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UNITED STATES
DEPARTMENT OF THE INTERIOR
GEOLOGICAL SURVEY
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December 7, 1956

AEC-284/7

Mr. Robert D. Nininger
Assistant Director for Exploration
Division of Raw Materials
U. S. Atomic Energy Commission
Washington 25, D. C.

Dear Bob:

Transmitted herewith are three copies of TEI-480, "Laboratory study of high-grade uranium-bearing lignite from Harding County, South Dakota," by James M. Schopf and Ralph J. Gray, March 1956.

We are asking Mr. Hosted to approve our plan to publish this report as part of a Geological Survey professional paper.

Sincerely yours,

John H. Eric

for W. H. Bradley
Chief Geologist

Geology and Mineralogy

This document consists of 59 pages.
Series A

UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

# LABORATORY STUDY OF HIGH-GRADE URANIUM-BEARING LIGNITE FROM HARDING COUNTY, SOUTH DAKOTA*

By

James M. Schopf and Ralph J. Gray

March 1956

Trace Elements Investigations Report 480

This preliminary report is distributed without editorial and technical review for conformity with official standards and nomenclature. It is not for public inspection or quotation.

*This report concerns work done on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission.

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# LABORATORY STUDY OF HIGH-GRADE URANIUM-BEARING LIGNITE FROM HARDING COUNTY, SOUTH DAKOTA

By James M. Schopf and Ralph J. Gray

## ABSTRACT

Samples of three coal beds with individual layers containing more than 0.1 percent uranium have been studied; two from the Riley Pass area of North Cave Hills and one from the southern part of the Slim Buttes. The coal beds do not show top-preferential distribution of uranium, but each high-grade layer appears to be adjacent to a zone of permeability. Analcite spherulites and crystals provide additional evidence of ground-water movement through the coal. Although all the coal beds have been more or less freely penetrated by ground water, not all show evidence of weathering.

Thick woody layers usually contain least uranium and least analcite. These layers have the lowest ash content, low porosity, and low permeability. Attrital coal generally includes more extraneous mineral matter. This helps to explain why the greater concentrations of uranium are usually associated with layers having more than 10 percent ash. Relatively permeable attrital coal, long exposed to uranium-bearing ground water, is most likely to have a high uranium content.

## INTRODUCTION

Coal containing sufficient uranium to be classed as a uranium ore was reported in Harding County, S. Dak., by Gill in 1954. This significant find was the result of extensive studies in the area started by the

U. S. Geological Survey in 1948. In 1951 laboratory studies of the uraniferous coal were started and results of these studies have recently been reported (Schopf and Gray, 1954; Schopf, Gray, and Felix, 1955). Nearly all material studied previously contained relatively small amounts of uranium, but was of special interest because of the ease with which uranium may be concentrated by ashing coal. Such coal would not be salable for its uranium alone, but the ashed concentrate of uranium and other mineral elements might be of value. Uraniferous coal appears in a different economic perspective when uranium is so concentrated that the coal itself can be classed as a potential uranium ore.

The present report is concerned with relatively high-grade uranium occurrences in coal at two separate localities in Harding County, S. Dak. Studies of this material seem particularly appropriate in view of our extensive previous study of lower grade uraniferous material. This work has been carried on by the U. S. Geological Survey under the auspices of the Division of Raw Materials of the U. S. Atomic Energy Commission.

## Acknowledgments

The column specimen from southern Slim Buttes was obtained by J. R. Gill and N. M. Denson. Gill provided notes giving excellent field data. Core sections from the Riley Pass district of North Cave Hills were provided for this study through cooperation of the Homestake Mining Company. Roy Kepferle of the U. S. Geological Survey supervised collection and packing of this material for shipment and supplied a field description of the core and notes on the geologic section of this locality. Coal analyses of samples cut from this material were made by the U. S. Bureau of Mines Coal Analysis Section under supervision of

Roy F. Abernethy. Determinations of ash, uranium (fluorimetric), and mineral elements in the ash (semi-quantitative spectrographic) were made in the Washington laboratory of the U. S. Geological Survey under supervision of Irving May and Claude Waring. Bruce D. Middleton of the U. S. Geological Survey prepared most of the thin sections used in this study and drafted the illustrations.

## LOCATION AND GEOLOGIC SETTING

Figure 1 shows the locations from which samples of high-grade uraniferous coal were obtained for the present study. It also shows a number of other areas in the same region that contain uraniferous lignite studied by the U. S. Geological Survey. Samples from the southern part of the Slim Buttes, east of Cedar Canyon, occur in the Ludlow member of the Fort Union formation of Paleocene age. Material from the Riley Pass district of the North Cave Hills occurs in the overlying Tongue River member of the same formation. The two localities are nearly forty miles apart.

An unconformity truncates the Fort Union formation in northwestern South Dakota. The Fort Union dips gently to the northeast so that, to the southwest, older rocks underlie the unconformity. Thus the southern Slim Buttes material is older than that from North Cave Hills. Tuffaceous clastics of Oligocene and Miocene age occur above the unconformity in this region. Zeller (1952), Gill (1954, 1955), Gill and Moore (1955), Denson and Gill (1955), Denson, Bachman, and Zeller (1950, 1954), and King (1955) have discussed regional and local geology and emphasized the importance of overlying tuffaceous deposits to the occurrences of uraniferous lignite. Baker (1952) has discussed the physiography and general geology of Harding County, S. Dak.

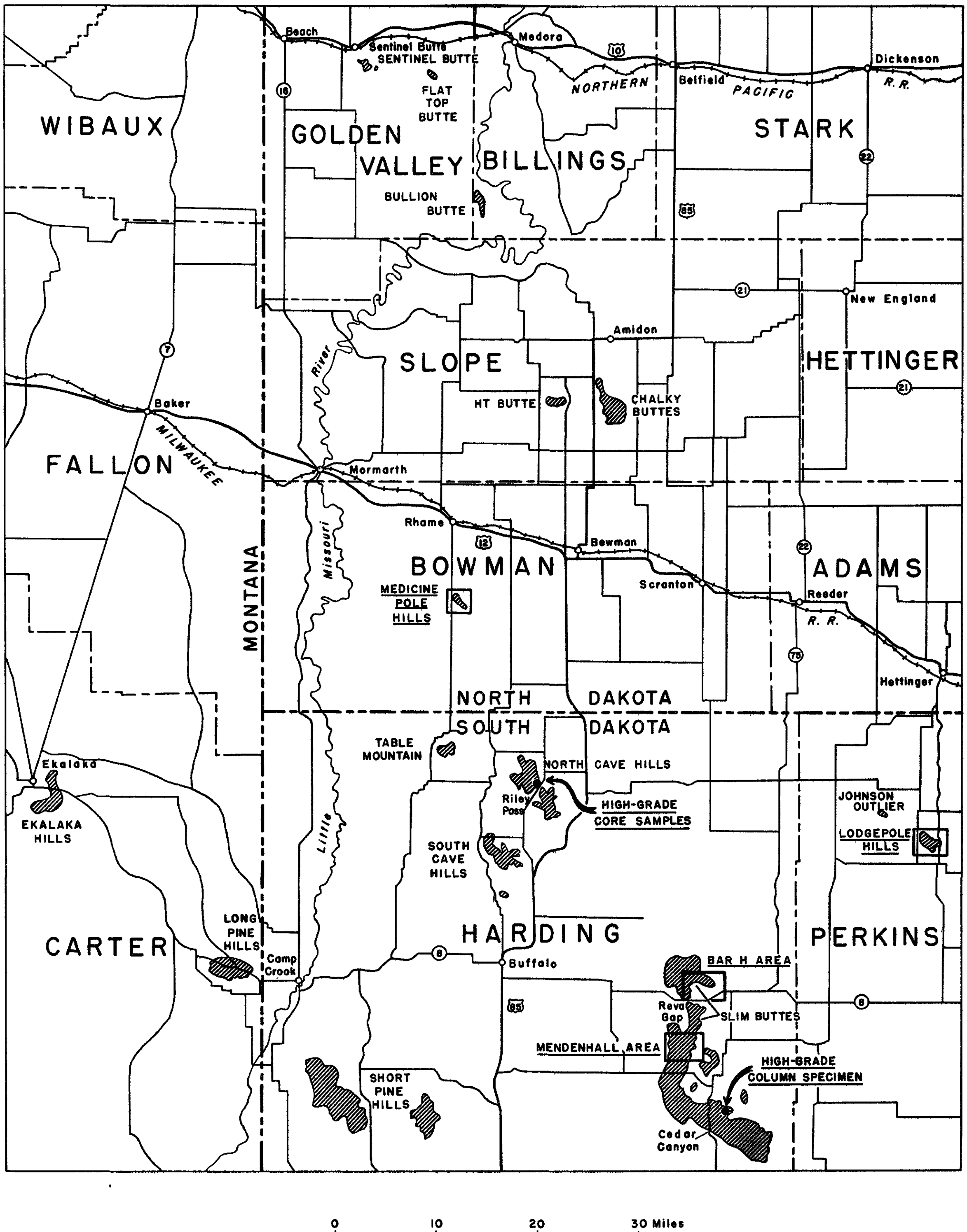


Figure I.— Index map showing areas of uranium-bearing lignite.

# MATERIAL STUDIED

The following section, at the southern Slim Buttes locality where the column specimen was obtained, has been summarized from notes supplied by J. R. Gill. Gill and Denson (1955) have discussed the occurrence of uraniferous lignite in the southern part of the Slim Buttes in greater detail.

| Depth, in feet | | Thickness |
|---|---|---|
| 0 | | |
| | Soil and clay, brown, bentonitic | 4.0 ft. |
| 4.0 | | |
| | Sandstone, gray, silty and clayey | 14.0 ft. |
| 18.0 | | |
| | Sandstone, gray (locally pink), coarser than above, with pebbles of quartzite and silicified wood | 8.0 ft. |
| 26.0 | <-- Unconformity (Chadron above; Ludlow below) | |
| | Sandstone, gray, cross-bedded, irregular, with clay pebbles up to 4" diameter | 2.0 ft. |
| 28.0 | | |
| | Clayey lignite, soft | 0.25 ft. |
| 28.2 | | |
| | Lignite, sparsely banded, dense, pyritic | 1.6 ft. "Z" bed |
| 29.8 | | |
| | Claystone, carbonaceous | 1.0 ft. |
| 30.8 | | |
| | Siltstone, dark gray, sandy, hard | 3.6 ft. |
| 34.4 | Bottom of exposed section | |

Only the dense coal of the bed Gill and Denson designate as "Z" was shipped to the laboratory. A detailed laboratory description of the coal and the identification of analytic sample intervals is given in the appendix (p. 56). The locality is in the NE 1/4 SW 1/4 Sec. 2, T. 16 N., R. 8 E.

Following is a summary description of the core from the Riley Pass area from notes taken in August 1955 by Roy C. Kepferle.

| Depth, in feet | | Thickness |
|---|---|---|
| 0 | | 0 |
| | No samples taken | |
| 10.0 | | |
| | Claystone and siltstone, with abundant analcite spherulites; .8 ft. of banded analcite below 10.9 ft. | 22.3 ft. |
| 22.3 | | |
| | Claystone, olive, with coaly streaks | ) |
| 22.7 | | ) |
| | Loss in drilling | ) |
| 24.0 | | ) 4.2 ft. |
| | Claystone, carbonaceous, with spherulites | ) "F Rider" bed |
| 25.0 | | ) |
| | Claystone, carbonaceous and brownish, with spherulites | ) |
| 26.2 | | ) |
| | Clayey lignite | ) |
| 26.5 | | |
| | Claystone, gray, with spherulites | |
| 27.2 | | |
| | Loss in drilling | 4.5 ft. |
| 30.0 | | |
| | Claystone, gray, with spherulites | |
| 31.0 | | |
| | Lignite, woody | ) |
| 31.4 | | ) |
| | Claystone, woody streak near top, with spherulites | ) |
| 32.0 | | ) |
| | Lignite, with pyrite and analcite crystals | ) |
| 32.3 | | ) 3.5 ft. |
| | Claystone and clayey lignite | ) "F" bed |
| 32.6 | | ) |
| | Lignite, woody and attrital | ) |
| 34.2 | | ) |
| | Claystone, with spherulites | ) |
| 34.4 | | ) |
| | Lignite, woody, pyritic | ) |
| 34.5 | | |
| | Claystone and siltstone, with abundant spherulites | 14.3 ft. |
| 48.4 | | |
| | Lignite, weathered, broken in drilling | ) |
| 49.1 | | ) |
| | Sandstone, limonitic, hard | ) 1.4 ft. |
| 49.9 | | ) "E" bed |
| | Lignite, weathered, broken in drilling | ) |
| 50.2 | | |
| | Sandstone, medium to fine grained, limonitic | 4.8 ft. |
| 55.0 | Total depth drilled | |

Core sections, including coal and strata adjacent to the three coal beds designated as "F Rider", "F", and "E" by Kepferle, were shipped to the laboratory. The detailed description of material studied at the laboratory and analytic sample identifications are given in the appendix (p. 57). The core was obtained on Homestake-Riley No. 4 claim near the center of the NW 1/4 NW 1/4 sec. 26, T. 22 N., R. 5 E.

A general summary of the lithologic character and radioactivity of samples from both localities is given in figure 2. Sample numbers given on figure 2 correspond with the intervals for which detailed descriptions are given in the appendix. Intervals sampled for coal analyses by the Bureau of Mines also are indicated by B of M analytic sample numbers. Radioactivity has been shown in units of pulses per minute per gram (P/M/G) for all material submitted. The crosshatched areas of the radioactivity profile correspond with samples of highest radioactivity; these areas represent double values wherever they overlap the non-hatched portion of the profile. At normal scale all these points would be displaced to the right a distance corresponding to approximately 5-1/2 P/M/G units. The P/M/G determinations are in accord with specific uranium determinations.

Uranium was determined fluorimetrically for all samples having radioactivity exceeding one P/M/G unit. These "TE" samples are not numbered in figure 2 but are indicated in the appendix (p. 56). Radioactivity determinations are less accurate as a basis for predicting uranium content of these samples, however, than in material where variations in uranium content are less extreme. Although, on the average, one P/M/G unit appears to correspond with 25 to 30 parts per million of uranium, radioactivity appears to be too low in the very rich samples, in

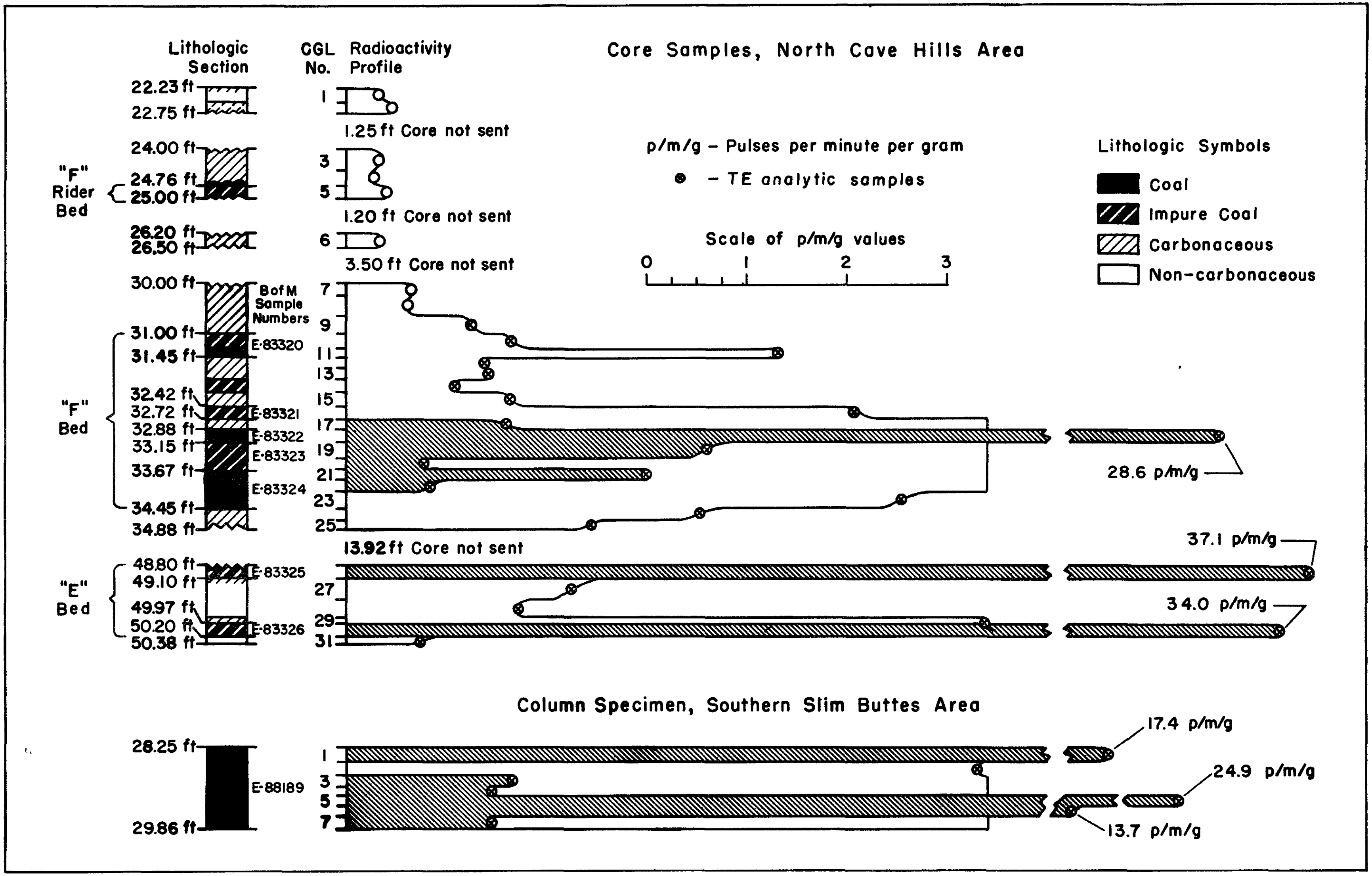


FIG. 2 .–LITHOLOGIC SECTION, SAMPLING INTERVALS AND RADIOACTIVITY OF MATERIAL STUDIED.

terms of uranium actually determined, and too high in the lower range of uranium concentration in the samples that were analyzed. The P/M/G results are sufficiently accurate, however, to illustrate the great range of difference in different layers of material in the two deposits, and also to show that radioactive materials in these coal beds do not have an evident top-preferential distribution. Under these circumstances one might expect composition of the coaly material to be more important than it seems to be for low-grade uraniferous coal previously studied (Schopf, Gray, and Felix, 1955, p. 82).

## Chemical analyses

Chemical analyses of coal are obtained by meticulous adherence in the laboratory to standard analytic procedures (ASTM standards, p. 582, 1949). Coal analyses made by these procedures afford a basis for comparison only if the samples are truly comparable. Lignitic coal is highly susceptible to deterioration through natural weathering and can even be altered to some extent while awaiting analysis. For these reasons, it is often difficult to determine precisely the condition of coal as it occurs in its natural state underground.

The composition of unmined coal is important in the present connection because this may be directly related to the conditions of uranium emplacement. The question arises, for example, whether uranium was introduced early in the geologic history of the coal deposits or relatively late during the present cycle of denudation. Condition of the undisturbed coal is a starting point for such consideration.

All material studied was moist when received. Analyses of the southern Slim Buttes column specimen may be the most reliable because the analytic sample consisted of an interior section cut from two large blocks. Samples taken from drill cores generally contain excess visible moisture from drilling operations. This was particularly evident for samples of broken coal from the "E" coal bed, important for their high uranium content, which were virtually "slurry" when received at the laboratory. Unfortunately, some material from these coal layers was lost in drilling and not enough coal was available for a complete set of analyses. The "slurry" samples were dried to a moist granular consistency (free of visible moisture) before being submitted for chemical analysis. Thus, values that can be reported for the "E" bed do not represent fully the condition of the coal as it exists underground. Five coal core samples taken from layers of the "F" bed did not contain visible excess moisture and are more representative of the coal in its natural condition. Moisture is important in evaluating analyses of lignite because an inaccuracy in moisture is reflected in the amounts of all other moist-basis components of the coal. A deficiency in moisture could indicate excessive exposure subsequent to sampling.

Analytic data are given in table 1 for seven coal samples from the Riley Pass drill core and for the complete column specimen from the Cedar Canyon district of the southern Slim Buttes. Ash content varies from 7.8 percent to 34.9 and moisture from 41 percent (column specimen) to 49.2 percent for one of the "dried" samples from the "E" bed of the Riley Pass drill core. The relatively low calorific values for some of the drill core samples may be the result of weathering, regardless of the considerable depth from which they were taken.

Table 1. -- Chemical analyses of uraniferous coal from Harding County, S. Dak.

| Basis of reporting 1/ | Proximate, percent | | | | Ultimate, percent | | | | | | | | Calorific value |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Moisture | Volatile matter | Fixed carbon | Ash | Total sulfur | Sulfate sulfur | Pyritic sulfur | Organic sulfur | Hydrogen | Carbon | Nitrogen | Oxygen | Btu |
| | Riley Pass Core, depth 31' - 31'5-1/2" (5-1/2"); B of M E-83320 | | | | | | | | | | | | |
| AR | 46.0 | 16.7 | 20.2 | 17.1 | .5 | .04 | .27 | .21 | | | | | 4,170 |
| MF | | 30.9 | 37.4 | 31.7 | 1.0 | .07 | .50 | .39 | | | | | 7,720 |
| M&AF | | 45.3 | 54.7 | | 1.4 | .10 | .74 | .58 | | | | | 11,300 |
| | Riley Pass Core, depth 32'5" - 32'8-5/8" (3-5/8"); B of M E-83321 | | | | | | | | | | | | |
| AR | 41.9 | 20.1 | 21.0 | 17.0 | .8 | .02 | .24 | .49 | | | | | 4,940 |
| MF | | 34.6 | 36.1 | 29.3 | 1.3 | .03 | .42 | .84 | | | | | 8,510 |
| M&AF | | 48.9 | 51.1 | | 1.8 | .05 | .59 | 1.19 | | | | | 12,030 |
| | Riley Pass Core, depth 32'10-1/2" - 33'1-3/4" (3-1/4"); B of M E-83322 | | | | | | | | | | | | |
| AR | 41.4 | 22.8 | 28.0 | 7.8 | 1.2 | .03 | .47 | .71 | 7.2 | 34.9 | .4 | 48.5 | 6,240 |
| MF | | 38.9 | 47.8 | 13.3 | 2.1 | .05 | .80 | 1.21 | 4.4 | 59.6 | .7 | 19.9 | 10,650 |
| M&AF | | 44.9 | 55.1 | | 2.4 | .06 | .92 | 1.40 | 5.0 | 68.7 | .8 | 23.1 | 12,290 |
| | Riley Pass Core, depth 33'1-3/4" - 33'8" (6-1/4"); B of M E-83323 | | | | | | | | | | | | |
| AR | 33.9 | 17.4 | 13.8 | 34.9 | 1.0 | .03 | .53 | .45 | | | | | 3,530 |
| MF | | 26.3 | 21.0 | 52.7 | 1.5 | .05 | .81 | .67 | | | | | 5,340 |
| M&AF | | 55.5 | 44.5 | | 3.2 | .11 | 1.71 | 1.42 | | | | | 11,280 |
| | Riley Pass Core, depth 33'8" - 34'5-1/2" (9-1/2"); B of M E-83324 | | | | | | | | | | | | |
| AR | 42.3 | 24.4 | 24.1 | 9.2 | .6 | .01 | .05 | .57 | 7.3 | 34.3 | .4 | 48.2 | 5,980 |
| MF | | 42.3 | 41.7 | 16.0 | 1.1 | .02 | .08 | .99 | 4.5 | 59.4 | .7 | 18.3 | 10,360 |
| M&AF | | 50.4 | 49.6 | | 1.3 | .03 | .10 | 1.18 | 5.4 | 70.7 | .8 | 21.8 | 12,340 |
| | Riley Pass Core, depth 48'9-5/8" - 49'1-1/4" (3-7/8"); B of M E-83325 | | | | | | | | | | | | |
| AR | 49.2 | 12.9 | 4.8 | 33.1 | .3 | .04 | .10 | .14 | | | | | |
| MF | | 25.4 | 9.5 | 65.1 | .6 | .09 | .21 | .27 | | | | | |
| | Riley Pass Core, depth 49'11-5/8" - 50'2-3/8" (2-3/4"); B of M E-83326 | | | | | | | | | | | | |
| AR | 46.9 | 18.1 | 9.8 | 25.2 | .3 | .07 | .13 | .13 | | | | | |
| MF | | 34.1 | 18.4 | 47.5 | .6 | .13 | .25 | .24 | | | | | |
| M&AF | | 64.9 | 35.1 | | 1.2 | .25 | .48 | .45 | | | | | |
| | Southern Slim Buttes Column, 1'7-1/4" thick, B of M E-88189 | | | | | | | | | | | | |
| AR | 41.0 | 21.5 | 24.1 | 13.4 | 1.1 | .05 | .71 | .33 | 6.8 | 32.6 | .5 | 45.6 | 5,670 |
| MF | | 36.4 | 40.9 | 22.7 | 1.8 | .08 | 1.21 | .56 | 3.8 | 55.3 | .8 | 15.6 | 9,620 |
| M&AF | | 47.1 | 52.9 | | 2.4 | .10 | 1.56 | .73 | 4.9 | 71.5 | 1.0 | 20.2 | 12,450 |

1/ AR, as received; MF, moisture free; M&AF, moisture and ash free.

Weathered coal is softer than unweathered and tends to hold more moisture. It is frequently pulverized in coring, as were the two samples of the "E" bed. However, these criteria are not specific enough to indicate when coal is only slightly weathered. Calorific values are more generally useful to indicate weathering of coal because they can be measured accurately and always are lower if the coal samples have been partially oxidized. Nevertheless, it is sometimes difficult to determine whether a slight decrease in calorific value is owing to incipient weathering or whether it is a result of impurity or peculiarities of organic composition. Effects of weathering on calorific value of coal is most apparent if results are compared on a dry, mineral-free (unit coal) basis, but moisture is so important in lignite that comparisons on a moist, mineral-free basis usually are preferred. Some incidental variation in the moisture of core samples seems unavoidable and it would appear that moist Btu values would be more readily interpreted as to weathering if variations in moisture can be minimized. To serve this purpose, we have calculated mineral-free Btu values on three different bases, including one set of figures for all suitable samples on a common moisture basis of 42 percent. A moisture content of 42 percent was chosen, as this appears to be about the normal moisture content for unweathered coal beds in this particular area. The mineral-free Btu values are given on the dry or moisture-free basis, on the basis of as-received moisture, and calculated for uniform 42 percent moisture, in columns 4, 5, and 6 of table 2. The normal as-received, moisture-free, and moisture-and-ash-free Btu values, more commonly used, were included in table 1.

Table 2. — Moisture and impurity, calorific values, and sulfide-sulfate ratio of uraniferous samples.

| Analysis numbers (Corresponding TE numbers in parentheses) | Moisture AR 1/ | Ash AR | Mineral matter 2/ | Mineral matter free calorific values (Btu) | | | Sulfide-sulfate ratio ($S_2/SO_4$, AR basis) | Comments |
|---|---|---|---|---|---|---|---|---|
| | | | | Moisture AR | Moisture 42% | Dry basis (Unit coal) | | |
| NORTH CAVE HILLS CORE SAMPLES | | | | | | | | |
| E-83320 (TE 2 & 3) | 46.0 | 17.1. | 18.7 | 5100 | 5480 | 11,760 | 6.7 | Slight weathering |
| E-83321 (TE 8) | 41.9 | 17.0 | 18.8 | 6040 | 6030 | 12,470 | 12.0 | Not weathered |
| E-83322 (TE 10) | 41.4 | 7.8 | 9.1 | 6800 | 6730 | 12,480 | 15.6 | Not weathered |
| E-83323 (TE 11 & 12) | 33.9 | 34.9 | —— | —— | —— | —— | 17.6 | Not weathered, coal impure |
| E-83324 (TE 13 - 15) | 42.3 | 9.2 | 10.3 | 6630 | 6610 | 12,540 | 5.0 | Slight weathering, pyritic oxidation |
| E-83325 (TE 18) | 49.2 | 33.1 | —— | —— | —— | —— | 2.5 | Highly oxidized, coal impure |
| E-83326 (TE 22) | 46.9 | 25.2 | —— | —— | —— | —— | 1.9 | Highly oxidized, coal impure |
| SOUTHERN SLIM BUTTES COLUMN SPECIMEN SAMPLE | | | | | | | | |
| E-88189 | 41.0 | 13.4 | 15.0 | 6490 | 6500 | 12,550 | 14.2 | Not weathered |

1/ AR = as received.

2/ Mineral matter calculated as 1.08 ash + 0.55 sulfur (Parr, p. 49, 1932)

Another indication of weathering, independent of other analytic data, may be derived by comparison of pyritic (sulfide) and sulfate sulfur. Forms of sulfur were determined for all samples studied (table 1). Very little, if any, sulfate is naturally present in coal. The ubiquitous pyritic minerals oxidize readily on exposure, however, so that nearly all samples show at least a trace of sulfates. The ratio of sulfide to sulfate sulfur may, therefore, be useful as a supplemental index to the weathering of samples. The sulfide-sulfate ratio, obtained by dividing the amount of sulfur determined as sulfide by the amount determined as sulfate, is given in column 7 of table 2 _/. High values indicate little sulfide oxidation and low values may be taken as an indication that some oxidation probably has occurred prior to field sampling of the coal.

_/ The inverse relationship ($SO_4/S_2$) could be used alternatively, and might have some advantage in expressing amount of pyritic oxidation when pyritic sulfur is dominant. By this procedure, values exceeding 0.125 probably would be interpreted as showing effects of weathering.

The apparent relation of calorific values and sulfide-sulfate ratio is shown for samples under consideration in figure 3. It is doubtful that either criterion can be rigorously applied even though the correlation seems good for four of these samples. The lowest sample from the "F" bed (E-83324) falls far from the line indicated by the others. This sample suggests that in some instances pyritic material can be oxidized without a noticeable effect on the calorific value of associated organic matter. An evaluation of the degree of weathering of these samples is given in the comments column of table 2.

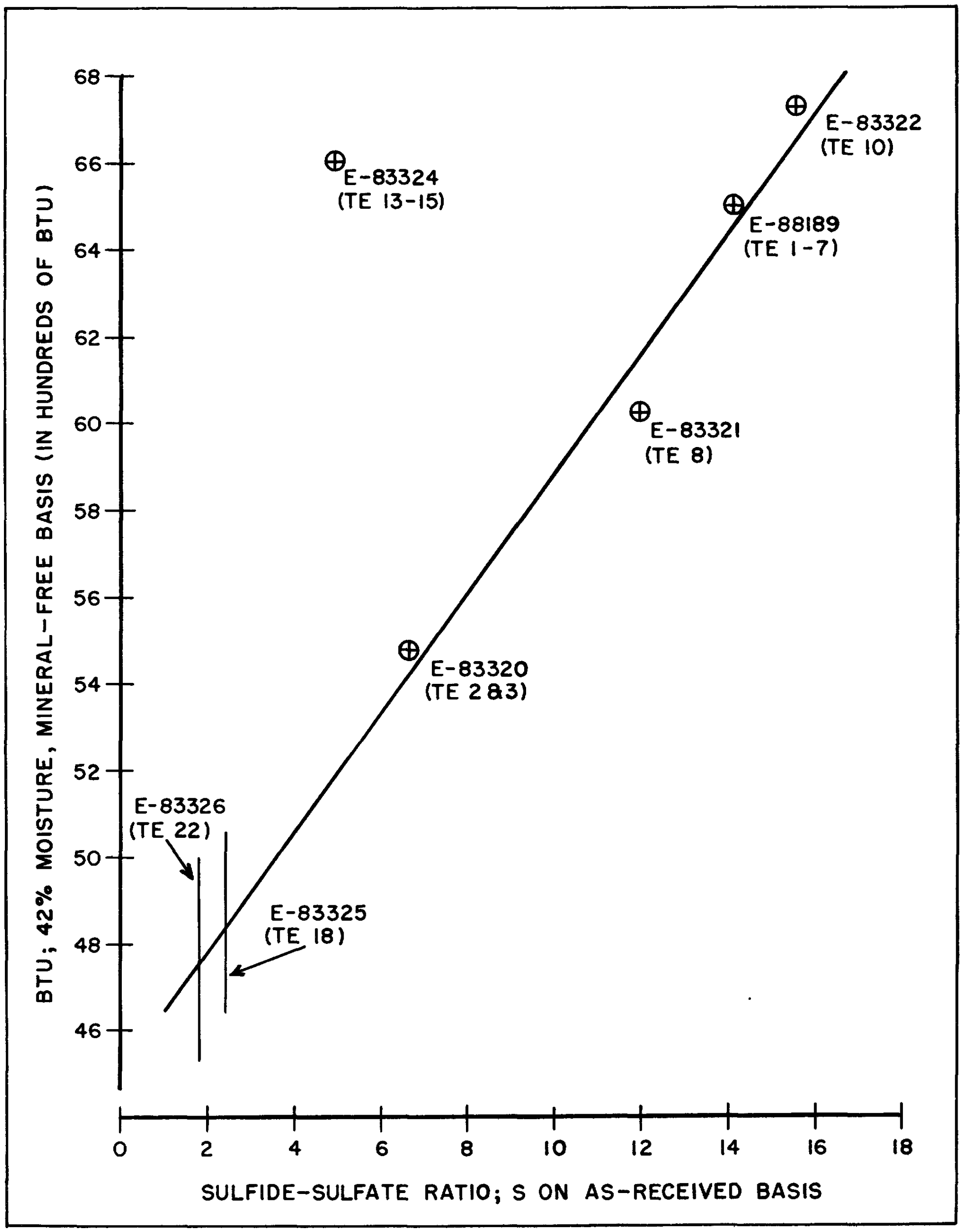


FIG. 3.-RELATION OF CALORIFIC VALUE TO SULFIDE-SULFATE RATIO.

Ash fusion data, shown in table 3, were obtained for two of the samples from North Cave Hills and for the southern Slim Buttes column specimen. The fusion temperatures all are relatively low. Crystalline analcite ($NaAlSi_2O_6 \cdot H_2O$), one of the zeolites, is visible in microscopic preparations of these samples and might be expected to lower the ash fusion temperature. The fusion temperature of analcite is about 2060° F and its eutectic effect would depend on relations with other mineral constituents of the ash. These effects may have some importance in recovery of uranium from the ash of coal deposits.

Table 3. -- Fusibility of ash of uraniferous coal samples from Harding County, S. Dak.; temperatures given in degrees Fahrenheit.

| Locality | Initial deformation | Softening | Fluid |
|---|---|---|---|
| North Cave Hills Core | | | |
| "F" bed (part) Depth 32.88' - 33.15' | 1930 | 2050 | 2130 |
| "F" bed (part) Depth 33.67' - 34.15' | 1870 | 2010 | 2150 |
| Southern Slim Buttes Column | | | |
| "Z" bed Including 1.61' of coal | 1910 | 2000 | 2310 |

## COAL PETROLOGIC STUDIES

Quantitative results of megascopic and detailed microscopic examination can be presented for the "Z" bed of the southern Slim Buttes and for the "F" bed of the Riley Pass core from North Cave Hills. The "F Rider" bed is represented at this locality by very thin impure coal and carbonaceous claystone, and the two coal layers of the "E" coal bed, near the bottom of the drill hole, were pulverized in coring. Material representing the "F Rider" bed was essentially non-radioactive (fig. 2) and was not studied for this reason. Layers of the "E" coal bed were highly radioactive, but it was impossible to obtain as complete information from them, owing to their fragmented condition, and only the maceral composition can be given. Studies of the "Z" and "F" beds are based on oriented specimens that permit consideration of texture as well as composition. Except for the studies of the "E" bed, discussed later, methods of study are similar to those employed in our previous laboratory investigations of uranium-bearing coal (Schopf and Gray, 1954, 1955; Schopf, Gray, and Felix, 1955).

### "Z" bed, southern Slim Buttes

The stratigraphic relations of the "Z" bed to other beds in the southern part of the Slim Buttes have been illustrated by Gill and Denson (1955, fig. 48). Water from a nearby spring contained 0.2 ppm uranium (Gill and Moore, 1955). The column specimen reported on has an average uranium content exceeding 0.04 percent.

The coal studied is somewhat less woody and finer textured than the average of the coal beds in the central Slim Buttes, Mendenhall area, a few miles to the northwest. If two dominantly woody layers are excluded, less than 18 percent of the coal is pre-vitrain. All told, thick pre-vitrain (woody bands more than 5 mm in thickness) comprises about 30 percent of the coal and less than 10 percent pre-vitrain is in the thin and medium size range (1/2 to 5 mm). About 1-1/2 percent of the bed consists of fusain in lenticles and fragments more than 1/2 mm thick. Thus, the matrix of very fine-textured coal presents a considerable contrast with the thick woody bands. This contrast is the result of a moderate deficiency in pre-vitrain bands of the intermediate sizes.

About thirty thin sections were prepared from the column specimen. Seven of these are essentially duplicates but, in all, more than 94 percent of the total coal thickness has been studied in detail. The small part not represented in thin sections is well distributed and not selective. The results of critical microscopical studies of this suite of preparations can be considered representative.

The results of microscopical studies are summarized in the chart showing petrologic composition in figure 4. Two of the layers are dominated by thick woody bands but, other than this, the composition is dominantly attrital. About 57 percent of the coal can be classed as anthraxylon. Thick anthraxylon is chiefly derived from gymnospermous wood of a moderately resinous type. About 5 to 10 percent of the woody pre-vitrain consists of red resinous inclusions in wood parenchyma cells. Fibrous anthraxylon is chiefly derived from roots and rootlets now much flattened and lenticular. The rootlets often show an exodermal layer of the type previously observed in coal of the central area of the Slim

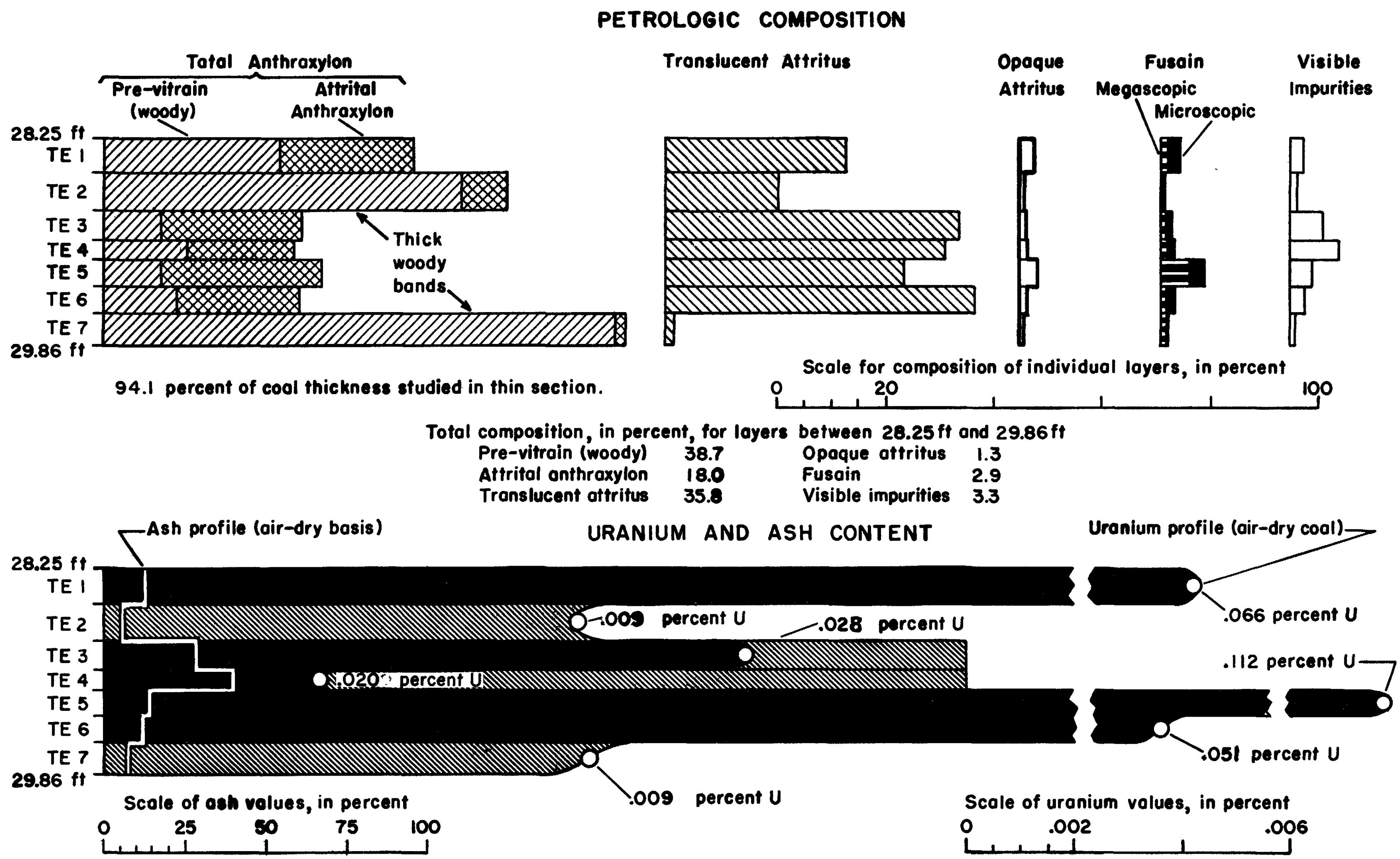


Figure 4.—Composition of uranium-bearing lignite from southern Slim Buttes, Harding County, South Dakota.

Buttes where they commonly are more abundant. Microscopic study shows a total of nearly 3 percent fusain and 1.3 percent opaque attritus. About 36 percent is translucent attritus and, combined with anthraxylon, about 93 percent of the coal consists of relatively reactive constituents. Thus, a significantly smaller amount of relatively inert organic constituents is present than in most of the Slim Buttes coal previously studied (Schopf, Gray, and Felix, 1955).

The coal presents a relatively clean appearance without obvious partings, although the central part of the bed has a high ash content and microscopical mineral matter is visible generally, interspersed in the fine-textured coal. It is noteworthy that very little mineral matter can be seen invading woody bands in this coal bed.

Three principal types of visible mineral matter can be distinguished. Disseminated pyritic minerals are common, but only a few aggregates of megascopic size were seen. A very finely granular form of iron sulfide was observed concentrated in the cambial zone of a woody lenticle in layer TE-2 to suggest that it was deposited as a result of decomposition of least resistant tissues. Commonly, small spheroidal pyritic aggregates are present. Some show a framboidal type of aggregation, but others seem to be composed of elongate radiating crystals. Both microscopic cubes and octahedra are dispersed as individual crystals in the coal. A few octahedra were large enough to be isolated for conclusive demonstration of the form. Evidently both pyrite and its isomer, marcasite, are represented. No apparent secondary occurrences of pyrites were noted. Most of the pyritic minerals, if not all, seem to have been deposited very early during diagenesis of peat.

Extremely fine-textured clayey mineral matter is evident in sections from highly attrital layers TE-2, 3, and 5. Apparently the clay is of detrital origin. No instances of kaolinitic "petrifaction" were seen like those described in several coal beds of the central Slim Buttes area.

Analcite (analcime) is the most abundant mineral present in this coal _/. It is present in the form of nucleated crystals, crystal aggre-

_/ Four batches of selected aggregates or crystals showing differences of habit were all identified as analcite by use of optical and X-ray diffraction methods by Jerome Stone and Daphne Riska of the Geochemistry and Petrology Branch of the U. S. Geological Survey. During the course of this work the identification of other specimens was checked by determining refractive index.

gates and most commonly as spherulites. Trapezoidal crystals and crystal aggregates are inconspicuous in thin sections, but some crystals, usually less than 100$\mu$ in diameter, are generally present in washed residues from crushed coal. Within the coal, analcite is most abundant in the attrital layers.

The spherulites most fully formed are oval in vertical section, with the longer diameter accordant with the banding of the coal, about 500 or 600 microns in diameter with a marginal clear zone and a large nucleus. Some of the spherulites are congruent and have marginal zones which are more or less joined. The nucleus is usually about 300 or 400 microns in diameter and includes microbands of anthraxylon and attritus that are evident continuations of the texture shown in adjacent unmineralized coal. Most of the assymmetry of the spherulites is associated with the marginal zone of nearly clear analcite that is thinner on bottom and top than it is on the sides. Within the clear zone, isolated tiny particles of organic matter are present in pseudo-radial lines that resemble cone-in-cone microstructure and appear to bear a relation to crystal growth against a restraining pressure. Small crystal facets can be seen terminating the marginal clear zone of some of the isolated spherulites, but this is rarely observable in the thin sections. In general, the margins abutting on unmineralized coal are ragged and irregular and the presence of these rough surfaces may explain why the spherulites are nearly invisible on megascopic examination of the "Z" coal bed.

The nuclei of both the spherulites and crystals are similar and include organic matter that is as heterogeneous as materials in the attrital coal. Crystal nuclei are generally much smaller than spherulitic nuclei. Many nuclei of larger spherulites consist of masses of typical attrital debris permeated by mineral matter, but scarcely disarranged by it. In other instances mineral-filled radiating fissures are present in the organic nuclei. In some instances the nuclear fissures have a stellate arrangement with many major and minor rays. In some spherulites, the marginal clear zone is lacking and the appearance is that of an incomplete stage of spherulitic formation. The simplest form of analcite occurrence is represented by mineral filling small lenticular fissures 10 or 20 microns long. These fissure fillings evidently represent the primary rays of "stellate" nuclei; small nuclei that lack the fissures are not as evident. The small initial fissures commonly are oriented obliquely and when four rays are evenly developed look like microscopic "x's".

Several lines of evidence suggest that the analcite was introduced into the coal after it had reached its present stage of compression. Nothing resembling vestiges of volcanic ash particles is visible in the coal, and numerous spherulites are present in plant tissue locations that could never have been accessible to solid particles of ash. Uncompressed (peat stage) plant cellular structure apparently is never preserved by analcite mineralization. In a few instances the compression lineation of the coal is slightly bowed above and below larger spherulites but, in general, the mineral seems to permeate, penetrate, and split the coal (as in the large stellate nuclei) rather than deform it. Inasmuch as this coal contains more than 40 percent moisture, crystallization of small spherulites of a hydrous mineral may not involve a proportional volume displacement. Peat deposits antecedent to coal beds are water saturated when overburden is deposited. Weight of overburden causes reduction in moisture as the deposit is transformed to consolidated coal. Thus, the direction of movement of moisture is <u>out</u> of the super-hydrated mass of peaty organic matter during diagenesis. It seems unreasonable to suppose that enough sodium and aluminum silicate ions to form the observed quantity of analcite could come from precipitation out of fluids indigenous to the original peat deposit. For these reasons we believe analcite formed from ionic solutions brought into the coal <u>after</u> the organic matter had reached its present state of compaction.

A significant ground water movement through coal beds could occur at any time after the coal deposits were elevated above the regional water table and, until the coal is near the erosion surface, saturation conditions could be maintained to inhibit oxygenation of the coal. In the Dakota region, ground water could readily serve to transfer sodium, aluminum, and silicon ions into the coal from overlying tuffaceous deposits and permit the forms of secondary analcite, just described, to be deposited. Accordingly, the presence of analcite spherulites is regarded as a rough indication of the extent of ground water circulation.

Uranium and ash content of the seven samples of coal from the "Z" coal bed are shown graphically in the chart at the bottom of figure 4. The black areas of the chart correspond with samples of greatest uranium content; these areas represent double values wherever they overlap crosshatched portions of the uranium profile. The points at the tips of black bars would all be displaced to the right a distance corresponding to about .016 percent U, except where bars are broken, if drawn to normal scale. Layers TE-2 and TE-7, both dominantly woody, contain the least amount of ash and only about 90 parts per million of uranium each. Layer TE-5, consisting of dominantly attrital coal, moderate in ash content and having a relatively large amount of opaque attritus and fusain, contains about 1120 parts per million uranium or nearly twice that of any other layer in the bed. Its position below a layer of high ash content may be significant. The two layers having next highest content of uranium (TE-1, 660 ppm; TE-6, 510 ppm) are correspondingly intermediate in ash content, contain large amounts of attrital coal and more opaque attritus and fusain than the layers having less uranium. The negative correlation of uranium with low-ash bands of thick woody anthraxylon is most striking, but the concurrence of the other factors should also be considered.

A detailed analysis of the composition of different layers of the "Z" bed coal was made to test the possibility that specific components may have a quantitative relationship to uranium concentration. Fourteen organic entities and four types of mineral matter have been determined quantitatively by microscopic study. Results are presented in table 4, on the basis of area (= volume), arranged according to uranium content.

Several of the entities present in minor amounts tend to parallel the concentration of uranium. The fungal phyterals, chiefly sclerotia with a few fungus spores and little visible mycelium, show a relative coincidence, except for a reversed relationship in the two layers of greatest uranium concentration (TE-5 and TE-1). Some rough relationship with fungus remains might be expected on the basis of our earlier work (Schopf and Gray, 1954) if uranium emplacement is related to in situ degradation of organic matter. A similar, but more pronounced, parallelism also is shown by opaque attritus and fusain.

Opaque attritus and fusain are thought to have a similar diagenetic origin and, when combined, are generally called simply "opaque matter." This combination of entities also is approximately the same as "inertinite" of European terminology. The latter term has reference to the relatively inert chemical properties of these organic materials (cf., Schopf, 1948, p. 220-222). The detailed data given in table 5 can be combined to give values equivalent to opaque matter.

In view of the general coincidence of occurrence of opaque matter and uranium in this series of samples, it seemed advisable to make calculations for a more specific comparison. The relationship of opaque matter and uranium that is suggested here has not been indicated by our previous studies of lower grade Dakota coal.

In order to compare petrologic determinations and uranium determinations, it is necessary to express both on either a volumetric or gravimetric basis. Furthermore, for a volumetric comparison, it is

Table 4. — Composition (area percent) of uraniferous coal layers from southern Slim Buttes, Harding County, S. Dak. Layers presented in order according to their uranium content.

| Constituents or Components | TE-5 | TE-1 | TE-6 | TE-3 | TE-4 | TE-7 | TE-2 |
|---|---|---|---|---|---|---|---|
| Anthraxylon: | | | | | | | |
| Coarse | 10.82 | 32.88 | 13.46 | 10.83 | 15.48 | 95.02 | 66.89 |
| Attrital | 29.47 | 24.61 | 22.72 | 25.78 | 19.86 | 1.37 | 8.92 |
| Total | 40.29 | 57.49 | 36.18 | 36.61 | 35.34 | 96.39 | 75.81 |
| TRANSLUCENT ATTRITUS: Subanthraxylon | 2.26 | 4.55 | 2.69 | 1.55 | 2.45 | .03 | 1.19 |
| Humic Matter | 40.49 | 28.07 | 53.50 | 51.73 | 48.56 | 1.36 | 19.55 |
| Total | 42.75 | 32.62 | 56.19 | 53.28 | 51.01 | 1.39 | 20.74 |
| Red Att. Resins | .08 | .15 | .31 | .44 | .18 | .01 | .16 |
| Cuticle | — | — | .01 | — | — | — | — |
| Spores | .21 | .20 | .22 | .10 | .05 | .01 | .08 |
| Yellow Att. Resins | .05 | .09 | .09 | .11 | .11 | — | .07 |
| Waxy Amorphous | .36 | .26 | .38 | .58 | .38 | .04 | .11 |
| Total | .62 | .55 | .70 | .79 | .54 | .05 | .26 |
| Fungal Phyterals | .03 | .07 | .02 | .01 | .01 | — | T |
| Brown Matter | 1.04 | .38 | .30 | .19 | .21 | .02 | .15 |
| Total Trans. Attritus | 44.52 | 33.78 | 57.52 | 54.71 | 51.95 | 1.47 | 21.31 |
| Opaque Attritus | 3.23 | 2.69 | 1.26 | .63 | 1.23 | .05 | .57 |
| Microfusain | 2.40 | 2.20 | 1.62 | .98 | .86 | .38 | .77 |
| Megafusain | 5.62 | 1.53 | .86 | 1.09 | 1.50 | 1.38 | .58 |
| Total | 8.02 | 3.73 | 2.48 | 2.07 | 2.36 | 1.76 | 1.35 |
| Disseminated Pyrites | .75 | .73 | .33 | .68 | 1.05 | .17 | .22 |
| Transparent Minerals | .17 | .37 | .10 | .15 | .25 | T | .17 |
| Analcite (Analcime) | 2.13 | .84 | 1.61 | 1.50 | 2.55 | .09 | .46 |
| Clayey Minerals | .89 | .37 | .52 | 3.65 | 5.27 | .07 | .11 |
| Total | 3.94 | 2.31 | 2.56 | 5.98 | 9.12 | .33 | .96 |
| Total | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |

Table 5. — Composition (area percent) of translucent attritus in the uraniferous coal layers from southern Slim Buttes, Harding County, S. Dak.

| Constituents or Components | TE-5 | TE-1 | TE-6 | TE-3 | TE-4 | TE-7 | TE-2 |
|---|---|---|---|---|---|---|---|
| Subanthraxylon | 5.07 | 13.48 | 4.68 | 2.84 | 4.71 | 2.09 | 5.60 |
| Humic Matter | 90.96 | 83.11 | 92.99 | 94.56 | 93.47 | 93.03 | 91.72 |
| Total | 96.03 | 96.59 | 97.67 | 97.40 | 98.18 | 95.12 | 97.32 |
| Red Attrital Resins | .18 | .43 | .55 | .81 | .36 | .35 | .77 |
| Cuticle | — | — | .02 | — | — | — | — |
| Spores | .46 | .59 | .38 | .18 | .10 | .35 | .37 |
| Yellow Attrital Resins | .10 | .27 | .15 | .19 | .20 | — | .33 |
| Waxy Amorphous | .82 | .76 | .66 | 1.05 | .73 | 2.79 | .51 |
| Total | 1.38 | 1.62 | 1.21 | 1.42 | 1.03 | 3.14 | 1.21 |
| Total Fungal Phyterals | .08 | .22 | .04 | .02 | .03 | — | T |
| Brown Matter | 2.33 | 1.14 | .53 | .35 | .40 | 1.39 | .70 |
| Total | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Percent of Layer Represented by Translucent Attritus | 44.52 | 33.78 | 57.52 | 54.71 | 51.95 | 1.47 | 21.31 |
| Percent Uranium | .120 | .066 | .051 | .028 | .020 | .009 | .009 |

necessary to make an assumption as to density of the form in which uranium occurs in the coal, because the forms of occurrence are not definitely known. Breger, Deul, and Rubinstein (1955) have reported that it may exist as an organo-uranium complex or compound, but the physical properties of this combination have not been determined _/.

_/ Breger (written communication to J. M. S., Dec. 12, 1955) suggests that the uranyl ion may be retained, in Dakota coal or in coalified logs of the Colorado Plateau, primarily as a uranyl humate which may subsequently be reduced to uraninite.

When high concentrations of uranium are present in coal, it seems possible that some of it might be present as uraninite (ideally $UO_2$), just as it is in some of the coaly materials known from the Colorado Plateau (Alice Weeks, personal communication; also, Breger and Deul, 1955, p. 28), and as it is known to exist in thucholite (Frondel and Fleischer, 1955). If uranium is present in the form of an unknown organo-complex or compound, the only assumption possible is for the elemental uranium part of the compound, even though this is not really satisfactory. The theoretical uraninite relationship can be calculated approximately. The relationship with elemental uranium serves only as a reference computation.

It should be emphasized that we have not observed uraninite in this coal and that no other unusual mineral forms, other than analcite, have been observed by us in association with any coal component. Hydrated uranium minerals have been reported by Gill and Moore (1955) and by Gill (1955) but these occur on shrinkage cracks in weathered coal and on rock exposures and are not regarded as primary. It is possible, however, that a dispersed colloidal phase of an opaque uranium mineral could escape observation by thin section study, because opaque matter is least amenable to light transmission microscopy of any of the organic components of coal.

The relative concentration of opaque matter (about 1.4 sp gr) to uranium, calculated on a volumetric basis for the seven coal layers (considering elemental uranium sp gr 18.7, and uraninite sp gr 9.4), is as follows:

| (Volumetric) | TE-5 | TE-1 | TE-6 | TE-3 | TE-4 | TE-7 | TE-2 |
|---|---|---|---|---|---|---|---|
| Op. Mat./U | 1750 | 1820 | 1370 | 1800 | 3360 | 3770 | 4000 |
| Op. Mat./$UO_2$ | 780 | 810 | 610 | 800 | 1400 | 1660 | 1760 |

The very high volumetric ratios probably serve to explain not only why uranium associated with opaque matter would be invisible, but why it would also be invisible in any other organic component in which it is distributed. On volumetric basis, either as elemental uranium or as uraninite, the progression is not as uniform as it appeared when normal analytic values were compared (gravimetric uranium with volumetric opaque matter). The ratios show about a threefold range of variation.

The relative concentration of opaque matter to uranium on a gravimetric basis (assuming that pure opaque matter, the densest organic material in the coal, has a specific gravity of about 1.4) is as follows:

| (Gravimetric) | TE-5 | TE-1 | TE-6 | TE-3 | TE-4 | TE-7 | TE-2 |
|---|---|---|---|---|---|---|---|
| Op. Mat./U | 130 | 140 | 105 | 135 | 250 | 380 | 300 |
| Op. Mat./$UO_2$ | 115 | 120 | 90 | 120 | 220 | 250 | 265 |

The progression and variance is similar to the volumetric ratios previously given, but the actual ratios of weight of opaque matter to weight of uranium are naturally much lower even though the contrast still is substantial. If uranium is calculated as uraninite, there is, by weight, only 90 to 265 times as much opaque matter as uraninite present in the different layers of this coal.

This is the only coal bed known to us in which the agreement between uranium and inert organic matter is consistent even to this extent. A negative relationship or lack of a relationship is shown by other coals that have been studied in Dakota (compare especially layers 10-12, hole SD-19, Upper Mendenhall bed; Schopf, Gray, and Felix, 1955, p. 36). The inert organic matter also is exceptionally low in uraniferous coals that have been studied in Wyoming (Schopf and Gray, 1955) and in Idaho (Schopf and Gray, 1954) and the relationship is not shown in material from the "F" coal bed, North Cave Hills, discussed in a later section of the present report. Consequently, we believe the apparent relationship between inert organic material and uranium content of the "Z" bed column specimen probably is only fortuitous.

In order to determine the specific composition of translucent attritus in this coal, the component data given in table 4 were recalculated as given in table 5. Most of the translucent attritus in the layers of the "Z" bed consists of subanthraxylon and humic matter which, when taken together, are equivalent to translucent humic degradation matter of Thiessen and Sprunk (1936). Translucent humic degradation matter (reported here in two texture fractions) composes over 95 percent of the translucent attritus in all layers. Brown matter (humic material in a state comparable to semi-fusain), comprising less than one percent of the whole coal, shows a progression similar to opaque attritus and fusain. The proportional amount of fungus materials has been increased, but the values do not appear to have more significance than as presented in the preceding table 4. It appears that whatever properties can be attributed to translucent attritus in this coal must be largely determined by the properties of the subanthraxylon plus humic fractions.

Some general agreement exists between the concentration of fine textured or attrital coal and uranium content. This is a relationship that also appears in many other coal beds, usually modified by access to the coal of uranium-bearing ground water. Although the relation of attrital coal to uranium content is not quantitative, it apparently has general application. The relationship is most apparent or more pronounced in coal layers below mineral-rich (permeable?) strata, whether these form the roof of the bed or simply thin partings or layers of impurity in the coal. These observations also may be stated conversely, as a negative relationship with bands of dense woody anthraxylon, the least permeable of the layers in the coal bed. It would seem that the greatest importance must be placed on permeation of the coal by uranium-bearing ground water.

## "F" bed, North Cave Hills

The stratigraphic relations of the "F" bed to other beds in the Riley Pass area of the North Cave Hills have been mapped and shown in cross-section by Gill (1955, fig. 31, p. 156). Kepferle and Chisholm (1955) have studied the structural and ground water relations of the district. The core material we have studied was taken in Gill's "ore-grade" area on the promontory about a quarter mile northwest of the road crossing Riley Pass. Average uranium content of the "F" bed in this core (including partings) is about 0.02 percent. The underlying "E" bed, discussed in the subsequent section of this report, is much richer.

The "F" coal bed is much split by dark clay partings and impure layers. About the same total amount of thick woody pre-vitrain (31%) is present in the "F" coal bed as in the "Z" bed of the southern Slim Buttes, but it is more uniformly distributed and more evident. The pre-vitrain in thin and medium sizes (1/2 - 5 mm thick) is well represented and comprises about 16 percent of the bed. The coal shows only a trace of megascopic fusain. The top layer, soft and somewhat broken in drilling, shows evidence of weathering.

Thin sections were prepared for microscopical study of all the purer coal. Thin sections were obtained through a thickness of 1.9 feet, including a representation of over 85 percent of the coal of the deposit. Four gaps in the sequence of 26 sections represent portions that were so clayey that they did not warrant microscopical study for our present purpose.

The results of microscopical studies are summarized in the upper chart of figure 5. Over 60 percent of the coal consists of anthraxylon and it thus is a little more woody than most other beds of Dakota lignite. Some of the impure layers appear to consist of woody fragments closely set in a clayey matrix. Thin sections show that pieces of bark as well as actual wood contribute to the "woody" bands that are classed as pre-vitrain. The wood and bark apparently are all of gymnospermous type. The actual wood may contain from 5 to 20 percent red-translucent wood parenchyma resins. The bark presents a prominently striped appearance with lignified fiber bands appearing as yellowish sheets between red-translucent resinous sieve tube and phloem parenchyma layers, in which the original walls were cellulosic and not lignified. Fibrous rootlets of the type that contribute prominently to attrital coal of the

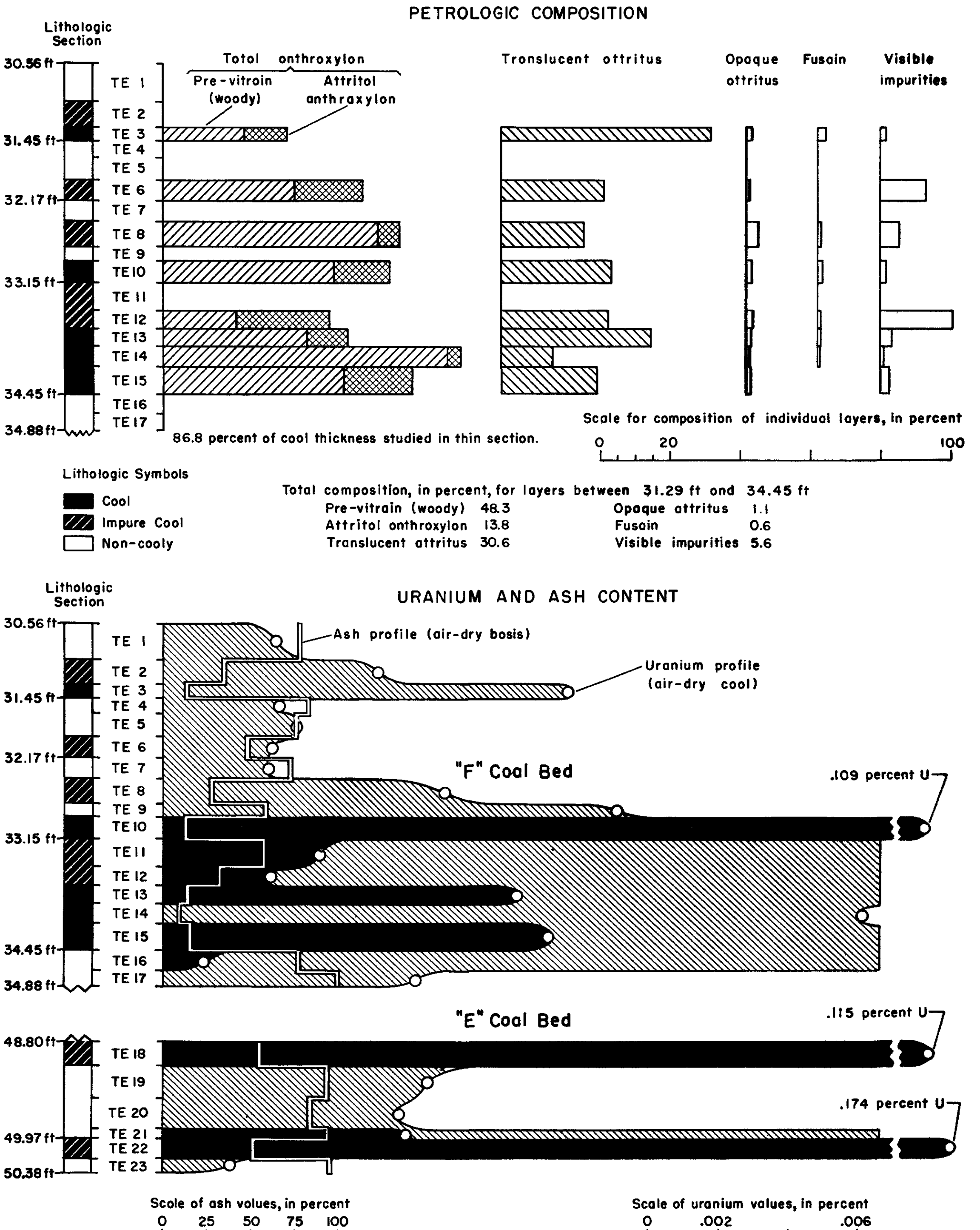


Figure 5. —Composition of uranium-bearing lignite from North Cave Hills, Harding County, South Dakota.

Mendenhall and other beds of the central Slim Buttes area are not common; somewhat larger gymnospermous roots contribute more abundantly to the attrital anthraxylon. Only about one percent opaque attritus and half a percent of fusain is present in the coal. Opaque matter is thus relatively low and a strong contrast is presented with the coals of the central Slim Buttes area that usually contain 8 or 10 percent of these relatively inert constituents (Schopf, Gray, and Felix, 1955). More than twice as much opaque matter is present in the "Z" bed of the southern Slim Buttes as is present in the core from the "F" bed of the North Cave Hills.

The same types of mineral matter are present in the "F" bed of the North Cave Hills that were reported in the "Z" bed of southern Slim Buttes. Disseminated pyrites are somewhat less abundant, perhaps owing to pyritic oxidation in some samples (cf., p. 18). In addition to pyrite, in the form of cubes, octahedra, and small aggregates, marcasite also has been identified. One lenticular aggregate from layer TE-11 shows diagnostic twinned "cocks-comb" and "spear-shaped" crystal forms. Small spherulitic aggregates of radiating crystals also are relatively common. Limonite, jarosite, and gypsum, all of which may derive from oxidation of pyrite or marcasite, are present in the upper portion of the bed. Clayey minerals are abundant in the more impure layers of the bed and dominate the four high-ash layers omitted from the petrologic composition chart (figure 5).

Analcite is present in greater quantity in the "F" bed than in the "Z" bed of the southern Slim Buttes. Spherulites and crystals of analcite are present in the associated clay as well as in the coal (p.10). In a few areas the spherulites are tightly packed to the virtual exclusion of other material. Nearly all spherulites and crystals show a prominent marginal zone of clear analcite, but the incipient spherulites, as described in the "Z" bed (p. 26), do not seem to be present. This is interpreted as indicative of a more advanced stage of analcite deposition and is consistent with greater evidence of weathering shown by chemical analyses (p. 13-20).

A special study was made from thin sections to illustrate the nature of the spherulites and nucleated crystals of analcite in the coal. Results are given in table 6 showing maximum and minimum dimensions and relative proportions of "clear" analcite (mostly of the marginal zone) and nuclear materials as they appear in sections.

Table 6. -- Size and composition of forms of analcite in the "F" coal bed.

| | Clear analcite in coal (area %) | Vertical Diameter (in mm) | | | Composition of spherulites and crystals (in percent apparent area) | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | Clear | Nuclear inclusions | | |
| | | Min. | Av. | Max. | analcite | Clay | Pyrites | Organic |
| TE-3 | .73 | .11 | .25 | .51 | 32.7 | 3.3 | .4 | 63.6 |
| TE-6 | 7.73 | .09 | .16 | .34 | 39.4 | 48.7 | 1.4 | 10.5 |
| TE-8 | 3.72 | .10 | .37 | .76 | 44.7 | 9.2 | 1.0 | 45.1 |
| TE-10 | .92 | .22 | .28 | .36 | 19.9 | 4.1 | .4 | 75.6 |
| TE-12 | 6.28 | .40 | .37 | .58 | 49.6 | 41.7 | .8 | 7.9 |
| TE-13 | 2.24 | .14 | .20 | .36 | 37.7 | 22.0 | .5 | 39.8 |
| TE-14 | .48 | .14 | .18 | .22 | 28.1 | 9.4 | -- | 62.5 |
| TE-15 | 1.47 | .18 | .27 | .36 | 31.6 | 39.5 | -- | 28.9 |
| Average | 2.9 | | .26 | | 35.5 | 22.2 | .6 | 41.7 |

The nuclei of spherulites and crystals are undoubtedly permeated with analcite so that less clay and organic matter is actually present in the nuclear areas than is evident from their appearance in thin sections. As in the "Z" coal bed, previously discussed, the clear marginal zones of spherulites commonly are asymmetrical and thicker on the edges parallel to the bedding. In a few instances nucleated spherulites are twice as broad as high so that the horizontal diameter may exceed a millimeter. It is noteworthy that TE-14 contains the greatest amount of anthraxylon, the least amount of analcite, and that the spherulites present in this layer are small in the size range. The only smaller examples are present in the very impure layer, TE-6, where they are very abundant.

The habit of occurrence of analcite in the "F" coal bed is similar to that of the "Z" bed except that somewhat larger spherulites and crystals are present in greater abundance. It would appear that more sodium, aluminum, and silicon ions have been supplied by ground water solutions permeating the coal at the North Cave Hills location than at the sample location in the southern Slim Buttes.

Uranium and ash content of the 17 samples from the "F" coal bed and associated rocks, together with values for the "E" coal bed that will be discussed later, are shown graphically in the chart at the bottom of figure 5. As in figure 4 (see p. 23) the black bars represent double values wherever they overlap crosshatched portions of the uranium profile. At normal scale the tips of the black bars all would be displaced to the right a distance corresponding to about 0.021 percent U. The relations of uranium to woody and impure layers are less clear than for the "Z" coal bed because of the additional complexity of partings and impurities. If one regarded layer TE-10 as the top of the coal bed, the distribution of uranium might well be considered as top preferential. Certainly it is located below a layer of high ash content. Layer TE-10 is the only layer with more than 0.1 percent uranium. Layers TE-13 and TE-15 have little more than a fourth as much. However, the layer intervening between TE-13 and TE-15 contains about 80 percent pre-vitrain and presents the strongest contrast with respect to amount of woody material to be found in this coal bed. It also has the lowest ash content and the lowest uranium content of the more coaly sequence between depths of 32.88 and 34.45 feet (layers TE-10 to TE-15). It thus appears to show the same negative relationship to uranium concentration that was illustrated by the dominantly woody layers of the "Z" bed in the southern Slim Buttes. The amount of fusain and opaque attritus in this bed is so low that its effects probably are negligible; there is no apparent relationship with the occurrence of uranium.

Results of detailed study of the composition of layers of the "F" coal bed are given in table 7. The layers have been arranged according to uranium content as in table 4, which represents layers from the "Z" bed. The data given in these tables are comparable and will be compared with reference to selected details on a later page. In view of the unusually high concentration of uranium in layer TE-10, it should be easy to determine any organic components that have a special affinity for this element. None are apparent, however, and the parallelism of opaque attritus and fungal materials, that was observed in the coal from southern Slim Buttes, is wanting. It must be concluded that whatever effects organic composition may have on uranium concentration have become subordinated to the relations of permeability and position within the bed.

Table 7. -- Composition (area percent) of uraniferous coal layers from North Cave Hills, Harding County, S. Dak. Layers presented in order according to their uranium content.

| Constituents or Components | TE-10 | TE-15 | TE-13 | TE-12 | TE-14 | TE-3 | TE-8 | TE-6 |
|---|---|---|---|---|---|---|---|---|
| Anthraxylon: | | | | | | | | |
| Coarse | 48.35 | 51.84 | 40.93 | 20.72 | 80.57 | 23.44 | 61.20 | 37.83 |
| Attrital | 15.91 | 19.27 | 11.29 | 27.26 | 3.76 | 12.04 | 6.02 | 18.92 |
| Total | 64.26 | 71.11 | 52.22 | 47.98 | 84.33 | 35.48 | 67.22 | 56.75 |
| TRANSLUCENT ATTRITUS | | | | | | | | |
| Subanthraxylon | 3.25 | 2.04 | 1.65 | 6.72 | 1.44 | 5.01 | 3.54 | 7.45 |
| Humic Matter | 26.20 | 22.94 | 38.24 | 21.93 | 12.39 | 52.35 | 18.91 | 20.96 |
| Total | 29.45 | 24.98 | 39.89 | 28.65 | 13.83 | 57.36 | 22.45 | 28.41 |
| Red Att. Resins | .48 | .82 | .65 | .33 | .27 | .41 | .21 | .64 |
| Cuticle | .05 | .03 | — | — | — | — | — | — |
| Spores | .19 | .13 | .39 | .16 | .20 | .28 | .10 | .08 |
| Yellow Att. Resins | .15 | .11 | .89 | .19 | .15 | .12 | .18 | .14 |
| Waxy Amorphous | .34 | .26 | .53 | .36 | .12 | .34 | .25 | .32 |
| Total | .73 | .53 | 1.81 | .71 | .47 | .74 | .53 | .54 |
| Fungal Phyterals | .05 | .07 | .03 | — | .02 | .14 | .01 | .05 |
| Brown Matter | .36 | .25 | .45 | .37 | .20 | .41 | .52 | .14 |
| Total Trans. Attritus | 31.07 | 26.65 | 42.83 | 30.06 | 14.79 | 59.06 | 23.72 | 29.78 |
| Opaque Attritus | 1.40 | .17 | .77 | 1.50 | .03 | 1.38 | 2.89 | .36 |
| Microfusain | .72 | — | .54 | .43 | .05 | 2.24 | .16 | — |
| Megafusain | .65 | — | — | — | — | — | .34 | — |
| Total | 1.37 | — | .54 | .43 | .05 | 2.24 | .50 | — |
| Disseminated Pyrites | .71 | .18 | .39 | .62 | .12 | .75 | .49 | .67 |
| Transparent Minerals | .09 | .04 | — | .24 | .01 | .04 | .19 | .16 |
| Analcite (Analcime) | .92 | 1.47 | 2.24 | 6.28 | .48 | .73 | 3.72 | 7.73 |
| Clayey Minerals | .18 | .38 | 1.01 | 12.89 | .19 | .32 | 1.27 | 4.55 |
| Total | 1.90 | 2.07 | 3.64 | 20.03 | .80 | 1.84 | 5.67 | 13.11 |
| Total | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |

Table 8. -- Composition (area percent) of translucent attritus in the uraniferous coal layers from North Cave Hills, Harding County, S. Dak.

| Components | TE-10 | TE-15 | TE-13 | TE-12 | TE-14 | TE-3 | TE-8 | TE-6 |
|---|---|---|---|---|---|---|---|---|
| Subanthraxylon | 10.48 | 7.66 | 3.86 | 22.34 | 9.74 | 8.48 | 14.92 | 25.03 |
| Humic Matter | 84.33 | 86.07 | 89.28 | 72.96 | 83.75 | 88.64 | 79.73 | 70.39 |
| Total | 94.81 | 93.73 | 93.14 | 95.30 | 93.49 | 97.12 | 94.65 | 95.42 |
| Red Attrital Resins | 1.54 | 3.10 | 1.51 | 1.11 | 1.84 | .69 | .88 | 2.13 |
| Cuticle | .16 | .10 | — | — | — | — | — | — |
| Spores | .61 | .48 | .92 | .53 | 1.32 | .48 | .44 | .25 |
| Yellow Attrital Resins | .48 | .41 | 2.07 | .62 | 1.04 | .21 | .75 | .48 |
| Waxy Amorphous | 1.09 | .97 | 1.24 | 1.20 | .81 | .57 | 1.05 | 1.08 |
| Total | 2.34 | 1.96 | 4.23 | 2.35 | 3.17 | 1.26 | 2.24 | 1.81 |
| Total Fungal Phyterals | .16 | .28 | .06 | — | .12 | .24 | .04 | .16 |
| Brown Matter | 1.15 | .93 | 1.06 | 1.24 | 1.38 | .69 | 2.19 | .48 |
| Total | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Percent of Layer Represented by Translucent Attritus | 31.07 | 26.65 | 42.83 | 30.06 | 14.79 | 58.79 | 23.72 | 29.78 |
| Percent Uranium | .1094 | .0314 | .0305 | .0235 | .0197 | .0115 | .0080 | .0031 |

The specific composition of translucent attritus is shown in table 8. Translucent humic degradation matter (subanthraxylon plus humic matter) accounts for over 93 percent of the translucent attritus in all layers of this coal. In this respect the "E" bed is very similar to the "Z" bed of the southern Slim Buttes.

"E" bed, North Cave Hills

Samples of the "E" coal bed were obtained from the same core hole as samples of the "F" bed. Stratigraphic relations of the "E" bed were established by Gill (1955, p. 156). Although he did not indicate that the "E" bed was present at this drill hole location, it is represented by two thin impure layers of coal with high uranium content. Slightly less than half a foot of coal is present, but this coal has an average uranium content of 0.14 percent.

A hard, indurated, iron-stained layer, with some clay and siltstone, separates the two thin layers of coal. On casual inspection the hard layer appears to be sandstone, but microscopic examination reveals that quartz probably is not present and that the rock is porous and consists chiefly of cemented sideritic granules, 0.3 - 0.6 mm in diameter, with limonitic coatings or alteration layers about 40 - 60 microns thick. Interstitial soft gray clay occurs in minor amount between the granules. When the sideritic grains are etched with hot hydrochloric acid, a clayey residue remains that shrinks drastically on drying. The siderite probably was deposited during diagenesis before the clay matrix was consolidated; the layer may be permeable now because the granular and porous sideritic deposits were resistant to normal compaction. It seems evident that this layer has served as an aquifer and oxidation channel which was responsible for advanced underground weathering of the adjacent coal.

These two layers of highly uraniferous coal were pulverized in drilling so that a relatively small amount remained and none of the pieces were large enough to show lithologic relationships. This necessitated a revision in methods of study and limited both the amount of information and the reliability of information that could be obtained. The coal has been weathered to the point where some of it appeared soluble in the drilling fluid as the core was taken (Roy C. Kepferle, field notes). Weathering evidently also affected the sandy and silty rocks above and below the coal and some fragments of these rocks and of the sideritic parting contaminate the pulverized coal samples.

The pulverized coal was wrapped separately and was wet when received at the laboratory. It was dried until surface water evaporated and a split from each small quantity of coal was bottled as a sample for chemical analysis. The remaining coal was dried further and used for radioactivity determinations. These samples were again split (1) for ash, uranium, and spectrographic analyses and (2) the remainder used for microscopic study.

The results of uranium and ash determinations for layers of the "E" coal bed and the intervening parting are shown in relation to the "F" bed at the bottom of figure 5 (p. 36).

Microscopical information is based on thin sections and polished surface sections prepared by embedding the granular coal in hot Schneiderhöhn's mixture, a resinous medium recommended by German coal microscopists (Stach, 1949). The dried coal was extremely friable and the largest lumps, which appeared to be about 1/4 inch in diameter, were used for sections. These lumps proved to be aggregates of much smaller pieces that had stuck together on drying. Orientation of particles was at random and only small areas were thin enough to permit critical study. The components appear to be generally similar to those of the other coals of this area. Polished surface sections were prepared from additional granulated coal embedded in blocks of the Schneiderhöhn medium. The latter procedure is more conservative of a limited quantity of pulverized material.

Quantitative estimation of macerals present in the polished surface sections was attempted. Considering the nature of the samples, probably little reliance should be placed on the results, tabulated below, as an indication of the original composition of the coal. They are, however, indicative of the nature of the material represented in other correlated analyses, and they are subject to interpretation.

Table 9. — Composition (area percent) of pulverized "E" coal bed samples.

| | Vitrinite | Exinite | Inertinite | Pyrites | Clay | Analcite | Contaminants |
|---|---|---|---|---|---|---|---|
| Layer TE-18 (U = 0.115%) | 54.7 | 0.2 | 19.2 | T | 12.7 | 9.3 | 3.9 1/ |
| Layer TE-22 (U = 0.174%) | 60.5 | 0.8 | 5.6 | 0.1 | 9.4 | 8.5 | 15.1 1/ |

1/ Consists of sand and sideritic fragments.

Vitrinite includes all materials given as humic matter, subanthraxylon, attrital anthraxylon, and coarse anthraxylon or pre-vitrain, on other tabulations and charts of this report. No differentiation of various textures is implied in the identification of vitrinite. The samples available from the "E" bed do not permit textures to be quantitatively differentiated.

Some woody tissue structure is visible in the coal fraction identified with vitrinite from the "E" bed, but most of the vitrinite observed in polished surface fragments lacks visible tissue configuration. Much of it appears to have the fine noncellular texture equivalent to humic matter or subanthraxylon. This suggests that, in general, the two layers of the "E" bed consist mostly of attrital coal. The percentages of vitrinite shown in table 9 may be too low because, if solution of part of the weathered coal has occurred, it would be largely at the expense of the vitrinized fraction.

Exinite includes waxy materials of low reflectance that are generally presumed to be correlative with spore and pollen coats and similar substances that appear yellow-translucent by transmitted light. There is some danger of cracks and some other types of low reflectance areas being misidentified as exinite by polished surface methods of study. Apparently reflectance methods generally yield a somewhat higher exinite determination than transmission methods do (Parks and Teichmuller, 1955, p. 14). In any event, it appears from table 9 that exinite is present in low concentration in the "E" bed samples and that its concentration is of the same magnitude as shown by waxy materials in table 7, representing the "F" bed, and in table 4 representing layers of the "Z" coal.

Inertinite is a maceral that is substantially correlative with opaque matter (opaque attritus plus fusain) as given in other petrologic analytic tabulations in this report. It has become customary to speak of inertinite when polished surface studies are used for identification, and of opaque matter when constituents are identified by use of transmitted light. It is very probable that some systematic differences exist between determinations made by the different analytic procedures, although the differences are not great enough to alter a general evaluation of the entities. Inertinite and opaque matter are both recognized as more resistant to oxidation than the other organic materials in coal, in having greater fixed carbon, lower volatile matter and lower inherent moisture.

Whether either layer of the "E" bed naturally contains as much inertinite as shown in table 9, is questionable. It has been pointed out that some of the more reactive coal constituents were soluble in the drilling fluid. Proportionally much less inertinite would be lost by effects of solubility. Concentration on inertinite might have enhanced the uranium content of these samples if uranium has a relationship with opaque matter, as suggested by the "Z" bed of the southern Slim Buttes. It seems clear that the relationship does not apply, however, as the upper layer (TE-18) with 0.115 percent uranium appears to have over three times as much inertinite as the lower layer (TE-22) where the uranium content is 0.174 percent. If contact with uraniferous ground water was primarily responsible, these relations are reasonable, for the lower layer probably would have been exposed to more of the water supplied through the sideritic aquifer.

The same minerals are seen in the pulverized samples that were present in the "F" and in the "Z" coal beds. In addition, limonite, that appeared to fill some natural (?) cracks, and contaminating sand and siderite fragments are present in the samples. Isolated analcite spherulites and crystals, with large clayey and carbonaceous nuclei, are abundant. The nuclear inclusions of the spherulites provide the largest examples of relatively undisturbed lithology that these samples afford. From the number of spherulites with clayey nuclei, we infer that both coal layers are relatively impure. The minor traces of pyrites are mostly enclosed within analcite nuclei where they have been protected from oxidation. Nevertheless, in spite of evident mineral impurities, it seems certain that these coal layers do not have as much inherent ash as the analyses show. The contaminating materials must be extraneous and some of the analcite may be from clay layers that also are essentially extraneous to the coal.

# DISCUSSION

Denson, Bachman, and Zeller (1954, p. 52-3) describe the common occurrence of analcite crystals and rosettes in weathered lignite. They suggest that the analcite was derived from the overlying volcanic materials and introduced into the lignite by ground water subsequent to coalification. Our observations are fully in agreement with this interpretation. Although both uranium enrichment and deposition of analcite in coal are probably a result of ground water circulation, neither is necessarily contingent upon the other. Layers richest in uranium usually do not contain the most analcite. However, a more general relationship may exist in the Dakota area since both aluminum silicates and uranium might have been derived from overlying tuffaceous rocks.

Semiquantitative spectrographic determinations made on many of these samples require further study in relation to evidences of ground water movement that have been developed in this investigation. In the virtually unweathered portion of the "F" coal bed, the relationship of cobalt and molybdenum, mentioned in an earlier report (Schopf, Gray, and Felix, 1955), is maintained, but neither shows an apparent relationship with uranium. Germanium shows a pronounced "bottom preferential" occurrence in the base of the "F" bed and also has been detected in the highly uraniferous layer TE-10. Potassium, which has generally not been detected in Dakota lignitic samples, is present in excess of 0.5 percent in many samples from the North Cave Hills core. It shows a trend of negative correlation with carbonaceous material. Arsenic has been detected in six carbonaceous samples, most of which showed some indication of weathering.

Examination of figures 2, 4, and 5 show that all relatively high concentrations of uranium are limited to coaly material. The purest coal, however, does not have the highest uranium content. The non-coaly carbonaceous or essentially non-carbonaceous samples are notably less uraniferous. Table 10 shows a summary of the average relationships of ash and uranium content, as exemplified by this series of samples.

Table 10. -- Uranium content of different types of uraniferous material from North Cave Hills and southern Slim Buttes, Harding County, S. Dak. (All averages have been weighted according to thickness of individual samples.)

| | Number of samples | Percent ash or ignition residue (average & range) | Percent U in ash or ignition residue | Ppm 1/ uranium in sample; dry basis (110°C) |
|---|---|---|---|---|
| Coal; less than 10% ash | 3 | 8.8 ( 7.3 - 9.8) | 0.139 | 120 |
| Coal with 10 to 18% ash | 7 | 14.9 (12.5 - 17.1) | 0.408 | 610 |
| (Inadequate representation of samples containing 18 to 33% ash) | | | | |
| Impure coal & coaly shale; 33 to 60% ash | 8 | 47.3 (33.8 - 59.2) | 0.100 | 470 |
| Clay & shale (< 40% LOI 2/) | 7 | 82.2 (74.3 - 93.7) | 0.008 | 65 |
| Sandstone & siltstone (< 40% LOI) | 3 | 88.7 (83.8 - 95.0) | 0.010 | 90 |
| General average | | 49.2 ( 7.3 - 95.0) | 0.065 | 320 |

1/ Ppm = parts per million; 0.01% = 100 ppm.
2/ LOI = loss on ignition.

Probably it is not coincidental that the three samples with less than 10 percent ash contain only a moderate amount of uranium. All three are from dominantly woody layers that appear dense and impermeable and contain very little analcite. There is good reason to believe that these layers were less permeable to ground water from which analcite might be deposited or from which uranium could be derived.

Samples with an intermediate ash content of 10 to 18 percent are those with the greatest amount of uranium. These consist largely of attrital coal and have at least a moderate amount of analcite. To some extent analcite adds to the ash content in the coal, but the greatest ash and analcite concentrations are more likely to occur above the layers with greatest uranium concentration. Possibly, under natural conditions, uranium does not combine with the coal and become "fixed" as readily as the analcite is deposited. It seems likely that high uranium content may also be associated with material having more than 18 percent ash, but samples in the range of 18 to 33 percent ash content were few and questionable.

Impure coal and coaly shale with a range of ash from 33 to 60 percent has less uranium than samples with lower ash content. The contrast is exaggerated if values are regarded on the basis of the uranium in the ash, because the ash content is disproportionately greater. Considered in relation to the weight of the dry sample, the contrast is not nearly as great. This indicates that highly impure coal serves nearly as well as a host for uranium as coal of moderate impurity. The more highly impure layers, however, are not promising as a source of uranium.

Clay and shale layers with ignition residues exceeding 60 percent show the least concentration of uranium. Sandstone (sideritic) and siltstone samples are only slightly higher even when closely associated with coal containing a large amount of uranium.

The tendency for coal of intermediate ash content to contain greater amounts of uranium has previously been noted in coal from the Red Desert in Wyoming (Schopf and Gray, 1955). The relationship to overlying tuffaceous rocks, generally present in the Dakota region, is not known in the Red Desert area and relative position within the coal bed is evidently less important there than it is in the areas of uraniferous Dakota lignite. The definite, but more subdued, relationship of uranium with coal of lesser purity in Dakota suggests that, so far as the coal is concerned, similar geologic processes may be operative here, as in the Red Desert, although differences in geologic setting may cause an apparent contrast in the nature of the occurrences. Lack of an evident top-preferential distribution of uranium in Dakota high grade deposits, and a stronger apparent relationship with ash content, may thus hold more general significance.

The relationship of moderate ash and radioactivity apparently also exists for most "normal" coal beds where less than 10 ppm uranium is present. Newmarch (1950) and Davidson and Ponsford (1954) indicate that the radioactivity of coal beds is directly proportional to ash content. This conclusion also is borne out by Kronstadt's (1952) study of the radioactivity of coal ash from nearly 100 operating mines in the United States. The ash content of these coals was generally less than 20 percent and the eU, calculated on a dry coal basis, is generally less than 7 ppm. However, even this small indication of uranium showed a proportional decrease with decreasing ash content.

Studies of high-grade uraniferous coal indicate little relationship between organic petrologic composition and uranium concentration that can be entirely divorced from consideration of position and permeability.

No quantitative relationships are apparent and broader relationships largely disappear if coal layers are considered apart from their normal position with respect to each other and to possible sources of mineralization. An earlier study (Schopf and Gray, 1955) also indicated a possible relationship between the abundance of waxy matter and uranium content of coal from the Red Desert area in Wyoming and the Goose Creek area in Idaho. This possibility is practically excluded from present consideration because of the lack of these materials in any significant quantity from coals of the Dakota area. The series of relatively high-grade uraniferous coal samples has included so great a range of uranium concentration that the affinity of uranium for any single organic component or constituent of this coal seems improbable.

Relatively inert organic constituents (opaque attritus and fusain) probably do not favor uranium emplacement in low rank coal, although studies of the "Z" bed may be interpreted differently. Perhaps the best general evaluation of these materials is that they do not appear, at least, to militate strongly against it. It is possible that in some instances relatively inert organic constituents could favor uranium emplacement in low rank coal by improving its permeability. Lack of evidence favoring a highly specific relationship for inert organic materials and their characteristically dispersed occurrence in relatively minor or small quantity, leads us to discount their possible significance in this connection.

The physical character of coal constituents evidently has a direct bearing on uranium emplacement. The denser, least permeable, low-ash layers generally have the least uranium if access to uranium-bearing ground water is approximately equal. If beneficiation procedures ever are considered, it should not be difficult, provided the coal is not too badly weathered, to segregate low-ash and low-uranium woody bands from more highly uraniferous attrital coal of these deposits. Attrital coal, with somewhat greater inherent ash, is evidently more permeable and best adapted to retention of the uranium carried by ground water.

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# APPENDIX

## MEGASCOPIC DESCRIPTION OF MATERIAL STUDIED

1. "Z" coal bed, southern Slim Buttes, Harding County, S. Dak.

Stratigraphic occurrence and location: In the Ludlow member of the Fort Union formation at a prospect pit in the southeastern extension of the Slim Buttes, in the NE 1/4, SW 1/4 sec. 2, T. 16 N., R. 5 E.

| Depth | Description | Sample Number |
|---|---|---|
| 28.25' (top of coal in shipment) | | |
| | Coal, sparsely to moderately thin-banded, slightly pyritic, sparsely streaked with thin fusain lenses in the upper half; 7/8" woody band near the base. 17.4 P/M/G. | TE-1 |
| 28.53' | | |
| | Coal, 65 to 70% woody, thick-banded, slightly pyritic with some fusain flecks in the more attrital upper half; 1/4" fusain at the base. 6.3 P/M/G. | TE-2 |
| 28.8' | | |
| | Coal, sparsely thin- to medium-banded, with three pyritic nodules, 1/2" to 3/4" in diameter, near the top of the layer. 8.05 P/M/G. | TE-3 |
| 29.04' | | |
| | Coal, sparsely thin- to medium-banded, pyritic in upper half; lower part, more impure than the coal above, includes 1/8" fusain lens. 7.85 P/M/G. | TE-4 |
| 29.20' | | |
| | Coal, sparsely thin-banded, with numerous fusain flecks and lenses throughout; thin pyrite lens near bottom. 24.9 P/M/G. | TE-5 |
| 29.29' | | |
| | Coal, sparsely thin- and medium-banded, more woody in top half, slightly pyritic with some fusain lenses and flecks. 13.75 P/M/G. | TE-6 |
| 29.61' | | |
| | Coal consists of one thick woody band with a few fusinized streaks and 1/8" attrital coal at the base. 7.85 P/M/G. | TE-7 |
| 29.86' (bottom of coal in shipment) | | |

2. Including "F Rider", "F", and "E" coal beds, North Cave Hills, Harding County, S. Dak. ("NX", 2-1/8" diam. core).

Stratigraphic occurrence and location: In the Tongue River member of the Fort Union formation on the Homestake Mining Company Riley #4 claim, near the center of the NW 1/4, NW 1/4 sec. 26, T. 22 N., R. 5 E.

| Depth | Description | Sample Number |
|---|---|---|
| 22.23' | (top of core in shipment) | |
| | Claystone, olive gray with analcite spherulites; .02' black carbonaceous streak at top. CGL 1, 0.32 P/M/G. | (Note: Upper layers of core were not submitted for special analyses.) |
| 22.52' | | |
| | Claystone, olive gray with analcite spherulites and crystals; 0.03' black carbonaceous streak at top and 0.08' carbonaceous zone at bottom. CGL 2, 0.46 P/M/G. | |
| 22.75' | | |
| | Loss in coring. | |
| 24.00' | | |
| | Claystone, medium to dark gray with analcite spherulites and sparse coaly streaks. CGL 3, 0.32 P/M/G. | |
| 24.41' | | |
| | Claystone, medium to dark gray with analcite spherulites and crystals; 0.13' attrital coal, slightly pyritic, at base. CGL 4, .27 P/M/G. | |
| 24.76' | | |
| | Coal, highly impure, sparsely thin-banded, with streaks of black carbonaceous clay, a few analcite spherulites and crystals, and small pyritic aggregates. CGL 5, 0.40 P/M/G. | |
| 25.0' | | |
| | Core not sent. | |
| 26.2' | | |
| | Claystone, dark gray to black, highly carbonaceous and coaly with abundant spherulites. CGL 6, 0.33 P/M/G. | |
| 26.5' | | |
| | Core not sent. | |
| 27.2' | | |
| | Loss in coring. | |
| 30.0' | | |
| | Claystone, medium gray, slightly carbonaceous, with analcite spherulites and crystals locally abundant, and flecks of limonite. CGL 7, 0.64 P/M/G. | |
| 30.22' | | |
| | Claystone, medium gray, slightly to moderately carbonaceous, analcite spherulites locally abundant. CGL 8, 0.61 P/M/G. | |
| 30.56' | | |

(Cont'd. on next page)

| Depth | Description (Cont'd.) | Sample Number |
|---|---|---|
| 30.56' | | |
| | Claystone, dark gray to black, highly carbonaceous, analcite spherulites common. CGL 9, 1.26 P/M/G. | TE-1 |
| 31.0' | | |
| | Coal, attrital and impure, with sparse analcite crystals and much finely disseminated pyrite. CGL 10, 1.65 P/M/G. | TE-2 |
| 31.29' | | |
| | Coal, moderately medium- to thick-banded, with a few resin blebs up to 4 mm in greatest dimension and sparse analcite crystals. (Badly broken in drilling.) CGL 11, 4.31 P/M/G. | TE-3 |
| 31.45' | | |
| | Claystone, medium to dark gray, carbonaceous, with coaly streaks and lenticles; analcite spherulites and crystals abundant. CGL 12, 1.38 P/M/G. | TE-4 |
| 31.66' | | |
| | Claystone, as above, but more carbonaceous; bottom .07' black, with abundant analcite spherulites and crystals. CGL 13, 1.42 P/M/G. | TE-5 |
| 31.92' | | |
| | Coal, impure, and claystone, carbonaceous, black, with analcite spherulites and crystals. CGL 14, 1.09 P/M/G. | TE-6 |
| 32.17' | | |
| | Claystone, dark gray to black, highly carbonaceous, with abundant spherulitic analcite and some disseminated pyrite. CGL 15, 1.63 P/M/G. | TE-7 |
| 32.42' | | |
| | Coal, 60-65% woody, thick-banded, with black claystone streaks and analcite spherulites and crystals. CGL 16, 5.08 P/M/G. | TE-8 |
| 32.72' | | |
| | Claystone, black, coaly, with analcite spherulites and crystals; 0.03' of coal at base. CGL 17, 7.99 P/M/G. | TE-9 |
| 32.88' | | |
| | Coal, about 50% woody, medium- to thick-banded, with spherulitic analcite. CGL 18, 28.59 P/M/G. | TE-10 |
| 33.15' | | |
| | Coal, impure, sparsely thin-banded, with analcite spherulites and crystals; marcasite lens .02' thick at base. CGL 19, 10.00 P/M/G. | TE-11 |
| 33.49' | | |
| | Coal, impure, moderately medium-banded with one .02' woody band near middle; spherulitic analcite and crystals. (Badly broken in drilling.) CGL 20, 7.17 P/M/G. | TE-12 |
| 33.67' | | |

(Cont'd. on next page)

| Depth | Description (Cont'd.) | Sample Number |
| --- | --- | --- |
| 33.67' | | |
| | Coal, about 40% woody, medium- to thick-banded, with analcite spherulites and crystals. CGL 21, 9.40 P/M/G. | TE-13 |
| 33.91' | | |
| | Coal, about 80% woody, thick-banded, with few analcite spherulites and crystals. CGL 22, 7.25 P/M/G. | TE-14 |
| 34.15' | | |
| | Coal, about 50% woody, medium- to thick-banded, with analcite spherulites and crystals. CGL 23, 5.53 P/M/G. | TE-15 |
| 34.45' | | |
| | Claystone, dark gray and silty to carbonaceous and coaly, spherulites numerous in the bottom half. Scattered spores are visible with hand lens. CGL 24, 3.53 P/M/G. | TE-16 |
| 34.68' | | |
| | Claystone, dark gray and silty to carbonaceous and coaly, with a few spherulites and one pyritic nodule near base. CGL 25, 2.45 P/M/G. | TE-17 |
| 34.88' | | |
| | Core not sent (interval includes 2' loss in drilling). | |
| 48.8' | | |
| | Coal, impure, weathered (water soluble in part) with numerous analcite spherulites. (Core pulverized in drilling.) CGL 26, 37.11 P/M/G. | TE-18 |
| 49.1' | | |
| | Claystone (above) and siltstone, medium gray, with numerous spherulites in the upper part; lower .13' iron stained. CGL 27, 2.24 P/M/G. | TE-19 |
| 49.48' | | |
| | Siderite, limonitic, porous, hard, with granular texture. CGL 28, 1.71 P/M/G. | TE-20 |
| 49.85' | | |
| | Siltstone, clayey, medium gray, carbonaceous and iron stained, with spherulitic analcite. CGL 29, 6.38 P/M/G. | TE-21 |
| 49.98' | | |
| | Coal, impure, weathered (water-soluble in part) with numerous analcite spherulites. (Core pulverized in drilling.) CGL 30, 34.03 P/M/G. | TE-22 |
| 50.20' | | |
| | Sandstone, fine- to medium-grained, cream colored, with sparse analcite crystals. CGL 31, 0.73 P/M/G. | TE-23 |
| 50.38' (bottom of core in shipment) | | |