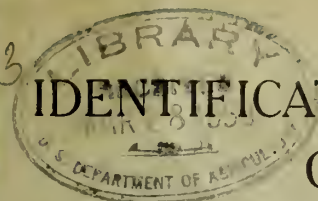


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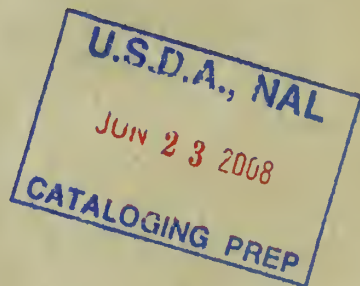
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IDENTIFICATION AND OCCURRENCE
OF SULPHIDES
ON LAND STRIPPED FOR COAL

BY
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FOREST EXPERIMENT STATION

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IN COOPERATION WITH THE
CHEMISTRY DEPARTMENT
SOUTHERN ILLINOIS UNIVERSITY

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4
IDENTIFICATION AND OCCURRENCE OF SULPHIDES

ON LAND STRIPPED FOR COAL †

by

Glenn H Deitschman^{1/} and J. W Neckers^{2/}

Whether or not strip-mined lands are suitable for plant growth depends largely upon the characteristics of earth strata that formerly overlaid the coal. Overburden composition varies greatly by localities and coal seams. Similar variations are often reflected in the composition and quality of spoil-bank surface materials. The principal bank characteristics affected are acidity, texture, amount of soil-sized material,^{3/} and mineral content. The important influence of these characteristics upon the success of reclamation programs has been pointed out in recent reports (2, 3, 4, 5, 7).^{4/}

Upon exposure to weathering, materials on the bank surfaces usually undergo rapid physical and chemical changes. In addition to increasing the "soil" percentage by disintegrating larger fragments, weathering releases chemical compounds that may have an important effect upon acidity and upon the availability of plant nutrients. In most instances, the exposed materials remain alkaline or low in acidity and the site quality is improved. However, where highly sulphurous strata are deposited upon the tops of the banks, oxidation of the sulphur compounds (pyrite, marcasite, and complex polysulphides) to sulphuric acid may eventually create conditions that are toxic or injurious to plant life. This occurs to some extent on nearly all strip-mined land, and occasionally entire areas are rendered unproductive for many years. As pH tests^{5/} do not measure potential acidity, pre-planting examinations made soon after mining have not always been reliable guides for successful reclamation work.

A practical field method for determining the approximate concentration of sulphides in overburden materials has been developed. This test should be of value in obtaining early and more complete information of the future acidities of fresh banks as a basis for preparing reclamation plans. Also, knowledge of the types of

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^{2/} Professor of chemistry, Southern Illinois University.

^{3/} Particles measuring less than 2 mm in diameter.

^{4/} Numbers in parentheses refer to Literature Cited, p. 9.

^{5/} The pH scale of acidity ranges numerically from 0 to 14. A pH below 7.0 is acid and above 7.0 is alkaline. Materials testing less than pH 4.0 are generally toxic to most plants.

materials causing acid-toxicity might enable mine operators to bury them in the banks with only slight modifications of usual stripping techniques.

A number of strip-mine highwalls at various locations in Illinois and Indiana were examined in a preliminary study to find out if occurrence of sulphur compounds was typical of a given coal seam or locality. The results of this survey are presented in a later section.

FIELD TEST FOR ACTIVE SULPHIDES

In past investigations of coal seam overburdens, a sodium azide test has occasionally been used for sulphide detection. Application of the sodium azide solution to a small piece of sulphurous material causes the evolution of tiny nitrogen bubbles, visible with a hand lens. As this method offers little opportunity for obtaining a quantitative estimate of sulphur content, a more informative test was recently adapted for field use (6).

Materials

A field kit for this test should include the following equipment:

Geologist's pick (for obtaining unweathered rock samples)
Wide-nosed pliers (for crushing the samples)
12 test tubes (20 x 150 mm.)
1/4 lb. of granular C.P. zinc (20-30 mesh)
Spiral of wire (a bent paper clip will do)
4-oz. bottle of 6 N. hydrochloric acid (with eye-dropper)
Box of filter paper (Whatman No. 1, 5.5 cm.)
4-oz. bottle of 0.6 N. lead acetate solution (with eye-dropper)

Procedure

Crush 2-3 grams of the material to be tested and place in a test tube. Add about 1 gram of zinc and mix. Insert the spiral of wire part way down the tube to disperse bubbles that otherwise might rise to the top of the tube and spoil the test by contact with the test paper. Add 2-3 ml (1 eye-dropperful) of hydrochloric acid. Wait about 5 seconds for the fumes to displace the air in the test tube and then cover the mouth of the tube with a small filter paper freshly moistened with 2 or 3 drops of lead acetate solution. Remove the filter paper after 5 seconds. The color and appearance of the deposit produced on the filter paper indicate the amount of potentially active sulphide in the sample, as illustrated in figure 1 and described below.

<u>Color</u>	<u>Sulphide concentration</u>
Slight tan coloration	Very low
Tan	Low
Brown with tan margin	Moderate
Dark brown	High
Black with silvery cast	Very high

Verification of the Field Test

To determine the validity of this test, laboratory analyses for sulphur content were made on several fresh samples of overburden materials. The samples were taken from strata overlying No. 6 ("Harrisburg") coal at a strip-mining operation of the Sahara Coal Company in Saline County, Illinois. By the field tests, sulphides were detected in the lower two-thirds of the overburden. The degree of concentration tended to increase with depth, abruptly becoming very high in the sandy shale stratum immediately above the rider vein.

Samples of five of the strata were analyzed in the laboratory by the Eshka method (1). The content of total sulphur was found to range from 0.06 percent to more than 2 percent, and the results showed good correlation with the field determinations (table 1).

(Although the actual initial acidity of the overburden materials averaged about pH 6.0, ^{6/} acid-toxic spots will undoubtedly develop on the mine banks where highly sulphurous sandy shale and rider coal are exposed. In nearby experimental forest plantings on banks derived earlier from the same stripping operation, 8 percent of the seedlings died from acid-toxicity after two growing seasons and an additional 4 percent were seriously injured by high, but nonlethal, acidity.)

^{6/} The acidities reported in this paper were measured by a colorimetric method ("Soiltex") to the nearest 0.5 pH

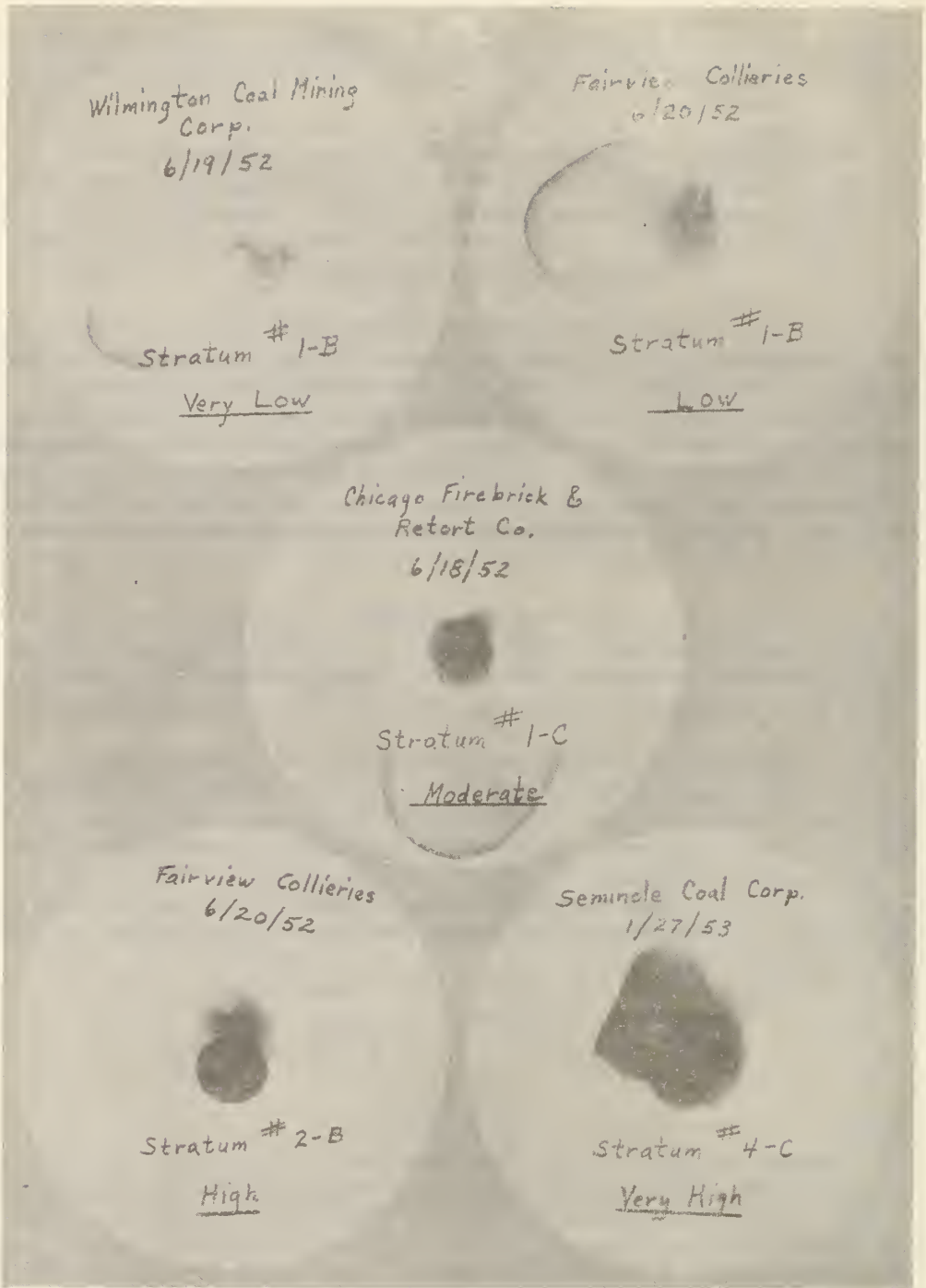


Figure 1.--Test filter papers showing the relative coloration produced by very low to very high sulphide concentrations. The margin of the filter paper disc can also be used to record pertinent field notes, as shown.

Table 1.--Descriptions and analyses of strata overlying No. 6 coal,
Saline County, Illinois

Description of strata	Thickness Feet	Acidity pH	Average sulphur content	
			Field	Laboratory
Silty clay loam, gray- brown	1	4.5	None	-
Silty clay, mottled red-brown	10	6.0	None	-
Sandstone, coarse- grained, yellow or red	3	6.0	None	-
Sandstone, silty, light tan	6	6.0	Very low	0.06
Shale, light gray, thin-bedded	3	5.5	Low	0.11
Shale, gray, thick- bedded	4	5.5	Moderate	0.20
Sandy shale, light gray, thin-bedded	2	5.0	Moderate	0.22
Sandy shale, dark gray, thin-bedded with black laminations	10	5.5	Very high	2.33
Impure coal	1	6.0	Very high	-
Limestone, dark gray, massive	5	7+	Moderate	-
No. 6 coal seam	-	-	-	-

OCCURRENCE OF ACTIVE SULPHIDES

Examinations were made of materials overlying extensively strip-mined coal seams in Illinois and Indiana to explore the general characteristics of sulphide occurrence. The survey included 23 currently active stripping operations in Illinois and 32 in Indiana (figs 2 and 3) ^{7/}

At each mine, the rock strata in the overburden were described as to (a) type and thickness, (b) approximate acidity, and (c) sulphide concentration as shown by the field test described above. Fresh unweathered samples for the tests usually were taken from the midpoint of each stratum. In very thick beds, or those which appeared to vary somewhat in color, texture, or composition, two or more samples were obtained at different depths.

Descriptions of overburden characteristics of each Illinois and Indiana coal seam investigated are summarized in the Appendix, together with charts illustrating diagrammatically the composition of the individual highwalls. The upper layers of dissociated earth material--soil, loess, glacial drift, or decomposed rock--often greatly influence stripped-land quality but are not described here. They contain very little sulphur and vary principally by topography and locality rather than by different coal seams.

Illinois Coal Seam Overburdens

Rock strata above the five coal seams presently being strip-mined in Illinois consisted largely of calcareous shales and limestones. In these, the amount of sulphides usually was greatest in dark-colored, carbonaceous materials and in strata lying nearest the coal. However, the degree of sulphur concentration and its distribution in the overburden varied with coal seams and between different parts of the State. According to the highwalls sampled, an especially high proportion of potentially acid strata overlies No. 2 coal in LaSalle County and No. 5 coal in practically all localities.

Indiana Coal Seam Overburdens

The materials overlying the seven major strip-mined coal seams in Indiana differ markedly from those found in Illinois. The Indiana overburdens are usually more complex and, consequently, vary more in composition within short distances. In general, the materials are much more acid and frequently include beds of sandstone, sandy shale, and rider coal which are relatively high in sulphur. Each of the coal seams and localities listed below contain high sulphide concentrations in about a third or more of the total depth of the overburdens sampled.

^{7/} Names of the coal companies and dates of examination are listed in the Appendix.

Block Coal	- Northern Clay County
Minshall Coal	- Fountain County
Coal III	- Southern Clay County
Coal IV	- Daviess and Pike Counties
Coal V	- Vermillion, Pike, and Warrick Counties
Coal VI	- All areas sampled except one in southern Vermillion County
Coal VII	- Sullivan County

ACIDITY OF STRIP-MINED LAND

Predictions of the future acidity of many fresh banks can only be rough estimates because detailed information is lacking on the complex relationships among sulphur content, initial acidity, and physical composition of the materials. From general observations, it is apparent that the neutralizing capacity of highly calcareous material such as limestone and fossiliferous shales may partly or completely offset the acidity developed by sulphide oxidation. Also, the high base exchange properties of material composed largely of fine clay particles act as a buffer against high acidity. On the other hand, the coarser-grained sandstones or sandy shales which have a lower buffering capacity may become acid quite readily, even if initially alkaline.

Because of variations in stripping methods, the concentration of sulphide in the overburden usually does not accurately indicate how much potentially acid-toxic material will cover the mine-bank surfaces. For this reason, there is little consistency in the occurrence of acid-toxic banks as related to a given coal seam, even within a small district. Therefore, each strip-mined area must be examined individually to determine its capabilities for plant growth.

On a broad scale, the effects of widely differing overburdens upon acidity of stripped land can be illustrated by comparing conditions in Illinois and Indiana. In addition to the abundance of calcareous shale and limestone overlying the Illinois coals, nearly all of this State is covered with a fairly thick mantle of calcareous loess and glacial till. The Indiana coal seams, however, are commonly overlaid by relatively acid and more sulphurous beds of sandstone, sandy shale, or rider coal. Extensive, deep loessal deposits are mostly absent in the Indiana coal-producing region. Much of the strip-mining has been done in the southern end of the State where calcareous glacial drift is not present to modify the high acidities of other strata. As a result of these general contrasts in overburden characteristics, extensive surveys of strip-mined land have shown that nearly 80 percent of such areas in Illinois are predominantly calcareous, whereas in Indiana about 80 percent are judged to be predominantly acid (4).

SUMMARY

Tests of acidity on freshly stripped mine banks may be misleading. On some of these areas, weathering of highly sulphurous material causes subsequent development of acid-toxic conditions. A field test for estimating the concentration of sulphides in overburden materials is reported. Results obtained by the test have shown a good correlation with laboratory analyses of total sulphur content.

Using this field test, examinations were made of 55 highwalls in Illinois and Indiana. In general, sulphide concentrations were found to be highest in dark-colored, carbonaceous material and in strata lying near coal seams. The degree and extent of these concentrations, as well as types of overburden material, varied greatly among different coal seams and mining localities. Predictions of mine-bank acidity cannot be based upon sulphide tests alone. Consideration must also be given to the influence of initial pH and physical properties of the bank materials. Furthermore, the extent to which acid-producing strata are exposed on the mine-bank surfaces depends upon the proportion of such materials in the overburden and upon the methods of strip-mining employed.

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APPENDIX

Materials overlying the coal seams in Illinois and Indiana are briefly described here as to the general types of materials present, their acidity, and sulphide content. More specific details of highwall composition are shown in the accompanying charts. Approximate locations of the highwalls studied are shown in figure 2. For anyone engaged in rehabilitating coal-stripped lands, this information should be helpful in pointing out the localities, coal seams, and strata where concentrations of potentially acid-toxic material have been found. It may also suggest practical ways of modifying stripping methods in certain areas to keep the injurious strata from being left exposed on the mine-bank surface when mining is completed.

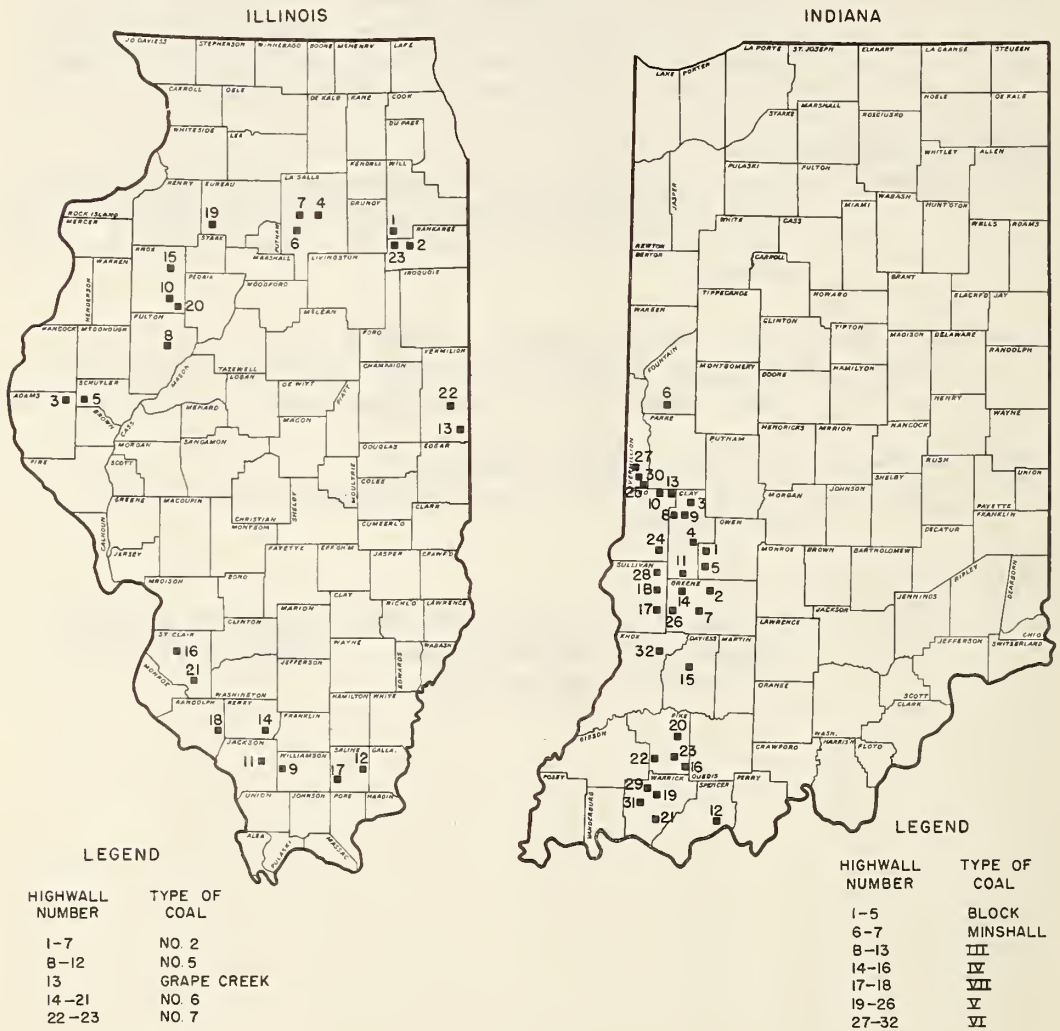
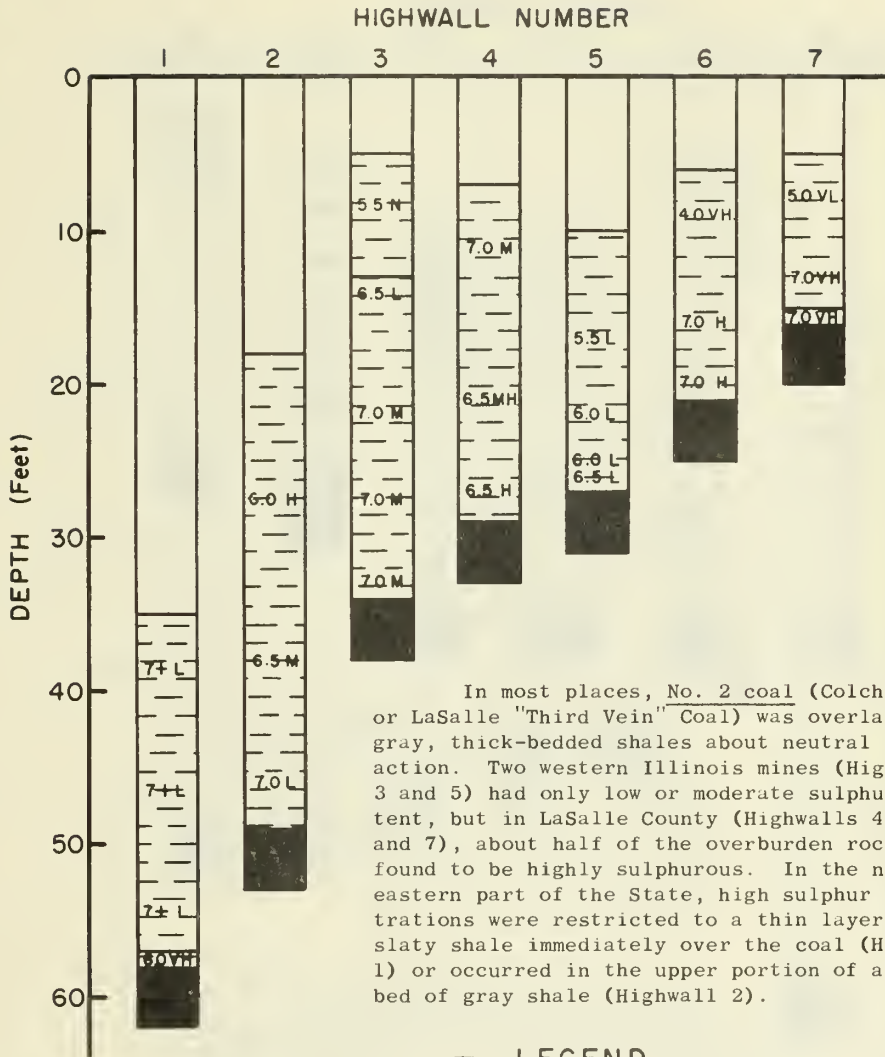
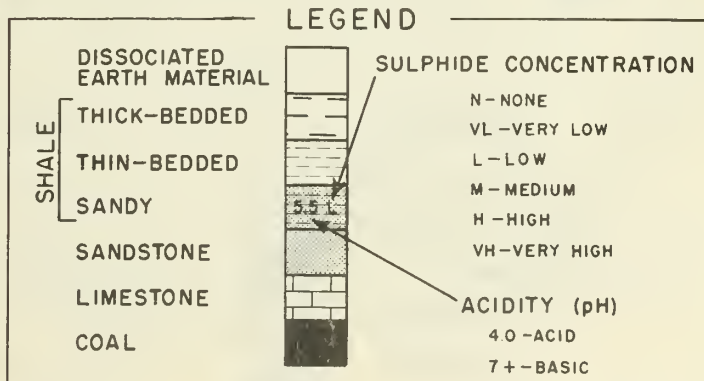


Figure 2 --Locations of highwalls

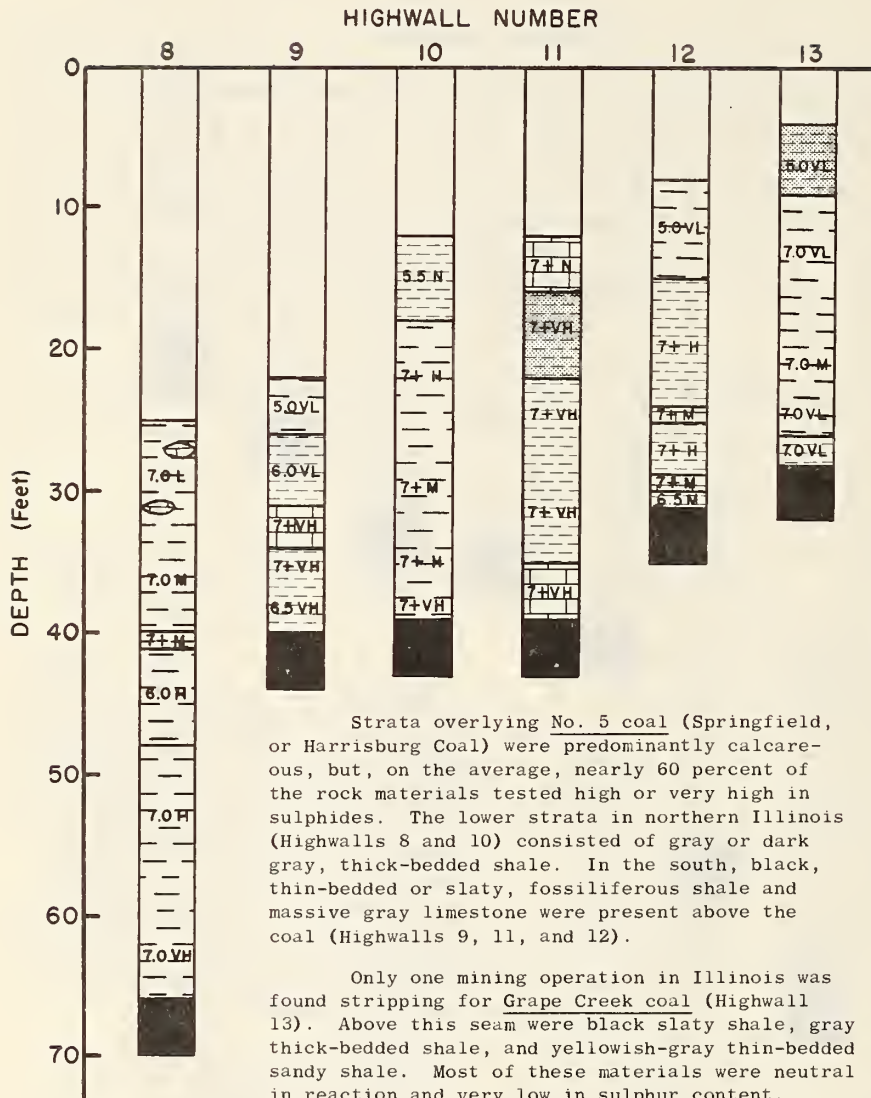
ILLINOIS COAL SEAM NO. 2



In most places, No. 2 coal (Colchester, or LaSalle "Third Vein" Coal) was overlaid by gray, thick-bedded shales about neutral in reaction. Two western Illinois mines (Highwalls 3 and 5) had only low or moderate sulphur content, but in LaSalle County (Highwalls 4, 6, and 7), about half of the overburden rock was found to be highly sulphurous. In the northeastern part of the State, high sulphur concentrations were restricted to a thin layer of black slaty shale immediately over the coal (Highwall 1) or occurred in the upper portion of a thick bed of gray shale (Highwall 2).

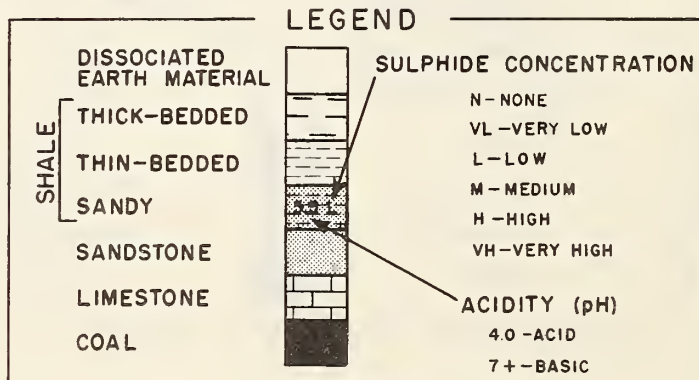


ILLINOIS COAL SEAMS NO. 5 AND GRAPE CREEK

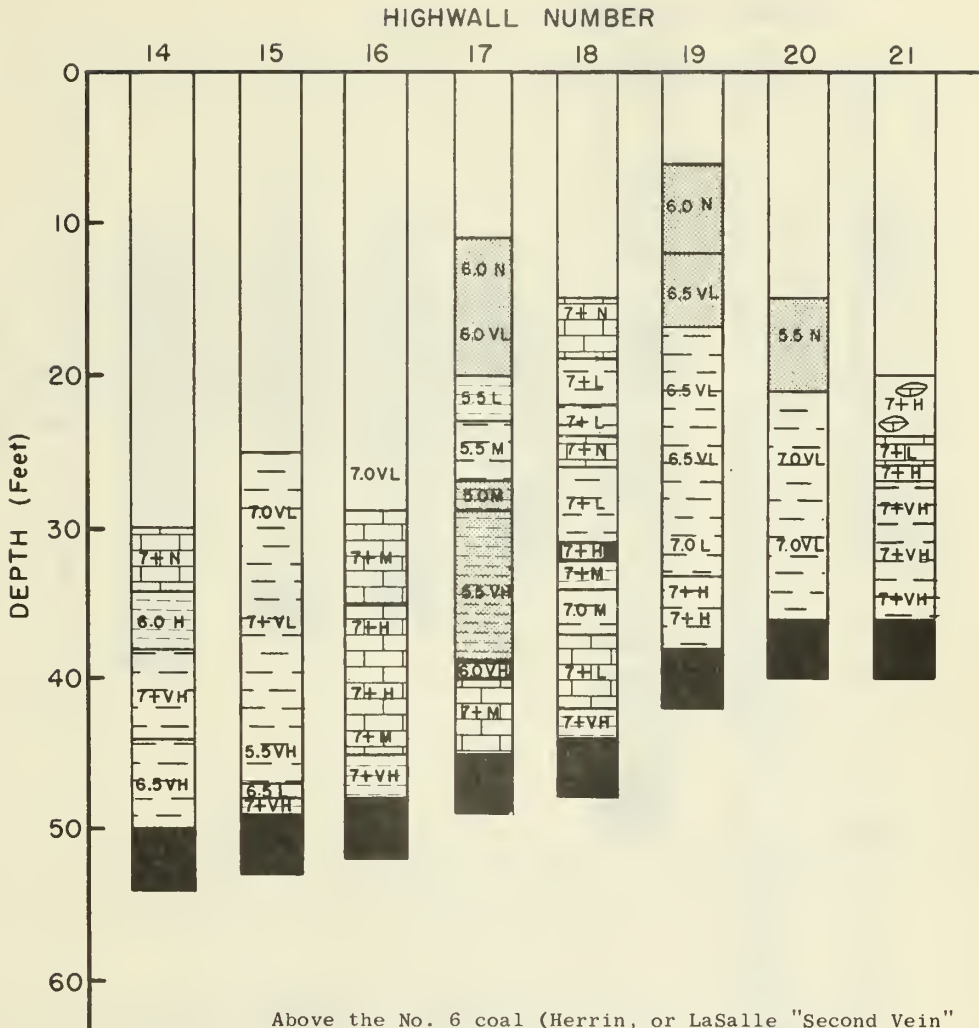


Strata overlying No. 5 coal (Springfield, or Harrisburg Coal) were predominantly calcareous, but, on the average, nearly 60 percent of the rock materials tested high or very high in sulphides. The lower strata in northern Illinois (Highwalls 8 and 10) consisted of gray or dark gray, thick-bedded shale. In the south, black, thin-bedded or slaty, fossiliferous shale and massive gray limestone were present above the coal (Highwalls 9, 11, and 12).

Only one mining operation in Illinois was found stripping for Grape Creek coal (Highwall 13). Above this seam were black slaty shale, gray thick-bedded shale, and yellowish-gray thin-bedded sandy shale. Most of these materials were neutral in reaction and very low in sulphur content.

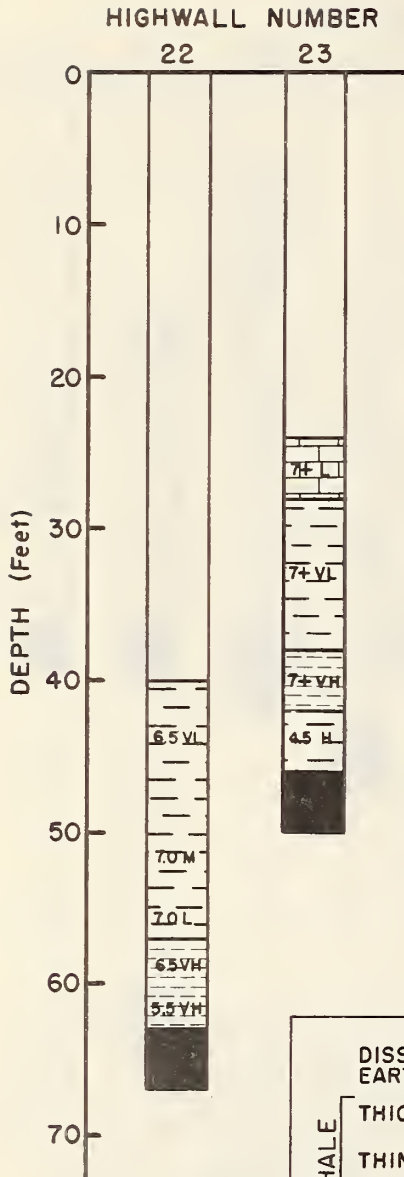


ILLINOIS COAL SEAM NO. 6

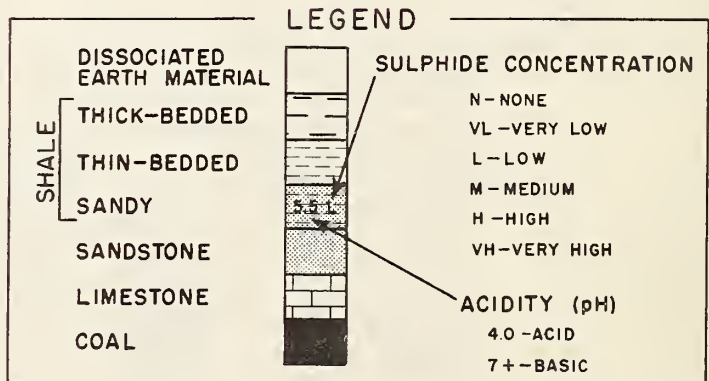


Above the No. 6 coal (Herrin, or LaSalle "Second Vein" Coal) the materials were mainly slightly acid to calcareous. In the northwest section of the State (Highwalls 15, 19, and 20), an average of three-fourths of the rock strata consisted of gray thick-bedded shales and brown or gray sandstones which were low in sulphides. At two mines (Highwalls 15 and 19), the coal was overlaid by about 5 feet of highly sulphurous black shale. The southern Illinois overburdens (Highwalls 14, 16, 17, 18, and 21) usually contained thick strata of dark-colored massive limestone and calcareous shales. A layer of impure coal, thought to be the No. 7 seam, appeared in two of the highwalls. High sulphide concentrations occurred in more than half the rock materials in the average overburden and were not confined to the lowermost strata as in the northwest section of the State.

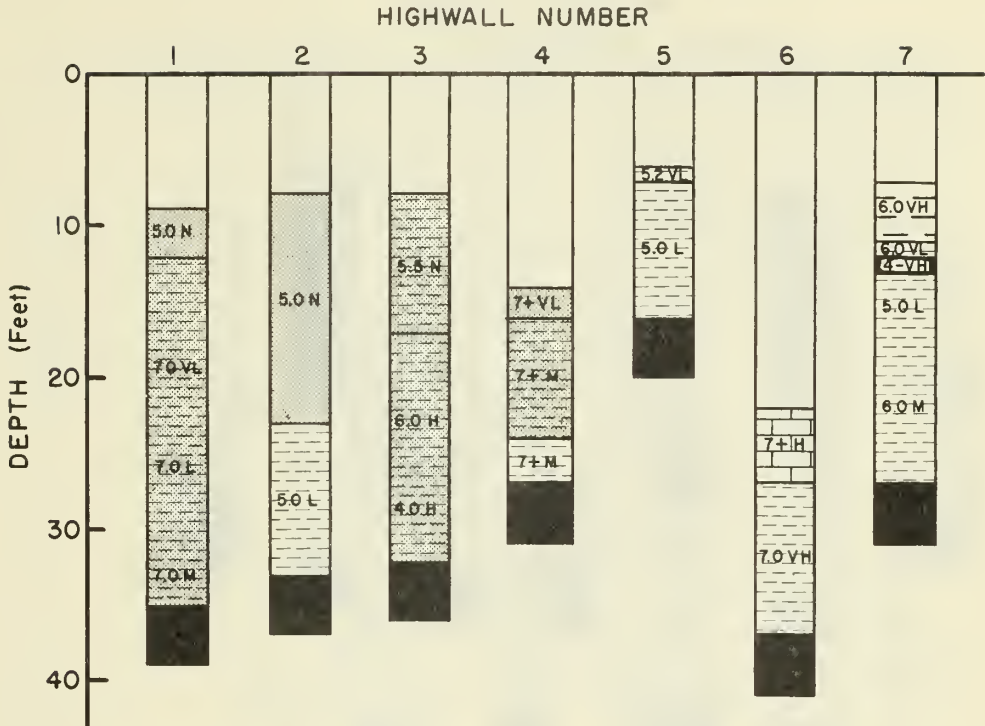
ILLINOIS COAL SEAM NO. 7



The rock materials overlying No. 7 coal (Danville, or LaSalle "First Vein" Coal) at the two mines examined consisted principally of black or gray, thick- to thin-bedded shales. These tended to become acid and highly sulphurous in the lower 6 or 8 feet.



INDIANA COAL SEAMS BLOCK AND MINSHALL



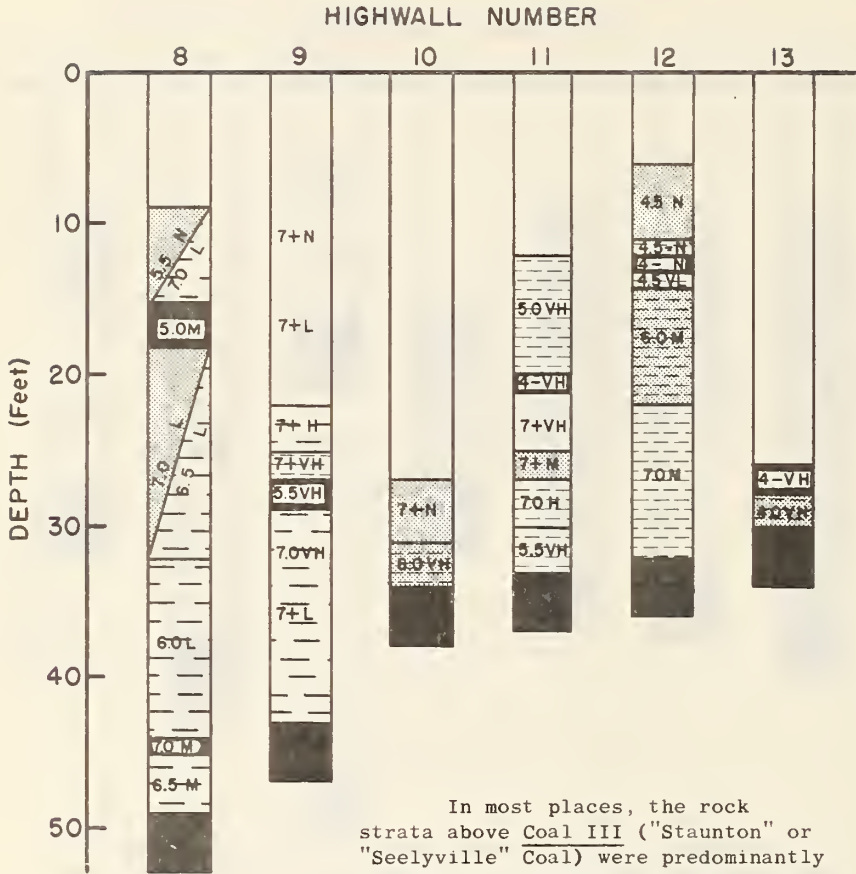
Two coal seams, Upper and Lower Block, are combined here under the single name of "Block Coals" because, where the Upper Block is of strippable thickness, it is commonly mined in conjunction with the Lower Block seam.

Rock material above the Block Coals consisted of black slaty shale, and massive sandstone. The initial acidity of these strata varied widely from place to place, but except for one case (Highwall 3), only moderate or low sulphide content was noted.

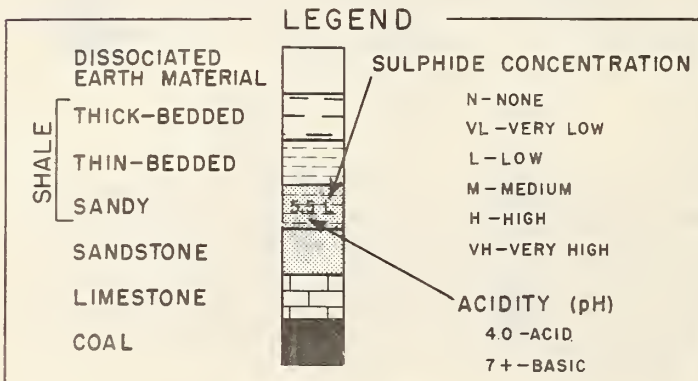
Examinations of the overburden of the Minshall vein (Highwalls 6 and 7) were made at two widely separated locations. In Fountain County, the coal was overlaid by calcareous dark gray slaty shale and massive limestone, both of which contained high sulphide concentrations (Highwall 6). Farther to the south, the materials were more acid and sulphides were most abundant in and above the overlying Block Coal seam (Highwall 7).

INDIANA COAL SEAM

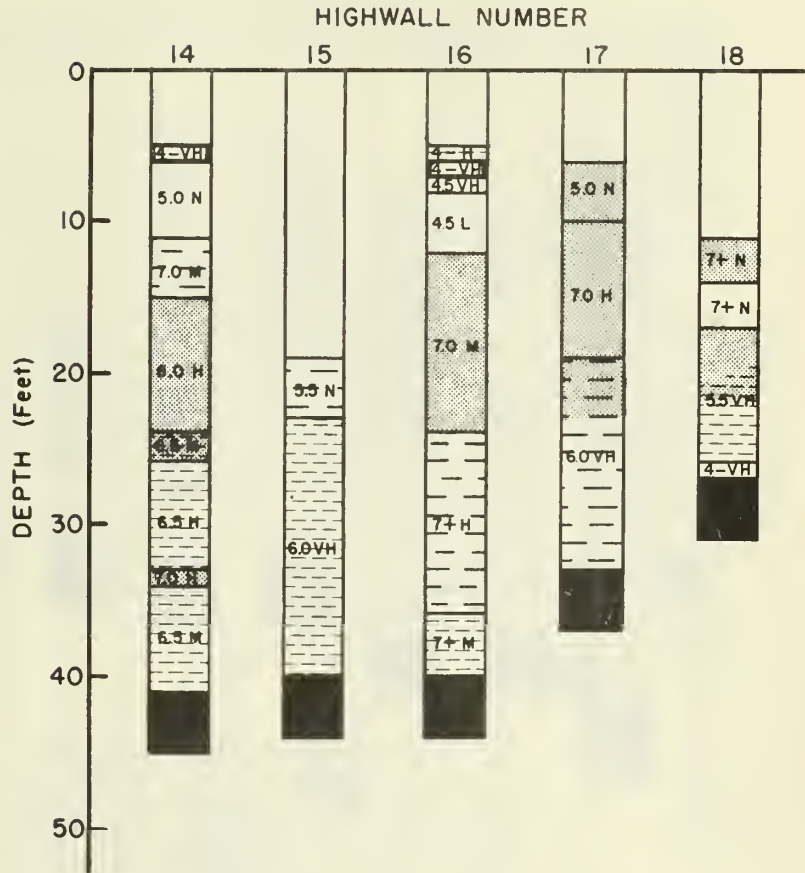
III



In most places, the rock strata above Coal III ("Staunton" or "Seelyville" Coal) were predominantly acid. High concentrations of sulphides most commonly occurred in a bed of laminated sandy shale immediately above the coal and in the vicinity of a rider vein, Coal IIIa.



INDIANA COAL SEAMS IV AND VII

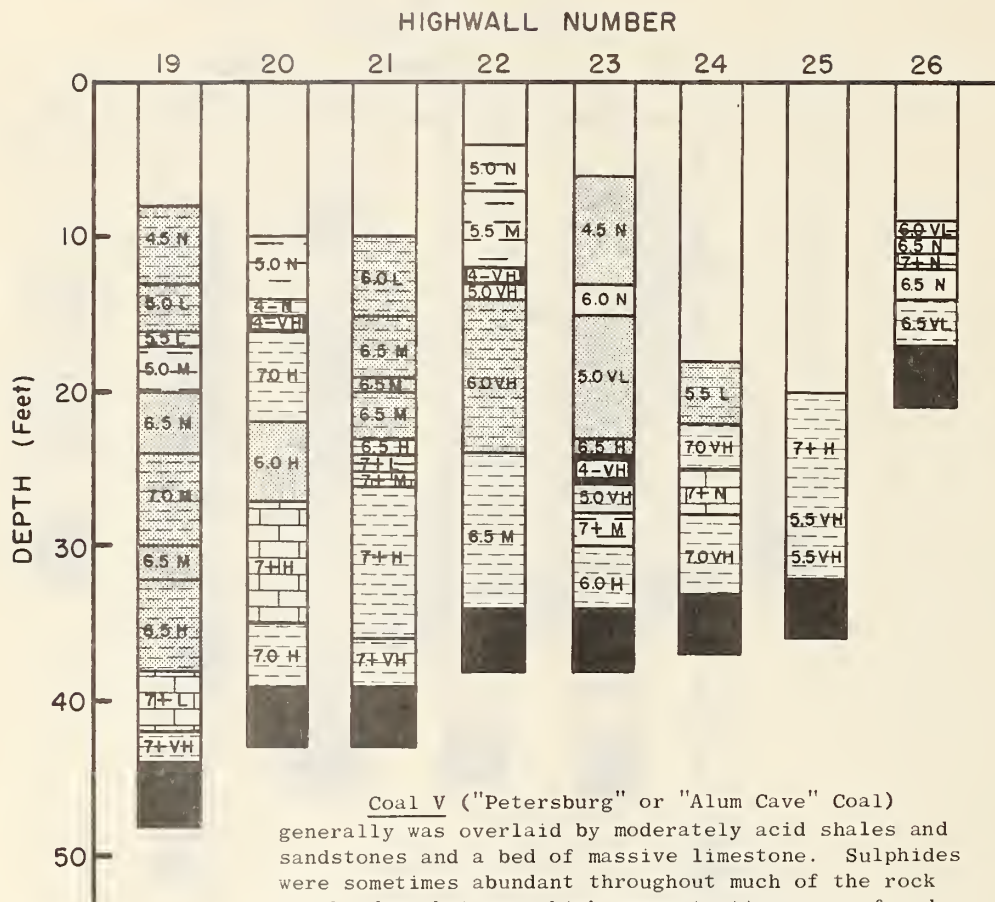


Above Coal IV ("Linton Block" Coal) (Highwalls 14, 15, and 16) most of the strata were moderately acid and contained moderate to high concentrations of sulphur. Especially high concentrations were found in the rider vein, Coal IVa, and in a thick bed of black slaty shale immediately above Coal IV in Daviess County (Highwall 15).

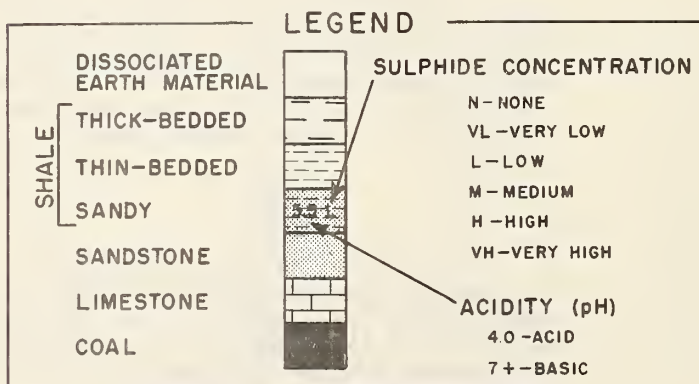
The shale and sandstone above Coal VII (Highwalls 17 and 18) tended to be slightly acid with very high sulphur concentrations in the lower half of the overburden. At one mine (Highwall 18), an extremely acid bed of sulphurous gray clay, about a foot thick, rested directly upon the coal.

INDIANA COAL SEAM

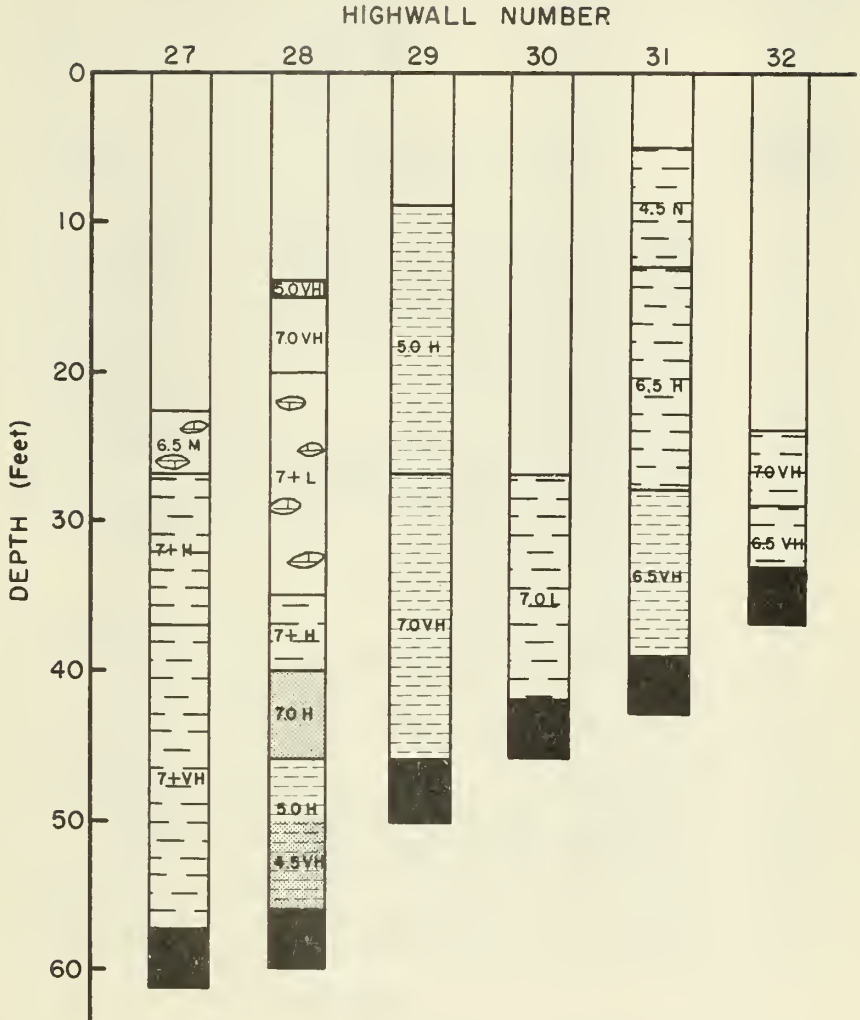
V



Coal V ("Petersburg" or "Alum Cave" Coal) generally was overlaid by moderately acid shales and sandstones and a bed of massive limestone. Sulphides were sometimes abundant throughout much of the rock overburden, but very high concentrations were found most frequently in the lowermost stratum of black slaty shale, and in and near the rider vein, Coal Va.



INDIANA COAL SEAM VI



The strata above Coal VI consisted largely of calcareous or slightly acid shales which were high in sulphur. At one location in Sullivan County (Highwall 28), sandy shale and sandstone replaced the shale in the lower portion of the overburden. Coal VII, which also appeared in this highwall, was underlaid by highly sulphurous white fire clay.

List of Companies Participating

The highwall descriptions and analyses reported in this paper were obtained from points near the active operations of the following mines on the dates indicated.

Illinois

<u>Highwall Number</u>	<u>Mining Company</u>	<u>County</u>	<u>Date</u>
1	Wilmington Coal Mining Corp.	Will	6-19-52
2	Northern Illinois Coal Corp.	Kankakee	6-19-52
3	Triple S Coal Co.	Adams	6-16-52
4	Chicago Firebrick & Retort Co.	LaSalle	6-18-52
5	H. C. Green Mine	Schuyler	6-16-52
6	Monmouth Stone Co.	LaSalle	6-18-52
7	Donovan Mine	LaSalle	6-18-52
8	United Electric Coal Co.	Fulton	6-16-52
9	Forsyth-Williamson Coal Co.	Williamson	8- 5-52
10	Midland Electric Coal Corp.	Knox	6-17-52
11	Truax-Traer Coal Co.	Jackson	8- 5-52
12	Southern Construction & Engineering Co.	Saline	6- 6-52
13	Grape Creek Coal Co.	Vermilion	6-20-52
14	United Electric Coal Co.	Perry	6- 5-52
15	Little John Coal Co.	Knox	6-17-52
16	Midwest Radiant Corp.	St. Clair	1-27-53
17	Sahara Coal Co.	Saline	11- 6-51
18	Southwestern Illinois Coal Corp.	Randolph	6- 5-52
19	Midland Electric Coal Corp.	Bureau	6-17-52
20	Midland Electric Coal Corp.	Knox	6-17-52
21	Seminole Coal Corp.	St. Clair	1-27-53
22	Fairview Collieries Corp.	Vermilion	6-20-52
23	Northern Illinois Coal Corp.	Kankakee	6-19-52

Indiana

1	Schaumecker Coal Co.	Owen	5-23-51
2	--	Greene	5-25-51
3	Quality Coal Corp.	Clay	5-23-51
4	Brown Coal Co.	Clay	5-23-51
5	Rohr and Son	Owen	5-25-51
6	Morgan Coal Co.	Fountain	5-22-51
7	Yake Coal Corp.	Greene	5-26-51
8	Ayrshire Collieries Corp.	Clay	5-23-51
9	Ayrshire Collieries Corp.	Clay	5-23-51
10	Lone Star Coal Co.	Vigo	5-21-51
11	Siepmann Coal Co.	Clay	5-24-51

12	Ohio Valley Co.	Spencer	5-28-51
13	Lone Star Coal Co.	Vigo	5-21-51
14	Maumee Collieries Co.	Greene	5-25-51
15	Central Indiana Coal Co.	Daviess	5-26-51
16	Ayrshire Collieries Corp.	Pike	5-28-51
17	Sherwood-Templeton Coal Co.	Sullivan	5-24-51
18	Maumee Collieries Co.	Sullivan	5-24-51
19	Wasson Coal Mining Corp.	Warrick	5-29-51
20	Ayrshire Collieries Corp.	Pike	5-27-51
21	Sunlight Coal Co.	Warrick	5-29-51
22	Enos Coal Mining Co.	Pike	5-28-51
23	Ayrshire Collieries Corp.	Pike	5-28-51
24	Maumee Collieries Co.	Vigo	5-24-51
25	Standard Collieries, Inc.	Vermillion	5-22-51
26	ABC Coal Co.	Greene	5-25-51
27	Ayrshire Collieries Corp.	Vermillion	5-22-51
28	Sherwood-Templeton Coal Co.	Sullivan	5-24-51
29	Sunlight Coal Co.	Warrick	5-29-51
30	Ayrshire Collieries Corp.	Vermillion	5-22-51
31	Sunlight Coal Co.	Warrick	5-29-51
32	Shasta Coal Corp.	Knox	5-26-51

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