packed tube and leveling the coal charge by rolling, the tube is ready for the test. At the end of the test it is reweighed and the loss of volatile matter is determined. The yield of coke is determined as the difference between the weighed sample and the amount of the volatile matter driven off.

The tar piece.—A fused silica sleeve, 128 mm. in length and with an outside diameter of 14 mm., accurately weighed, is inserted in the end of the packed and weighed distillation tube to receive the tar condensate that forms in the cooler end of the distillation tube. The amount of this tar is combined with the amount of tar found in the succeeding tar filter. In order that the high temperature tar condense in the tar piece it is necessary that it fit snugly in the distillation tube. Its length is such that one end touches the asbestos plug (fig. IV) and the other protrudes from the distillation tube.

The tar filter.—Also shown in figure IV is the Pyrex glass tar filter. It steps down from a tube of 1 inch inner diameter to the ¼ inch outlet tube, and is packed loosely with dried absorbent cotton. The function of the tar filter is to remove suspended tar from the evolved gases. It is weighed when packed and is connected to the distillation tube by means of a gas-tight cork (faces of cork, 1¼ inches and 1 inch, length 1 inch). From its weight at the end of the test the amount of tar condensed at 100°C. is computed.

During the test the tar filter is enclosed by a steam jacket consisting of a cylindrical copper can, figure II, fitted with a steam inlet and thermometer. The outlet end of the tar filter which is connected to the first Geissler bulb is kept hot by extending a small cylindrical sleeve from the steam jacket. This provision is necessary to prevent the collection of crude naphthalene in the small tubes.

The absorption train.—The first Geissler bulb which contains 8 cc. of normal sulfuric acid and one drop of methyl orange indicator solution added as an indicator is connected to the tar filter. This bulb collects water, naphthalene, and free ammonia. Connected to the outlet of the bulb is a glass stoppered U-tube containing calcium chloride previously saturated with CO_2 to absorb any water which the gas may pick up from the acid in the bulb. The two are weighed together.

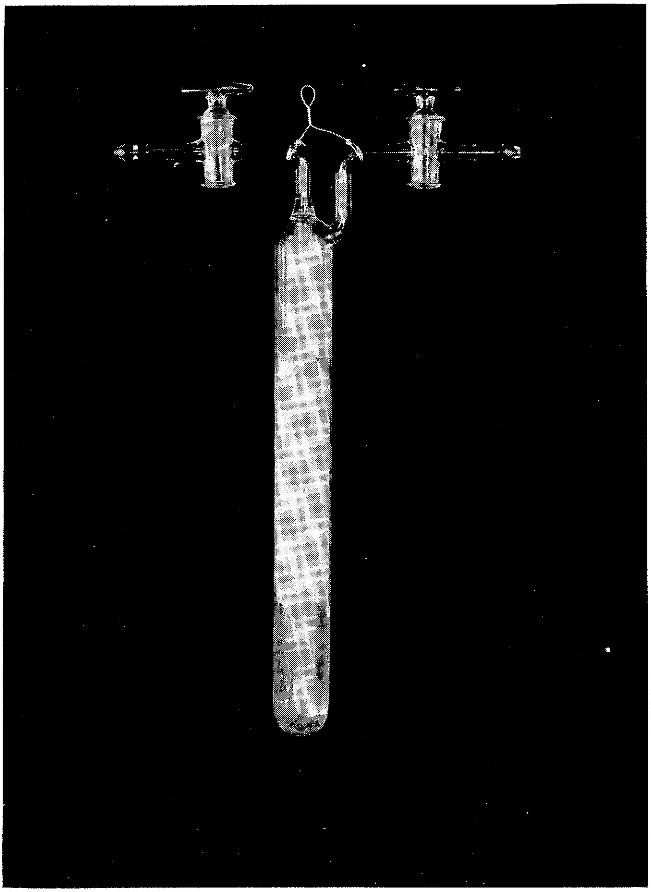


Fig. V.—FREEZING TUBE FOR THE COLLECTION OF LIGHT OIL. THE INLET IS TO THE LEFT.

The second Geissler bulb contains 13 cc. of 1:1 potassium hydroxide solution and absorbs hydrogen sulfide and carbon dioxide. A calcium chloride U-tube is also connected to the outlet of this Geissler bulb. It is connected to the inlet of the freezing tube. As in the first Geissler unit the potassium hydroxide bulb is weighed with its drying tube. Both Geissler bulbs are immersed in an ice bath during the test.

The freezing tube.—The light oils are condensed in the freezing tube, figure V, which is immersed in a mixture of dry ice and acetone. The freezing mixture is contained in a wide mouth quart Dewar flask which is supported in cotton packing. The amount of light oils is determined by the increase in weight of the tube. They are removed and discarded by sweeping the tube with dry air at room temperature.

The gasometer.—The apparatus (fig. 11) used to collect the fixed gases leaving the absorption train consists of two 12-liter bottles suspended on movable cages for convenient raising or lowering. The bottle used to receive the gas is graduated in 200 ml. divisions, the other bottle used for leveling the solution over which the gas is collected is not graduated. The gasometer bottles contain 13 liters of a saturated solution of sodium sulfate to which was added 5 per cent by volume of sulfuric acid. The solution was saturated with the fixed gases before starting the assay tests. The gas-receiving bottle is fitted with a rubber stopper carrying a thermometer, manometer, outlet tube (for sampling), inlet tube and siphon connecting the two bottles.

At the start of the test the receiving bottle is filled with the gasometer solution and during the test the height of the cage is adjusted so that a pull of about 6 inches of water is imposed on the system. At the end of the test the pressure of the collected gas is brought to atmospheric pressure by adjusting the relative positions of the bottles, and the temperature, barometric pressure, and gas volume are recorded.

Manipulation.—The packed and weighed distillation tube containing the tar piece and with the tar filter attached, is rolled in the hands and gently tapped to level the coal charge to give a free and open passage for the gases evolved during the test and to adjust the coal level uniformly for observation of the coking qualities. The tube is placed in position in the asbestos-lined trough for supporting it in the furnace, so that the first asbestos plug is immediately above the first burner. The copper steam bath is next placed in position and the small sleeve is placed over the outlet end of the tar-filter to keep the small end of the filter and the inlet tube of the first Geissler bulb hot to prevent condensation of naphthalene during the test. The first Geissler bulb, containing the sulfuric acid, is now attached to the tar filter with seamless rubber tubing and the outlet of the bulb is attached to its accompanying drying tube containing granular calcium chloride previously saturated with CO_2 for the absorption of water.

The potassium hydroxide bulb is connected to the drying tube of the first Geissler bulb and it in turn is connected to its drying tube which is connected to the freezing tube. The freezing tube is provided with its freezing jacket containing acetone and dry ice, and below each Geissler bulb is placed a battery jar ready for the subsequent addition of ice-water. The purified gas is lead from the freezing tube to the 12-liter gasometer, in which it is collected, with about three feet of ⅛-inch rubber tubing.

The train, connected as described above, is tested for possible leaks by lowering the leveling bottle to produce suction and successively opening the stopcocks on the gasometer and the absorption train, starting at the last tube and working toward the furnace.

Heating the combustion train.—When the train is found to be air tight, and after the silica brick becomes heated to a constant temperature of 720°C., which must be maintained for this section throughout the test, the gas inlet stopcock on the gasometer is closed, and the gasometer is refilled with solution. The leveling bottle is then set at a position such that there will be a suction of 6 inches of water from the gasometer at the end of the distillation. The fifth burner, that is, the one under the asbestos plug, is lighted immediately, and the stopcock on the gasometer is opened for the collection of gas. In a few minutes the gas pressure in the system increases so that ice and water may be safely placed in the battery jars containing the absorption bulbs. To cool the evolved gases so that a maximum absorption may be obtained, it is necessary to keep all of the absorbing bulbs in ice water. At the end of 10 minutes after the fifth burner is lighted, the sixth burner, or the first one under the coal charge, is lighted. The rest of the burners are lighted successively at approximately 10-minute intervals, the exact time being regulated by the speed of evolution of the gases. After all burners have been lighted, the heating is continued until there is practically no further evolution of gas.

Disconnection of the train at the end of the test.—When the evolution of gas has ceased the burners of the furnace and the steam jet are turned off, and the suction of the gasometer is left on the train. If no leaks have developed during the distillation, the solutions in the Geissler bulbs will start to draw back as the furnace cools. The stopcocks on the gasometer and on the first drying tube are then closed.

The apparatus is allowed to cool, after which the absorption train is disconnected from the tar filter and freezing tube. Two liters of air free from carbon dioxide and moisture are slowly drawn through the absorption bulbs and drying tubes to displace the gas to bring them back to the same conditions under which they were first weighed. The bulbs with their accompanying drying tubes are then removed from the ice bath and allowed to stand until they have reached room temperature, when they are dried and weighed. The freezing tube which was removed from the train before air was drawn through the train is treated in a manner identical with the treatment before its first weighing.

To avoid losses from oxidation due to air coming in contact with the hot tar and coke, the distillation tube and tar filter are allowed to cool nearly to room temperature before they are separated. The tar filter is then disconnected and weighed, and the distillation tube is allowed to stand in a vertical position for 20 minutes, so that it will fill with air, when it is also weighed.

Analytical Procedure

The separation and estimation of naphthalene.—The contents of the sulfuric acid bulb are transferred after the final weighing to a separatory funnel. The bulb is washed thoroughly with water and then with pentane and the washings are added to the separatory funnel. In washing the bulb with pentane care is taken to dissolve all of the naphthalene. After agitation the water layer is drawn off and reserved for the ammonia determination. The naphthalene-pentane layer in the separatory funnel is washed several times with water and the washings are added to the acid solution. The pentane solution is poured through a dry filter paper, this procedure is repeated, and in each step the filter paper and funnel are washed with fresh pentane. In this way water droplets are removed from the pentane solution. The last filtration is made into a weighed glass-stoppered 125 cc. Erlenmeyer flask and the pentane is evaporated in a stream of dry air at 0°C. When the evaporation is complete the flask is reweighed and the weight of naphthalene determined. The apparatus is pictured in figure VI.

Determination of free ammonia.—As distinguished from the ammonia present in the tars referred to as combined ammonia, the gaseous ammonia is absorbed in the acid bulb and may be determined by the usual method of evolution and absorption in a known amount of standard acid as follows.

The ammonia-acid liquor from the naphthalene separation is set aside in a Kjeldahl flask and is then ready for the ammonia determination. To the dilute acid solution approximately 1 gm. of granulated zinc and 20 cc. of 1:1 potassium hydroxide are added and the flask is connected at once to the ammonia distilling head. The distillate is collected in a dilute solution of a known amount of N/10 sulfuric acid. At the end of the distillation the excess acid is titrated with N/10 sodium hydroxide. Blank determinations are made on the reagents used. From the data obtained the amount of ammonia is computed.

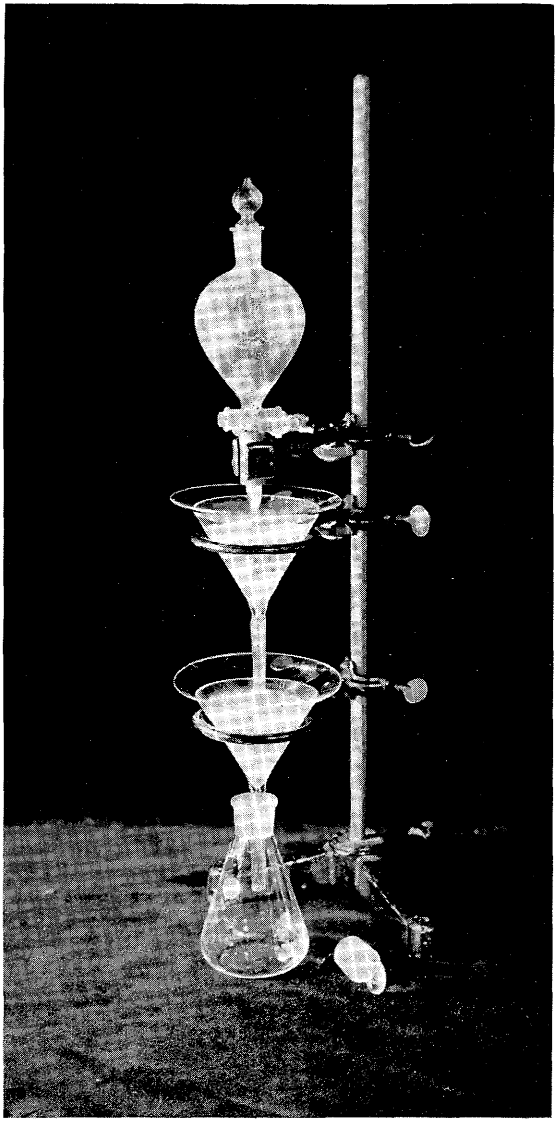


Fig. VI.—APPARATUS FOR FILTERING PENTANE-NAPHTHALENE SOLUTION.

Determination of combined ammonia.—The tars obtained in the coal carbonization assay contain combined ammonia. The contents of the tar piece and tar filter are extracted by placing them in boiling distilled water for ten minutes, which is then filtered.

The determination of the ammonia in the water used for the extraction is carried out as in the free ammonia determination. Less acid may be used in the flask receiving the ammonia distillate since the amount of combined ammonia occurring is much less than the amount of free ammonia. Blanks are also run.

Determination of hydrogen sulfide.—The potassium hydroxide in the second Geissler bulb is transferred into a 100 ml. volumetric flask, the bulb is washed several times with water and the washings are added to the volumetric flask. The solution is diluted to 100 ml. and portions are removed for the hydrogen sulfide determination.

An aliquot part of the potassium hydroxide solution is added to an acidified known excess of N/10 iodine solution diluted with 300 cc. of water. The excess iodine is determined by titrating with N/10 sodium thiosulfate.

Calculation of Results

The following is a guide to the computation of the weights in grams of the various products of the distillation. The conversion of the computed weights of each to weight per cent, on the basis of the dry coal as 100 per cent, is made by multiplying by 5.

(1) *Volatile matter* equals the loss in the carbonization tube and includes all the gaseous products and the tar.

(2) *Coke* equals the remainder obtained by subtracting the weight of the volatile matter from 20, the weight of the sample of coal.

(3) *Ammonia* equals the ml. of acid multiplied by the factor: equivalents per ml. × 17.03.

(4) *Tar* equals the increase in weight of the tar filter and of the tar piece, plus the naphthalene, minus the combined ammonia.

(5) *Water* equals the increase in weight of the sulfuric acid bulb and drying tube minus the naphthalene and free ammonia.

(6) *Hydrogen sulfide* equals the ml. of N/10 iodine required for the entire bulb multiplied by .0017. The liters of hydrogen sulfide equal its weight divided by 1.4564 which is the weight of one liter at 60° F. and 30″ Hg.

(7) *Carbon dioxide* equals the increase in weight of the potassium hydroxide bulb and drying tube, minus the hydrogen sulfide. The liters of carbon dioxide equals its weight divided by 1.873 which is the weight of a liter at 60° F. and 30″ Hg.

(8) *Light oil* equals the increase in weight of the freezing tube.

(9) *Gas.*—Grams of gas equal the grams of volatile matter minus the sum of the tar (including naphthalene), ammonia, water, hydrogen sulfide, carbon dioxide, and light oils.

(10) *Computations of the gas volumes.*—To convert the observed volume of gas to a dry basis at 30″ Hg and 60° F., formula No. 2, which is derived from formula 1, was used.

$$V^1 \times \frac{459.6^\circ + 60^\circ}{459.6^\circ + t} \times \frac{P - P_1}{30} = V_2 \qquad (1)$$

$$V_1 \times \frac{17.32\,(P - P_1)}{459.6^\circ + t} = V_2 \qquad (2)$$

V_1 = observed volume of gas

V_2 = corrected volume (30″ Hg and 60° F, dry)

P = observed barometric pressure in inches of mercury

P_1 = vapor pressure of water[1]

t = temperature of gas at the time of reading the gas volume (temperatures are all Fahrenheit)

---

[1] The vapor pressure of water is used in the computations. Although the gas is collected over the acidified sodium sulfate solution the vapor pressure lowering due to the salt was not great enough to alter the corrected volumes as recorded. The magnitude of the effect was investigated by analysis of the gasometer solution and by calculations based on vapor pressures found in International Critical Tables.

The gas yield in cubic feet per ton of coal is found by multiplying the corrected liters of gas into the factor 1600.[2]

(11) *Specific gravity of the gas.*—The specific gravity of the gas is computed using the gas analysis data corrected for air and by the use of the following values:

Constituent	Sp. Gr.
CH_4	0.5543
CO	0.9671
Illuminants	1.1520
H_2	0.0695
N_2	0.9673
O_2	1.1053

In addition to specific gravities determined by calculation from the gas analysis the specific gravities of the gases obtained in this study were determined experimentally. The determined values were corrected for air by subtracting the value: (per cent N_2 × Sp. Gr. N_2 + per cent O_2 × Sp. Gr. O_2) and by adding the value: (per cent [N_2 + O_2] × Sp. Gr. H_2).

At the beginning of the test the system is filled with air. This air is displaced by the carbonization gases during the test. At the end of the test the gas remaining in the train is nearly pure hydrogen. Since this hydrogen is not added to the collected gas, correction is made for it and the air it displaced which is in the collected gas.

(12) *The calorific value of the gas.*—The gross and net B.t.u. values of the gas are computed using the data:

	B.t.u. per cu. ft. Gross	B.t.u. per cu. ft. Net
CH_4	1012	911
CO	323	323
Illuminants	1853	1740
H_2	325	275

(13) *Therms* are calculated on gross and net bases:

Total B.t.u. of the gas per ton of Coal × $1/10^5$, or cu. ft. gas per ton × B.t.u. per cu. ft. × $1/10^5$ = therms per ton.

(14) *The practical yield per ton of coal.*

Gallons of light oil = 2.72 × per cent light oil
Gallons of tar = 2.0 × per cent tar
Pounds of sulphate = 77.6 × per cent total NH_3

(15) *Example*: To illustrate the assay the experimental data for a single test is given herewith:

Carbonization test No. 44
Date: August 17, 1932
Sample: R-24

---

[2] The volumes of gas are exclusive of carbon dioxide and hydrogen sulfide. If it is desired to increase the gas volume by the volumes of these gases it may be done using the following relationships:

Cu. ft. of CO_2 per ton of coal = per cent CO_2 × 170.8
Cu. ft. of H_2S per ton of coal = per cent H_2S × 219.8

CARBONIZATION DATA

	Before test	After test	Difference
Sample (dried at 105° C)	20.0000		
Tar piece	16.6988	17.1385[a]	+ .4397
Distillation tube + sample + packing	114.5668	108.6487	−5.9181
Distillation tube + sample + packing + tar piece	131.2656[a]	125.7872	5.4784
Tar filter	31.7306	32.1259	.3953
Sulphuric acid bulb + drying tube	136.2779	138.0778	1.7999
Potassium hydroxide bulb + drying tube	148.1874	148.7559	.5685
Freezing tube	82.2809	82.5052	.2243

Coke: strongly caked, no swelling.

Average temperature: of coil, 720° C.[b]; of coke, 880° C.

Observations on the gas

Gas volume	6.90 l.
Gas temperature	36.4° C.
Barometric pressure	29.15″ Hg.

Gas Density

Temperature gasometer	29.7°C
Temperature water bath	26.4 °C
Barometric pressure	29.29″ Hg
Weight bulb	102.9540
Weight bulb + gas	103.1939
Volume of bulb	519.74 cc.

Analytical data

Naphthalene

Flask 59 + naphthalene	56.0384 gm.
Flask 59	55.8619
Weight of naphthalene	.1765

Free ammonia		Combined ammonia
0.1196	Normality of acid	0.1196
41.75	Acid neutralized by ammonia	3.10
.0850	Gms. ammonia	.0063

Hydrogen sulfide

Normality of iodine	0.1048	
Iodine equivalent to 1/10 H_2S	3.59	(1/10 titrated)
Gms. H_2S	.0641	

The gas analysis

Gas laboratory number: G-82

	Per cent
CO_2	0.2
O_2	1.0
Illuminants	4.1
CO	10.4
H_2	52.3
N_2	3.2
CH_4	28.8
	100.0

---

[a] Calculated.

[b] Observed temperatures in Centigrade, subsequent gas volumes are corrected to 60° Fahrenheit.

PRELIMINARY RESULTS

Product	Grams	Per cent	Remarks
Coke	14.0819	70.41	
Tar	1.0052	5.03	(includes naphthalene, excludes combined NH_3
Volatile matter	5.9181	29.59	(includes tar)
Gas (fixed)	2.4904	12.45	(after passing absorption train)
Total NH_3	0.193	.46	
Carbon dioxide	.5044	2.52	
Hydrogen sulfide	.0641	.32	
Water	1.5384	7.69	
Light oil	.2243	1.12	

Gas corrected volume, liters	5.87
Gas yield, dry cu. ft. per ton	9392
Gas + CO_2 + H_2S, dry cu. ft. per ton	9893

Standardization of the Assay

Before the examination of the selected coals was started a number of runs were made to standardize the laboratory manipulation.

The first tests were made using Jena glass distillation tubes which required the removal of the open end of the tube at the end of the test. The tube was cut at the end of the first asbestos plug and this 4 to 5 inch section of glass contained the tar deposit corresponding to that found in the tar piece as used in the silica equipment. The Jena tubes were soon replaced by fused quartz tubes and the separate tar piece for the reason that the latter could be used repeatedly while the Jena tubes could be used only once. Furthermore, tar percentages were found to be less difficult to reproduce when using silica tubes.

TABLE II—DATA FOR COMPARATIVE ASSAYS

Products	Illinois Steel Company Per cent	State Geological Survey Average of two tests Per cent
Coke	73.57	73.58
Tar	4.50	4.25
Free ammonia	.29	.31
Combined ammonia	.01	.01
Water	5.49	5.73
Carbon dioxide	1.36	1.45
Hydrogen sulfide	.15	.19
Light oils	1.38	1.24
Gas (by weight difference)	13.25	13.22
Total volatile matter	26.43	26.42
Gas analysis:		
Nitrogen	1.2	.....
Hydrogen	58.0	54.4
Methane	29.1	33.2
Illuminants	4.7	5.2
Carbon monoxide	7.0	7.3
B.t.u. per cu. ft. of gas—gross	593	633
B.t.u. per cu. ft. of gas—net	529	566

Preliminary tests were made using the fused quartz tubes and they were used in all the subsequent by-product analyses.

Through the courtesy of the Illinois Steel Company it was possible to compare the by-product analysis of two laboratories on a single sample of coal. The Central Laboratory of the company supplied the Survey laboratory with a sample previously analyzed by them, and also a complete report of the analysis. The by-product analysis by the Central Laboratory was made by the use of the dry-distillation test method of the chemists of the United States Steel Corporation. Duplicate assays were made by us on this sample and pertinent items of the analyses are compared in Table II.

APPENDIX B

DESCRIPTION OF THE AGDE-DAMM PLASTIC RANGE TEST AS APPLIED TO ILLINOIS COALS

Method

A sample of compressed powdered coal is heated at a constant rate of temperature rise. Linear expansion or contraction of the sample is observed and temperatures are recorded. When plotted the data show the initial softening, active decomposition, and solidification temperatures in the plastic range of the coal. Some exceptions are found as in low rank coals or weathered samples.

Apparatus and Procedure

A cylindrical copper block, 3 inches in diameter and 7 inches long is fitted into an electric furnace built to specification. The copper block is bored with two ½-inch holes which are to receive test tubes with the dimension 9 mm. inside diameter by 75 mm. in length. Above one hole an easily-movable hollow rod is mounted vertically so that it rests on the test specimen. The lower end of the rod is hollow to receive a thermocouple. The weight of the rod is adjusted to one pound and during the test a micrometer distance gage is mounted to bear on the top of the rod. A Brown recording potentiometer pyrometer and an iron-constantin thermocouple are used for temperature measurements.

The auxiliary electrical apparatus consists of two carbon-pile resistances and an A. C. ammeter.

In the prescribed procedure[1] two test specimens are prepared, one of which is placed under the one-pound rod and the other is allowed to expand or contract freely in the second receptacle of the copper block.

A sample of 1 gram of 100-mesh coal is compressed in the small glass test tube under a weight of 5.8 kilos. The length of the briquet is recorded and the specimen is placed in the copper block at room temperature. The one-pound rod is placed so that it rests on the coal, and the thermocouple is adjusted to fit into the bottom of the rod. The distance gage is mounted with the pointer at an intermediate position on the scale.

The furnace is heated at a rate of 4.2° C. per minute and heating is terminated at 500°C. Temperature, distance, and time are recorded. The data are plotted with temperatures as abscissae and distance readings as ordinates.

Figure 34 (p. 90) shows the furnace, with distance gage in position, as well as auxiliary apparatus.

Corrections and Interpretation

Since the temperatures recorded are those for a position immediately above the coal it is necessary to correct the temperature data. To determine the amount of the correction

---

[1] Procedure as described in the U. S. Bureau of Mines Bulletin 344, p. 16, 1931.

two thermocouples were mounted in the furnace: one, in the prescribed position in the plunger-rod, and the second inserted directly into a compressed sample of the coal. Temperatures in the coal were higher than the temperatures recorded and corrections were accordingly applied.

Other corrections in connection with the differential expansion of the iron parts of the apparatus were not applied since the small uniform tendencies involved do not interfere with the interpretation of the data. This is true because the test is not concerned with the absolute expansion of the coal but rather with the temperatures at which the changes take place.

When the distance versus temperature data are plotted the following temperatures may be read from the curve:

(1) The *initial softening temperature* is the temperature at which the sample starts to contract due to fusion and softening of the coal particles

(2) The *initial active decomposition temperature* is the temperature at which active rapid decomposition of the coal commences, and this is indicated by the sudden expansion of the coal. In the case of some samples the expansion does not occur. If the particles of coal do not coalesce, the gases escape without causing swelling

(3) The *solidification temperature* is the point at which expansion ceases abruptly and at which the material sets into a rigid cellular mass

(4) The *plastic range* is the temperature interval between the initial softening temperature and the solidification point

Shrinkage of the solidified sample is also indicated by the curve.

APPENDIX C

THE TEST FOR AGGLUTINATING VALUE AS APPLIED TO ILLINOIS COALS

Method

The apparatus and procedure used in the test closely adhere to those proposed by the American Society for Testing Materials.[1] A sample of 200-mesh coal is mixed intimately with sized Ottawa sand[2] in the proportion of 14 parts of sand to 1 part of coal and carbonized at 950°C. for 20 minutes. The coke-sand button thus obtained is crushed in a compression machine which applies the load at a constant rate, and the crushing strength of the button is determined. Six buttons are tested for one sand-coal ratio and for each sample of coal. If one or more buttons have crushing strengths deviating more than 10 per cent from the average a new set of buttons is prepared.

Apparatus

(a) *Furnace.*—The furnace used in the standard volatile matter determination is used to carbonize the coal-sand buttons. It is regulated to maintain 950°C. in the crucible zone, and a calibrated chromel-alumel thermocouple is used to measure the temperature.

(b) *Compression machine.*—A Scott testing machine[3] ordinarily used for tensile tests of rubber or fabrics was adapted to the work by the use of a cage designed to provide compression. A constant-speed motor and reversible gear arrangement gave a constant rate of compression of one inch per minute, and the crushing strength of the specimen was observed in pounds. Machines designed specifically for the agglutination test are on the market.

(c) *Sieves.*—

(1) For the preparation of the coal: U. S. Standard Series sieve No. 200 (74 micron opening).

(2) For the preparation of the sand: U. S. Standard Series sieve No. 45 and No. 60 (350 micron and 250 micron respectively).

(d) *Porcelain crucibles.*—

(1) For moistening sand, and for mixing coal and sand: Coors high-form crucible, size 2: 52 mm. top diameter; 25 mm. bottom diameter; 43 mm. height; 55 ml. capacity.

---

[1] Proposed Draft, June 1934. Amer. Soc. Test. Material, Standards on Coal and Coke, pp. 89-94, Sept. 1936.

[2] Varying results are frequently obtained from duplicate tests using different batches of sand. This is no doubt due to the fact that the surface of the sand grains has much to do with the values obtained, and apparently no two batches are identical. Recently an attempt has been made to substitute carborundum as the inert material for this determination. Although it holds some promise, the difficulties have not yet been overcome.

[3] Henry L. Scott and Co., Providence, R. I.

(2) For carbonizing the coal-sand mixtures: Coors cylindrical crucible (with covers) No. 390: 28 mm. inside diameter; 30 mm. inside height; 18 ml. capacity. These crucibles must have circular cross-section, and must be free from irregularities on the inner surface. If a lip is on the inside of the rim it may be removed by grinding.

Covers are used. Since their outside diameter is nearly equal to that of the tube furnace two notches are ground in the sides of the cover where it comes in contact with the wires supporting the crucible.

(e) *Compressing device.*—The coal-sand mixture is compressed in the cylindrical crucible by a weight of 3.5 kg., and the apparatus was made in the laboratory. An easily movable piston with a machined face fits snugly, without binding, into the cylindrical crucibles, and the piston is weighted to make a total load of 3.5 kg.

Procedure

(a) *Calibration of the furnace.*—The temperature of the standard volatile matter furnace is controlled by means of a rheostat, and the temperature in the furnace is measured by a thermocouple installed permanently through the bottom of the furnace. However, the equipment is tested to ascertain the temperatures attained inside the crucible. A cylindrical crucible is filled with sand and a hole is ground in the center of a porcelain cover to admit a thermocouple. The crucible and sand are placed in position in the furnace and the standardizing thermocouple is inserted through the hole in the cover. The fused junction is fixed at the geometric center of the sand and crucible and the furnace rheostat is adjusted until the prescribed temperature of 950°C. is maintained inside the crucible. At this setting the temperature read on the permanent couple is recorded and used subsequently since it corresponds to the desired temperature in the crucible.

(b) *Preparation of the sand.*—The sand used for mixing the sand-coal mixtures is natural silica sand obtained from Ottawa, Illinois.[4] It is carefully graded to —45 +60 in particle size and this portion is washed with water. The turbid water is decanted and the washing is repeated 5 times, after which it is boiled in dilute HCl (1:1) for 30 minutes. It is then washed thoroughly (until free from chloride) and dried.

(c) *The sand-coal mixture.*—The fresh coal is ground to 200-mesh, and the weighed sample is mixed with the weighed portion of sand which previously has been moistened with glycerine. The 14:1 sand-coal ratio was used in the work on Illinois coals but other ratios, which have been tested, were as follows:

Ratio sand to coal	Weight of sand, gm.	Weight of coal, gm.
10:1	18.182	1.818
12:1	18.462	1.538
14:1	18.667	1.333
15:1	18.750	1.250
20:1	19.048	0.952
25:1	19.231	0.769

The sand is weighed into the high-form No. 2 crucible and moistened with a drop of glycerine weighing 0.07 gm.[5] The sand is thoroughly mixed with a spatula for 1 minute. The weighed portion of coal is added to the crucible and mixed into the sand for 2 minutes.

---

[4] Ottawa Silica Company, Ottawa, Illinois.

The mixture is carefully transferred into the cylindrical crucible and the top is leveled by means of a square-end spatula. It is compressed for 30 seconds under a load of 3.5 kg. by means of the compression device.

After compression the empty space at the top of the crucible is filled with sand-coke mixture from buttons previously crushed. With the cover in place the button is carbonized at 950° ± 10° C. for exactly 20 minutes. It is allowed to cool in air, and then it is removed by inverting the crucible. If the faces of the button have irregularities they are removed by the use of No. 00 sandpaper.

The prepared button is placed in the center of the ⅜-inch rubber pad on the bottom of the crushing cage and the crushing strength is determined.

---

[5] The drop of glycerine weighing .07 gm. is obtained from a suitable burette, or by use of a heavy-wall capillary tube ground plane on the end. With additional grinding to reduce the area of the plane face the weight of the drop may be reduced.

Index