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THE CONTRACT PROGRAM TO APRIL 1, 1954

by

Paul F. Kerr

The report is intended to cover studies conducted under contract AT(30-1)702 between the Division of Raw Materials, Atomic Energy Commission and Columbia University. A large part of the work for the current year will be covered in a report on the alteration and uranium mineralization at Marysvale, Utah, soon to be submitted for publication. Both a geologic map and a corresponding map showing the more significant alteration features are nearing completion. The descriptive and analytical data are also being assembled to accompany the maps. The Marysvale phase of the contract work, which has embraced a three year period, is now in its final stages.

The current Annual Report for the period up to April 1, 1954 is divided into two parts. Part I deals with field studies and mineralogical data derived from the laboratory examination of suites collected during the previous summer.

Two studies included in Part I are only to a small extent a result of the Contract Project. These cover a description of a significant and unusual mineral nodule from Temple Mountain, Utah, prepared with the assistance of Mr. Davis Lapham, and a preliminary report on the uranium mineralization at the Carlile deposit in the northern Black Hills area of Wyoming by Mr. Marc Bodine, Jr.

Studies concerning the more prevalent oxidized uranium minerals of the veins at Marysvale, prepared with the aid of Miss P. K. Hamilton, are also included in Part I of this report.

Extended investigations in which Mr. Philip Bethke has assisted involve the origin of uranium in the sedimentary series which occur southwest of Marysvale in the Tushar Range.
In the Marysvale field studies and in the concurrent laboratory phase of the contract work generous assistance has been received from many sources. Various members of the Division of Raw Materials of the Atomic Energy Commission have been most helpful in assisting the work of the project. Dr. Philip L. Merritt, Dr. Donald L. Everhart and Mrs. Muriel Mathez of the New York Office; Mr. Ernest Thurlow of the Salt Lake Office, and Mr. Stephen Steinhauser of the Richfield Office have been particularly concerned with the contract work, and through their ever cheerful cooperation have aided the study in many ways.

Part II concerns the synthesis of pitchblende, X-ray studies of the crystallites in both natural and artificial pitchblende, and the nature of the synthetic pitchblende produced.
REPORT ON A NODULE FROM TEMPLE MOUNTAIN, UTAH

by
Paul F. Kerr and Davis M. Lapham

ABSTRACT

This report is based on the study of a nodule from the collapse area at Temple Mountain, Utah. The following minerals were found to be present: quartz, sphalerite, pyrite, native arsenic, thucholite, and realgar. The abundance of native arsenic in the specimen is a matter of interest. Because of the bearing the presence of this mineral may have on the origin of the Temple Mountain collapse area, the X-ray data have been compared with native arsenic from other localities.

DESCRIPTION OF NODULE

In November, 1953, one half of a sliced nodule was submitted by Dr. Thomas W. Mitcham of the A. E. C. Office at Grand Junction, Colorado, to the senior author at Columbia University for examination. The nodule had been found within the uranium ore area at Temple Mountain in the "collapse" area and the nodule was radioactive.

The specimen is ellipsoidal, measuring 1-3/4" long by 1-5/16" wide by 5/8" in depth (Fig. 1). It is composed of three rings of differing minerals or groups of minerals which will be described in detail separately.

The inner area is distinguished by its predominantly brass yellow due to pyrite. The minerals of this section were identified with the ore microscope on the polished surface of the nodule. The following tests were applied to the pyrite under the ore microscope:

1. Optical character -- isotropic
2. Color -- cream; indicative of pyrite
3. Application of HNO₃ -- blue stain on pyrite
4. Application of FeCl₃, KCN, and HCl -- negative

The other predominant mineral in this area is quartz. One additional mineral, believed to be sphalerite, was identified by its strong internal reflection, transparent brown color, and isotropic optical character. This mineral was in insufficient amounts for X-ray identification.
The middle ring is silver-gray and has a hardness of 3.5. Upon heating this material in a closed tube, a black to silver-gray sublimate of metallic arsenic was formed. When the bottom of the closed tube was broken and the metallic mirror volatilized by additional heating, the characteristic garlic odor of arsenic was obtained. An X-ray photograph of this material was then taken. Upon comparison with the Hanawalt Card Index, the mineral was found to be native arsenic. As additional proof, X-ray photographs of known native arsenic from the Hartz Mts. and from Echizen Province, Japan, were made and compared with the Temple Mt. material. Close agreement of the lines established that the Temple Mt. material was native arsenic (see Table 1). As a more accurate measurement of the lines and their intensities was thought desirable, X-ray spectrometer graphs were made of the Japanese arsenic and the Temple Mt. arsenic. Again, a comparison of the results verified the presence of native arsenic in the nodule (see Table 2). The starred lines in Table 2 are listed as probable impurities in native arsenic. It is worth noting that the Temple Mt. arsenic is free from the usual impurities. The one starred line in the Temple Mt. material is the "100" intensity line of quartz with which the arsenic is closely associated in the nodule.

The outer section of the nodule is dull black. An X-ray pattern of this material yields quartz lines. However, upon inspection with the binocular microscope, several other minerals were found scattered throughout this part of the nodule. The most striking material was the red mineral found only on the curved outer surface of the nodule. Positive chemical tests for arsenic and sulfur combined with an X-ray photograph of the material showed it to be the unstable arsenic sulfide, realgar. The X-ray pattern contained no orpiment lines. Comparison of this photograph with other X-ray photographs of realgar and orpiment confirmed the identification.

Since the Geiger counter showed that the outside of the nodule was radioactive while the inside along the polished surface yielded negligible radioactivity, and since the realgar was not the source of the radioactivity, the surface was re-examined for the presence of uranium. Black nodular masses from 0.1 mm. to 0.3 mm. in diameter were noted scattered through this outer section. All exhibited a coal-like appearance, a conchoidal fracture, and a hardness just under four. These characteristics fit Ellsworth's description of thucholite given in 1928. In addition, thucholite, or asphaltite, which is a similar material but low in thorium content, is known to exist at Temple Mt. This material was carefully extracted under the binocular microscope. Sufficient material was obtained for an X-ray pattern. Three distinct sets of diffraction lines were observed. Two were identified as due to quartz and native arsenic. The third set did not
correspond to any lines given in the Hanawalt Index nor to any patterns on file at Columbia University. To determine this material, an X-ray photograph and an X-ray spectrometer graph of known thucholite from Ontario, Canada and an X-ray photograph of a sample of another Temple Mt. specimen resembling thucholite were made. The lines suggest that thucholite produced the unknown third set of lines in the original photograph. Since thucholite is a uranium-thorium-rare earth bearing hydrocarbon of variable composition, all of the lines from these patterns are not present in each sample. However, there are four lines which appear consistently. Upon comparison with the Hanawalt Index and with patterns of uraninite on file, it was found that these lines are due to uraninite (see Table 3). In addition, a broad band at 5.173 Å appears in both X-ray photographs. This is representative of an amorphous material and corresponds to the type of pattern ordinarily yielded by a hydrocarbon. As further proof that the thucholite is the radioactive material affecting the Geiger counter, a radiogram was made of this material.

**ORIGIN OF MINERALS**

Two possible modes of emplacement have been suggested for the Temple Mt. ore. Gruner, Gardiner, and Smith (1953) have recently postulated a ground water leaching theory. The hypothesis which they refute is the alternate theory of deposition by hydrothermal solutions. Although it is not the purpose of this paper to discuss the merits of these two theories, the thucholite and native arsenic found in the examination of this nodule may shed some light on the problem.

A concentric band of native arsenic (see Fig. 1) is an unusual occurrence in economically valuable mineral deposits. Since there is an abundance of sulfides in the nodule and associated with the carbonaceous material in the sediments, it would seem likely that the arsenic might have been carried as a sulfide. If this were the case, it would have been deposited as a sulfide and later reduced to native arsenic. This reduction would have proceeded inward from the outer edge of the arsenic sulfide ring by a process of diffusion. This would probably leave some unreduced arsenic sulfide along the inner boundary of the ring. No such sulfide has been found. If the arsenic were carried and deposited as an oxide, a similar relict of arsenic trioxide might be expected, but none has been found. It is conceivable that all of the arsenic sulfide or trioxide has been reduced, but the width of the native arsenic band makes this seem improbable. Considerable heat is needed to reduce arsenic trioxide to the native state (Mellor, 1929). This would not be present in ground water solutions. However, the fact that the native arsenic is almost entirely enclosed within the nodule would result in a negligible post-depositional reduction of an arsenic sulfide or trioxide.
Proceeding on the assumption that the arsenic was carried in the native state, pneumatolytic or hydrothermal solutions are a more probable carrier than low temperature ground waters. The additional heat due to a geothermal gradient of 3° to 5°C per thousand feet of sediments under the approximate load of 10,000 to 12,000 feet would result in a maximum total temperature of under 80°C. The melting point of arsenic is 814°C at 36 atm. pressure (Mellor, 1929). At temperatures greater than 814°C and pressures greater than 36 atm., arsenic exists in the liquid state. A search of the literature reveals no data on the melting point of arsenic at pressures greater than 36 atm. A rock depth of 12,000 feet corresponds approximately to 900 atm. pressure (Barth, 1952). This would reduce the melting temperature, or temperature of crystallization, considerably, but it is doubtful that it could be reduced to 80°C. In addition, native arsenic sublimes from the solid state before it begins to melt. At one atmosphere of pressure it sublimes constantly at 554°C (Mellor, 1929). Therefore it is possible that metallic arsenic could be deposited from a gas or from a liquid.

The nodular masses of thucholite previously described also indicate deposition from a hydrothermal solution. Ellsworth, who originally described thucholite, states that it is as much a primary mineral as feldspar, quartz, and mica (Ellsworth, 1928). Since it is established that carbon may be magmatic in origin, and since his analyses of thucholite showed a relatively high percentage of rare earths, the conclusion that thucholite is a hypogene mineral seems logical. This is substantiated by the recent work of Davidson and Bowie who conclude that the thucholite of the Witwatersrand is of hydrothermal origin (Davidson and Bowie, 1951). In addition, Barthauer, Rulfs, and Pearce (1953) are of the opinion that a thucholite lower in thorium content than that described by Ellsworth is of hydrothermal origin. In their recent paper they state that "thucholite has never been found in intimate contact with any carbon containing material, either in the form of limestone or organic matter." It would seem that they also believe that the carbon in thucholite is of magmatic origin. If such is the case, polymerization would produce some of the hydrocarbons, such as methane, noted by both Barthauer and Ellsworth. Since a high temperature in addition to the radioactive emanations from uranium is required for polymerization to occur (personal communication, William Pesold), the thucholite could hardly have been deposited by ground water solutions but must have resulted from higher temperature hydrothermal solutions. This theory is given further impetus by the amount of rare earth elements in the thucholite examined by Barthauer (1953).

The evidence for a hydrothermal origin based upon this analysis, although far from conclusive, seems preferable to the ground water leaching hypothesis. However, there are many problems yet to be solved. The reason for the concentric banding in the nodule is not known. The exact type of solution which carried both uranium and native arsenic, in addition
to pyrite and sphalerite, and which appears to have replaced pre-existing quartz, is not known. Further field work and laboratory research are needed before conclusions can be reached in this respect.

REFERENCES


TEMPLE MOUNTAIN NODULE

Scale: x 1.8

KEY

- Quartz, pyrite
- Quartz, pyrite, and sphalerite
- Banded native arsenic
- Thucholite

Figure 1
X-RAY PHOTOGRAPH MEASUREMENTS
OF
NATIVE ARSENIC

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*Extra lines due to impurities.

These samples were run with Cu radiation and a Ni filter for 4 hours. The intensities were estimated relative to the lines on each photograph. The "d" Å values are within ± 2%. This is the limit of accuracy of the measurements and mathematical calculations.

TABLE 1
# X-ray Spectrometer Measurements of Native Arsenic

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## Temple Mountain, Utah

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*Extra lines due to impurities.

Cu radiation; Ni filter.

**TABLE 2**
COMPARISON OF URANINITE AND THUCHOLITE

<table>
<thead>
<tr>
<th>Uraninite (Hanawalt Index)</th>
<th>Thucholite Temple Mt. Specimen X-ray Spectrometer</th>
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<td>( I )</td>
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Note: Most of the above unnamed lines were found in the Canadian thucholite patterns and may be representative of thucholite.

TABLE 3
MINERALOGY OF THE CARLILE DEPOSIT, CROOK COUNTY, WYOMING

by

Marc W. Bodine, Jr.

ABSTRACT

Preliminary mineralogical studies have been made of the uranium and vanadium mineralization of the Lakota sandstone in the Carlile deposit on the northwestern flank of the Black Hills uplift in Crook County, Wyoming. These studies indicate the presence of coffinite (?) and doloresite (?) as earlier and possibly primary black minerals, which occur in carbonized wood seams and fragments. Later secondary minerals include: rauvite (?), occurring in the wood seams; carnotite, the predominant ore mineral which saturates the sandstones as grain coatings below the mineralized carbon seams; and metarossite, surrounding the carbon fragments above the ore body.

Thin section study of the mineralized sandstone shows well rounded quartz grains with a primary cement of calcite which for the most part has been removed and replaced by quartz as overgrowths on the original quartz grains. This process was accompanied by the silicification of the clay or feldspar fragments of the sandstone. The latest stage is a partial replacement of the secondary quartz by carnotite and limonite with embayments into the original quartz.

Two interesting pseudomorphs of hematite and goethite after pyrite and galena were collected a short distance from the deposit. Their presence suggests that sometime during the history of the sediments sulfides were introduced in the form of PbS and FeS$_2$. Further, the hematite may not be entirely of sedimentary origin.

ACKNOWLEDGMENTS

The writer wishes to express his appreciation to Dr. Paul F. Kerr, Professor of Mineralogy, Columbia University, who proposed the project and spent several days in the field reviewing the problem and offering suggestions. Dr. Kerr has also followed the laboratory work and the preparation of the manuscript.
The writer wishes to acknowledge the cooperation of Mr. Charles Towle, Chief of the Denver Office, Division of Raw Materials of the United States Atomic Energy Commission, who made the project possible. The cooperation of the personnel in the Hot Springs Sub-office of the Commission has also been most helpful.

Mr. Walter Woodmansee, Carlile project geologist, United States Atomic Energy Commission, kindly offered suggestions and participated in the geologic mapping. The writer appreciates the many suggestions of Mr. Joseph Gray, District Geologist of the Northern Black Hills, United States Atomic Energy Commission. His knowledge of the district proved most helpful in outlining the work.

INTRODUCTION

During the summer of 1953 the author was employed by the Walker-Lybarger Construction Company under contract with the United States Atomic Energy Commission to assist in the preliminary geological investigations of the Homestake uranium holdings south of Carlile, Crook County, Wyoming. A major portion of the summer was devoted to the preparation of a drilling program. Some time was available, however, for collecting material suitable for a mineralogical study of the deposit. The latter has been undertaken since October, 1953, in the mineralogical laboratories of the Department of Geology, Columbia University. Three lines of investigation have been attempted:

1. Identification of the black (primary) and yellow (secondary) uranium and vanadium minerals.

2. Study of the relationships between the ore minerals and the host rock.

3. A description of the hematite and goethite pseudomorphs after pyrite and galena and their relationship to the history of the deposit.

Several of the mineral identifications are tentative, and mineralogic and petrographic descriptions of the clays over and under the ore bearing sandstone have not been completed; therefore, this report must be considered as preliminary in nature.

The Carlile deposit (Olson and Tennissen, 1952), owned and operated by the Homestake Mining Company, Lead, South Dakota, is located in Sec. 26, T 52 N, R 66 W, Crook County, Wyoming about two miles south of the small town of Carlile. The deposit is situated on the edge of a broad plateau, Thorn Ridge, which has been dissected to a depth of several
hundred feet by the Belle Fourche River and its tributaries. The dissection has resulted in a series of promontories separated by steep-walled canyons. The principal ore body is located at the southern tip of one of these promontories, about 250 feet above the river level (Fig. 2). Open cut operations have been employed in mining the main ore body; while underground mining has been used for a smaller, more deeply buried ore body to the north. A third ore body lies to the east in the Belle Fourche River valley some 200 feet below. It appears to be an eastern extension of the first-mentioned body which has slumped toward the river. The slump blocks are of sufficient size to warrant development, and will probably be mined by stripping methods.

GENERAL GEOLOGY

The ore occurs in the basal part of the upper member of the Lakota sandstone which overlies a dense, impervious mudstone with an undulating or rolling surface. Carnotite, the predominant ore mineral, is disseminated in the host rock as grain coatings and interstitial fillings.

The Lakota sandstone is the basal unit of the Inyan Kara group, which includes the Lower Cretaceous rocks of the Black Hills region. Lying above the Lakota sandstone are the Fuson shale and the Fall River sandstone (formerly referred to as the Dakota sandstone). The Inyan Kara group is underlain by the Morrison formation, Jurassic (?), and is overlain by marine shales of the Upper Cretaceous (Graneros shale) which contain beds of bentonite (Darton, 1907).

Locally the Lakota sandstone can be divided into three members (Fig. 3) -- a lower sandstone and conglomerate member, containing lenses of highly silicified sandstone particularly evident under the deposit; a middle mudstone member; and an upper sandstone member. It is within the basal eight feet of the upper member that all the carnotite mineralization occurs.

The walls of the canyons are heavily wooded, which makes lithic description and correlation difficult; however, it was noted that the middle shale member of the Lakota sandstone is extremely lenticular. In the vicinity of the Carlile deposit the middle member is 34 feet thick, while a mile to the south the mudstone has pinched out and the Lakota sandstone is comprised entirely of arenites. To the north of the deposit the middle shale thickens and then thins again farther north. The drilling program now in progress should give considerable information on the behavior of this unit.

The deposit lies on the northwest slope of the Black Hills uplift with a regional dip of about 3° to the northwest. Faulting is almost nonexistent. Only one small fault of about one foot displacement was noted in the under-
Location Map
Crook Co., Wyoming

P Carlile deposit.
M Myers deposit.

0 1 2 miles.

Geologic Sketch Map, Carlile uranium deposit,
Crook County, Wyoming

Geology by: M.W. Bodine, Jr.
W. Woodmansee

Figure 2
STRATA
From Belle Fon Homestake no.3, Carllle, Crook County, Wyoming

Fall River Sandstone
Conglomerate.
Cross-bedded sandstone.
Silicified sandstone.
Thick to thin bedded sandstone.
Fine grained, massive sandstone.
Fine grained sandstone with calcareous cement.
Red and green mudstone.
Dense, grey and green mudstone.
Purple, grey, and green silty mudstone.
Uranium mineralization

Fuson Shale

Inyan Kara group
Thick to thin bedded sandstone.
Fine grained sandstone with calcareous cement.

Lakota Sandstone

Lower Cretaceous
Morrison formation (?) Jurassic

Figure 3
ground workings near the tunnel portal. This may be a superficial displacement associated with the slumping. Joints are numerous, but because of the extensive cover could not be traced for any appreciable distance. It was noted that where jointing is prevalent the ore is generally of higher grade than where joints are absent or scarce.

URANIUM AND VANADIUM MINERALS

Weeks and Thompson (1953) have subdivided the uranium and vanadium minerals of the Colorado Plateau into two groups: the unoxidized or primary (black) minerals and the unquestionably supergene or oxidized (yellow) minerals. Both of these groups are represented in the mineral suite from the Carlile deposit. Although some of the identifications are tentative, particularly in the case of the black minerals, the probability of their existence should be noted.

Primary Minerals

Two primary minerals have been identified from the carbonized wood seams, prevalent in the sandstones of the host rock at the Carlile deposit. Because of the extremely fine grained nature of the material, mineral separation techniques were unsatisfactory. Thus the existence of the black uranium and vanadium minerals has been assumed on the following evidence:

1. A high radiometric count is given by the black material.
2. The presence of uranium and vanadium in the black material has been established by X-ray fluorescence.
3. Yellow is absent in the specimens observed.
4. X-ray diffraction lines corresponding to known yellow minerals are absent. (Samples of carbonized wood with a slight yellow concentration and low radiometric count give excellent X-ray diffraction patterns of carnotite.)
5. X-ray diffraction lines are present which could not have been caused by quartz, limonite, or clays, the principal impurities of the carbonized wood.

Coffinite has been reported by L. R. Stieff and T. W. Stern from the Colorado Plateau. It has been described by Weeks and Thompson (1953) as a black mineral with an adamantine luster, crystallizing in the tetragonal system, and having a specific gravity of about 3.3 to 3.5. It is usually
associated with coalified wood and is often found in association with pyrite.

Weeks and Thompson (1953) are uncertain about the chemical composition and note that:

"Analyses show up to 61 percent U and varying amounts of Si, As, and V. Coffinite may be analogous to thorite (ThSiO₄), i.e., USiO₄ with As, V, or OH, etc., substituting for Si, or it may be a hydrated oxide."

The mineral has been identified by comparison of X-ray measurements with four strong diffraction lines as given by Weeks and Thompson (1953) (Table 4). This identification is further supported by the detection of arsenic as shown by X-ray fluorescence.

The mineral is disseminated, submicroscopically, throughout the organic material in the ore body at the Carlile deposit. However, coffinite (?) is much more abundant in the lower few feet of the ore body than in the upper half, and is completely absent in the organic debris of the uppermost twelve feet of the Lakota sandstone above the deposit.

Doloresite, a second unoxidized mineral from the organic material, is described by Weeks and Thompson (1953) as a black to bronze, massive, fibrous or radial aggregate with an adamantine luster, commonly found with pitchblende and coffinite. The chemical composition is unknown except that it is probably a V⁺² or V⁺⁴ compound.

Doloresite (?) was identified in the same manner as the coffinite (?) (Table 4). Here again the identification must be considered as tentative.

Doloresite (?) is associated with coffinite (?) as the primary minerals, and both occur in organic debris. It is most abundant in the lower organic material and completely absent in the organic fragments above the ore body.

Secondary Minerals

With the exception of rauvite (?) the secondary minerals yield more definite data. Both metarossite and carnotite were examined with the microscope. Rauvite (?), as in the case of the primary minerals, was identified on the basis of X-ray diffraction data.
TABLE 4

X-ray Diffraction Data of Coffinite, Doloresite and Rauvite

<table>
<thead>
<tr>
<th>Carbonized wood from Carlile deposit&lt;sup&gt;1&lt;/sup&gt;</th>
<th>Coffinite&lt;sup&gt;2&lt;/sup&gt;</th>
<th>Doloresite&lt;sup&gt;2&lt;/sup&gt;</th>
<th>Rauvite&lt;sup&gt;2&lt;/sup&gt;</th>
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</thead>
<tbody>
<tr>
<td>Cu rad</td>
<td>Cu rad</td>
<td>Cu rad</td>
<td>Cu rad</td>
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<tr>
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<td>I</td>
<td>dR</td>
<td>I</td>
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</table>

*Due wholly or in part to quartz.

1 Cu radiation with Ni filter.
2 Weeks and Thompson (1953)
Carnotite $\left[K_2(UO_2)_2(VO_4)_2\cdot 3H_2O\right]$ is the most abundant of the uranium-vanadium minerals and occurs as disseminations in the sandstone and carbonized wood fragments. The material is extremely fine grained and usually is present as coatings and in pore spaces between sand grains. The indices were determined as $n_a=1.76$ and $n_\gamma=1.96$ and some pleochroism of the larger grains was noted. The X-ray diffraction data (Table 5) agree with figures given by Weeks and Thompson (1953), and also measurements obtained for a specimen measured by Messrs. P. B. Barton and P. M. Bethke in this laboratory.

Metarossite $(CaV_2O_6 \cdot 2H_2O)$ is fairly well developed in halos surrounding the carbon fragments, particularly in the sandstones above the ore horizon. The material is dull yellow and extremely fine grained. It is light yellow under the microscope, and has indices of refraction of about 1.85. X-ray data (Table 6) compared with the data of Weeks and Thompson (1953) and with a sample from the Buckhorn mine, San Miguel County, Colorado, provide satisfactory confirmation of the identification.

Rauvite $(Ca_2U_2V_2O_{36} \cdot 20H_2O)$ is a purplish to bluish black mineral which has been tentatively identified from the carbonized wood seams. It presents the same difficulties in identification as coffinite (?) and doloresite (?). However, the diffraction lines agree with lines given by Weeks and Thompson (1953) (Table 4).

Other secondary minerals may exist. Pintadoite and tyuyamunite are probably present; but insufficient relatively pure material was available for satisfactory identification.

ASSOCIATED MINERALS

The common minerals in association with the uranium and vanadium minerals include pyrite, aluminite, limonite, alum, manganese oxides, gypsum, and calcite.

Pyrite, although not observed at the main deposit, undoubtedly was present before oxidation as indicated by the halos of limonite surrounding the carbon seams and fragments. In addition, remnants of pyrite have been found in a hematite pseudomorph after pyrite which was collected about 300 yards south of the deposit.

Aluminite, a hydrous aluminum sulfate, is found in fracture fillings and appears as a white, massive, soft material. It has been identified by optical and X-ray methods (Table 7) and by comparison with data on record by Palache, Berman, and Frondel (1951), and Hollingsworth and Bannister (1950).
TABLE 5

X-ray Diffraction Data of Carnotite

<table>
<thead>
<tr>
<th>Carnotite(^1) Carlile Deposit, Wyoming</th>
<th>Carnotite(^2) Naturita, Colo.</th>
<th>Carnotite(^3)</th>
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*Wholly or partially due to quartz as an impurity.

1 Cu radiation with Ni filter.

2 Measured by P. B. Barton and P. M. Bethke, Columbia University

3 Weeks and Thompson (1953).
TABLE 6

X-ray Diffraction Data of Metarossite

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* Due to quartz as an impurity.

1 Cu radiation with Ni filter.
2 Weeks and Thompson (1953)
# TABLE 7

**X-ray Diffraction Data of Aluminite**

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</table>

1 Cu radiation with Ni filter.
2 Hollingsworth and Bannister (1950)
Palache, Berman, and Frondel (1951) conclude that aluninite is formed as a result of leaching clays by sulfuric acid derived from the oxidation of pyrite.

Limonite is observed throughout the deposit as halos surrounding the carbonized wood seams and fragments, and in irregular areas of saturation of the sandstone. Where limonite is absent the host rock is bleached white. Hematite is virtually absent from the ore body.

Alums are present as fracture fillings in the vicinity of the mine and probably represent deposition from sulfate solutions.

Manganese oxides are present as fracture coatings with minor concentrations in the Fuson shale.

Gypsum occurs as secondary crystals along fractures in and near the ore body and, in places, acts as a cementing material for the Lakota sandstone.

Calcite has been noted as a cementing medium in the Lakota sandstone of the ore body. Also in the vicinity of the ore body lenticular bodies of sandstone have been completely cemented by calcite.

RELATIONS OF ORE TO THE HOST ROCK

The primary minerals, coffinite (?) and doloresite (?), are found only within the carbonized wood. They appear to have been associated with pyrite which is now entirely oxidized to limonite. Microscopic examination of the organic debris to date has proved fruitless due to the fine grained nature of both the carbon and the uranium-vanadium minerals. A further attempt to resolve this problem might be made with the electron microscope.

The secondary or supergene minerals, including carnottite and metarossite as ore minerals, and limonite, aluninite and other sulfates as the gangue minerals are easily observed, both in the field and in thin section.

A thin section study of the sandstone host rock points to the following series of events:

1. The sandstone has been cemented by calcite.

2. The calcite cement was later almost completely removed. This process was followed by the introduction of silica as secondary overgrowths on the original sand grains, and complete silicification of the original clay or feldspar grains.
3. Coating and partial replacement of the original quartz, the secondary overgrowths, and the silicified material occurred first by carnotite and secondly by limonite (Fig. 4A). The gypsum, alunimonite and other sulfates are the youngest minerals, commonly filling fractures.

It would appear on the basis of the field data that the carnotite is derived from the uranium and vanadium minerals in the organic debris and through oxidation processes is deposited in the sandstone. This has been the general sequence as noted on the Colorado Plateau (Waters and Granger, 1953).

The east face of the Carlile deposit shows this relationship very well. The secondary minerals may be present as halos surrounding the organic debris, with the halos relatively thick beneath the seams and thin above. Concentrations of carnotite also occur along the planes of cross-bedding extending downward from carbon seams (Fig. 5).

A concentration of carnotite occurs along the mudstone floor of the ore body. This floor shows definite rolls with the highest concentrations of carnotite in the troughs. Here the percolating waters stagnated and deposited the uranium and vanadium ions carried downward from the primary sources.

Further evidence for the oxidation of the carbon fragments is observed in the uppermost Lakota sandstone above the deposit where barren carbon fragments are surrounded by a rim of metarossite (Fig. 4B). This rimming is probably due to the oxidation of the primary material in the carbon and precipitation of rossite with later dehydration to metarossite.

Discussion of the origin of the primary minerals has not been attempted because of the tentative nature of their identification. Several questions, however, may be raised:

1. Was the emplacement of the primary minerals contemporaneous with the deposition of the quartz overgrowths? Or, on the other hand, was the development of the overgrowths simply a ground water phenomenon of over saturation and deposition of silica? Also was the development of overgrowths contemporaneous with the development of the highly silicified or quartzitized lenticular sandstone bodies under the deposit?

2. What is the relationship between the primary minerals and the carbonized wood? A great deal of field and chemical data on the nature and origin of coffinite would be necessary to solve this problem.
A. Thin-section of the upper Lakota sandstone from the ore horizon.

Q- Rounded original quartz grains.
S- Secondary quartz overgrowths.
C- Calcite.
K- Carnotite.
L- Limonite.
N- Silicified (?) clay fragment.
T- Carnotitized silicified material.

B. Carbonized wood fragment from the upper Lakota sandstone above the ore horizon.

M- Metarossite saturated sandstone.
C- Carbonized wood fragment.
Q- Quartz grains.

Figure 4
Sketch of a Portion of the East Ore Face,

Homestake No. 3, Carlile, Wyoming

- Massive sandstone.
- Cross-bedded sandstone.
- Conglomerate.
- Carbonized wood
- Intense carnolite mineralization.
- Dense mudstone.

Figure 5
3. What is the relationship between the uranium and vanadium mineralization and the mineralogy of the clays over and under the deposit? Do the clays contain volcanic debris as was noted by Waters and Granger (1953) on the Colorado Plateau? Has the introduction of uranium and vanadium had any altering effect on the clays? This aspect of the problem is now being studied; however, insufficient data have been accumulated to warrant a summary of the findings to date.

**PSEUDOMORPHS AFTER PYRITE AND GALENA**

Two interesting iron oxide pseudomorphs were collected near the Carlile deposit, one after galena and the other after pyrite.

**Goethite after galena**

A pseudomorph of goethite after galena was found about two miles east of the main deposit in an area of high count and some visible carnotite mineralization, known locally as the Myers deposit. The stratigraphic relations are obscure; however, the specimen is probably from the lower Lakota sandstone, forty or more feet above the Morrison formation contact.

The crystal outlines are extremely sharp showing one large cube with several well developed cubic cleavage planes (Pl. 1A). This crystal measures 1 cm. square and 0.5 cm. in depth with the base imbedded in a sandstone matrix. Numerous other crystals, also after galena, are present ranging in size from 0.1 to 0.7 cm. square. All have well developed cubic cleavage traces with one exception where the striations typical of pyrite were noted. The sandstone matrix is well cemented by goethite with the crystals forming on either side of the matrix.

X-ray diffraction data indicate the replacing mineral to be goethite with a minor amount of hematite (Table 8).

**Hematite after pyrite**

This specimen was found in the upper Lakota sandstone on the northern nose of a promontory some 300 yards south of the main deposit. The area yields a high count accompanied by some visible carnotite mineralization.

The crystals are in a sandstone matrix cemented by hematite and goethite with the largest crystal measuring about 0.5 cm. square (Pl. 1B). It is cubic in form with typical pyrite striations. The cube is modified by a pyritohedron resulting in a crystal similar to Figure 147 of Goldschmidt (1920). This crystal is intergrown with another but smaller modified cube
A. Goethite pseudomorph after galena, showing remnants of cubic cleavage.

0 1 cm.

B. Hematite pseudomorph after pyrite, with typical pyritic striations and crystal intergrowths.

0 1 mm.

PLATE 1
**TABLE 8**

X-ray Diffraction Data of Pseudomorph After Galena

<table>
<thead>
<tr>
<th>Goethite after galena, Carlile deposit, Wyo.</th>
<th>Goethite²</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \bar{d} R )</td>
<td>I</td>
</tr>
<tr>
<td>4.188</td>
<td>10</td>
</tr>
<tr>
<td>2.696</td>
<td>8</td>
</tr>
<tr>
<td>2.447</td>
<td>7</td>
</tr>
<tr>
<td>2.219</td>
<td>2</td>
</tr>
<tr>
<td>1.92</td>
<td>5</td>
</tr>
<tr>
<td>1.802</td>
<td>2</td>
</tr>
<tr>
<td>1.721</td>
<td>4</td>
</tr>
<tr>
<td>1.58</td>
<td>5</td>
</tr>
<tr>
<td>1.57</td>
<td>10</td>
</tr>
<tr>
<td>1.519</td>
<td>3</td>
</tr>
<tr>
<td>1.463</td>
<td>3</td>
</tr>
<tr>
<td>1.31</td>
<td>5</td>
</tr>
<tr>
<td>1.20</td>
<td>5</td>
</tr>
<tr>
<td>1.14</td>
<td>5</td>
</tr>
<tr>
<td>1.12</td>
<td>5</td>
</tr>
<tr>
<td>1.05</td>
<td>5</td>
</tr>
<tr>
<td>1.02</td>
<td>5</td>
</tr>
<tr>
<td>1.01</td>
<td>5</td>
</tr>
</tbody>
</table>

1 Cu radiation with Ni filter.

in a fashion similar to Figure 655 of Goldschmidt (1920). Many smaller crystals were observed similar to the larger ones and ranging in size from 0.05 to 0.2 cm. square.

Kerr and Hamilton (1953) have noted similar but smaller hematite pseudomorphs after pyrite as vein fillings in the Todilto limestone at Grants, New Mexico in association with pitchblende and fluorite. Although no association with the primary minerals was noted at Carlile, showings of carnotite are present within fifteen feet of the pseudomorph.

X-ray examination of the material gave a diffraction pattern indicating the major mineral to be hematite with minor amounts of residual pyrite and some goethite (Table 9).

Although hematite after pyrite is rare, goethite being the usual replacing mineral, Rankama and Sahama (1950) state that hematite may form if the ferric hydroxide is precipitated rapidly in a colloidal state. It can then be dehydrated to form hematite.

Conclusions

1. The introduction of sulfides as PbS and FeS$_2$ occurred at some time during the history of the sediments in the vicinity of the uranium mineralization.

2. At least some of the hematite present in the sandstones in the vicinity of the deposit is not indigenous to the sediments but has been formed by the oxidation of the earlier sulfide minerals.
### TABLE 9

**X-ray Diffraction Data of Pseudomorph After Pyrite**

<table>
<thead>
<tr>
<th>Pseudomorph after pyrite, Carlile, Wyo.</th>
<th>Hematite&lt;sup&gt;2&lt;/sup&gt;</th>
<th>Goethite&lt;sup&gt;3&lt;/sup&gt;</th>
<th>Pyrite&lt;sup&gt;4&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>dλ</strong></td>
<td><strong>I</strong></td>
<td><strong>dλ</strong></td>
<td><strong>I</strong></td>
</tr>
<tr>
<td>4.176</td>
<td>1</td>
<td>4.06</td>
<td>6</td>
</tr>
<tr>
<td>3.004</td>
<td>2</td>
<td>3.06</td>
<td>2.69</td>
</tr>
<tr>
<td>2.704</td>
<td>2</td>
<td>2.69</td>
<td>100</td>
</tr>
<tr>
<td>2.514</td>
<td>2</td>
<td>2.51</td>
<td>80</td>
</tr>
<tr>
<td>2.392</td>
<td>1</td>
<td>2.39</td>
<td>60</td>
</tr>
<tr>
<td>2.204</td>
<td>1</td>
<td>2.20</td>
<td>60</td>
</tr>
<tr>
<td>1.910</td>
<td>2</td>
<td>1.92</td>
<td>5</td>
</tr>
<tr>
<td>1.840</td>
<td>2</td>
<td>1.84</td>
<td>80</td>
</tr>
<tr>
<td>1.697</td>
<td>4</td>
<td>1.69</td>
<td>90</td>
</tr>
<tr>
<td>1.631</td>
<td>1</td>
<td>1.62</td>
<td>55</td>
</tr>
<tr>
<td>1.60</td>
<td>50</td>
<td>1.60</td>
<td>50</td>
</tr>
<tr>
<td>1.57</td>
<td>10</td>
<td>1.57</td>
<td>10</td>
</tr>
<tr>
<td>1.486</td>
<td>3</td>
<td>1.48</td>
<td>70</td>
</tr>
<tr>
<td>1.451</td>
<td>2</td>
<td>1.45</td>
<td>70</td>
</tr>
<tr>
<td>1.44</td>
<td>22</td>
<td>1.44</td>
<td>22</td>
</tr>
</tbody>
</table>

1. Fe radiation with no filter.
3. Ibid., card no. 3-0251.
4. Ibid., card no. 3-0822.
REFERENCES


ABSTRACT

Uranophane, CaO·2UO₃·2SiO₂·6H₂O, is the principal secondary uranium mineral found in the upper adit level of the Freedom No. 2 mine. It is associated with its dimorph, β-uranotile, which is present in smaller amounts. The optical properties for these two minerals have been determined as well as their interplanar spacings as determined by X-ray diffraction. A differential thermal curve for uranophane has been obtained. The mineral appears to lose adsorbed and some lattice water up to 300°C after which it undergoes complete collapse, and remains amorphous up to 600°C. Above this temperature it recrystallizes. Supergene action has resulted in the concentration of a large amount of silica in the Marysvale area. It is not surprising, therefore, to find these uranium-silicates in the oxidized zone.

THE NATURE OF URANOPHANE

Uranophane was first named and described by Websky in 1853. It was found in the selenium bearing uranium ores of Kupferberg, Silesia associated with uraninite and torbernite in a granitic intrusion. Websky described the uranophane as a pale green amorphous mass with sub-conchoidal fracture composed of SiO₂, H₂O, Al₂O₃ and UO₃. In a later publication Websky (1859) found that the "amorphous" material actually was made up of needle-like crystals which showed anomalous birefringence. He believed that this material was isomorphous with thomsonite. In 1870 Bořícký described a hydrated calcium uranium silicate from Wölsendorf, Bavaria which he called uranotile. H. von Foullon (1883) in studying the uranophane from Kupferberg found that the Al₂O₃ determined by Websky was in fact caused by the presence of admixed kaolinite, and established that the uranophane of Websky and the uranotile of Bořícký were chemically identical. In subsequent literature the terms have been used interchangeably, but uranophane, the first name to be employed, has priority and should be used rather than uranotile.

In 1921 Buttgenbach identified uranophane from Katanga, Belgian Congo, and in 1924 Schoep showed that uranophane has an isomorphous
relationship with sklodowskite as Mg substitutes for Ca. Larsen, Hess and Schaller (1926) showed that the "lambertite" described as a new mineral from Lusk, Wyoming, by Lind and Davis (1919) was actually similar to uranophane. Larsen, Hess and Schaller (1926) also determined the indices of refraction for uranophane: $\alpha = 1.642$, $\beta = 1.667$, $\gamma = 1.669$. Various chemical formulas for uranophane were proposed until Novacek (1935) established the formula as $\text{CaO} \cdot 2\text{UO}_3 \cdot 2\text{SiO}_2 \cdot 6\text{H}_2\text{O}$.

On the basis of rotation and powder photographs Billiet (1936) determined the cell dimensions for uranophane as well as its magnesium analogue, sklodowskite, and showed that the two minerals are orthorhombic and have similar structures:

<table>
<thead>
<tr>
<th></th>
<th>$a_0$</th>
<th>$b_0$</th>
<th>$c_0$</th>
<th>$a:b:c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uranophane</td>
<td>6.68Å</td>
<td>15.28Å</td>
<td>7.31Å</td>
<td>0.4371: 1 : 0.4784</td>
</tr>
<tr>
<td>Sklodowskite</td>
<td>6.67Å</td>
<td>15.50Å</td>
<td>7.12Å</td>
<td>0.4303: 1 : 0.4593</td>
</tr>
</tbody>
</table>

MARYSVALE URANOPOHANE

Uranophane is the principal secondary uranium-bearing vein mineral found in the upper adit level of the Freedom No. 2 mine at Marysvale, Utah. It occurs as a pale lemon yellow dissemination or as a coating on fractures in the altered quartz monzonite and rhyolite. It exhibits a botryoidal habit, as well as a radiating form which is lighter yellow and intergrown with the more massive darker material. The darker material shows weak green fluorescence, whereas the pale material appears to be non-fluorescent.

Chemical Composition

A few grams of this material were hand picked and analyzed in the Philips X-ray spectrograph using a lithium fluoride crystal. Table 10 shows the recorded reflections exclusive of those due to impurities from the target throughout the range $14^\circ$-53$^\circ$ 2 $\theta$.

The lead and zinc appear to be minor impurities. Qualitative chemical tests indicate the presence of CaO, SiO$_2$, a large amount of water, together with a small amount of phosphate and sulphur. There is no indication of vanadium. The presence of lead and phosphorous suggests the possibility that the minerals phosphuranylite or dewindtite may occur as impurities.
TABLE 10

<table>
<thead>
<tr>
<th>Element</th>
<th>2θ (°)</th>
<th>Reflections</th>
<th>Relative Amplitude</th>
</tr>
</thead>
<tbody>
<tr>
<td>U</td>
<td>17.57</td>
<td>LY₁</td>
<td>20</td>
</tr>
<tr>
<td>U</td>
<td>20.60</td>
<td>Lβ</td>
<td>40</td>
</tr>
<tr>
<td>U</td>
<td>21.61</td>
<td>Lβ₂</td>
<td>45</td>
</tr>
<tr>
<td>U</td>
<td>26.15</td>
<td>Lα₁</td>
<td>100</td>
</tr>
<tr>
<td>U</td>
<td>30.72</td>
<td>L₁</td>
<td>30</td>
</tr>
<tr>
<td>Pb</td>
<td>34.10</td>
<td>Lα₁</td>
<td>25</td>
</tr>
<tr>
<td>Zn</td>
<td>41.85</td>
<td>Kα</td>
<td>20</td>
</tr>
</tbody>
</table>

Optical Data

Optically, the massive material appears as a fine-grained aggregate which is slightly pleochroic from greenish yellow to pale yellow, and exhibits anomalous bluish interference colors. Since the orientation was indeterminate, only two indices are given for this material: α = 1.642, γ = 1.660. The fibrous material shows parallel extinction, is length slow, is biaxial negative with 2V about 20°, shows r < v, and exhibits anomalous interference colors. The indices and pleochroism for this material have been determined:

<table>
<thead>
<tr>
<th>n</th>
<th>Pleochroism</th>
</tr>
</thead>
<tbody>
<tr>
<td>α  = 1.634</td>
<td>colorless</td>
</tr>
<tr>
<td>β  = 1.568</td>
<td>pale yellow</td>
</tr>
<tr>
<td>γ  = 1.667</td>
<td>greenish yellow</td>
</tr>
</tbody>
</table>

These indices are somewhat lower than those determined for uranophane by Larsen, Hess and Schaller (1926). This may indicate a higher state of hydration for this uranophane than previously determined. It would be analogous to the findings of Frondel (1951) in the case of sabugalite and zeunerite. On the other hand, the somewhat lower indices may be caused by some substitution of Mg for Ca in the lattice which would represent a member part way toward sklodowskite. The indices for this end member as determined by Schoep (1930) are distinctly lower than those for uranophane: α = 1.613, β = 1.635, γ = 1.657.
Differential Thermal Analysis and X-ray Diffraction Data

The differential thermal curve (Fig. 6) is well defined and has endothermic peaks at 150, 230, 330, and 570°C with an exothermic reaction at 730°C. The recording apparatus was kept operative while the sample cooled, and it was found that the reactions are not reversible.

In order to determine, if possible, the reactions involved, some of the uranophane was heated in an oven for two hours at each of the following temperatures: 300, 600, and 800°C, and afterwards an X-ray diffraction powder pattern was made of each product. The patterns were taken with copper radiation and a nickel filter. A small amount of quartz was present which gave persistent lines throughout the temperature range. These have been omitted from the interplanar spacings given below.

At 300°C the structure is partially collapsed due probably to the loss of adsorbed as well as some lattice water. The positions of the 4 strong lines (Table 11) of the unheated material remain unchanged, although their intensities are greatly decreased. The weaker 5.411 Å line apparently shifts to 6.03 Å. The material heated to 300°C has the following spacings:

<table>
<thead>
<tr>
<th>dÅ</th>
<th>I</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.8</td>
<td>2</td>
</tr>
<tr>
<td>6.5</td>
<td>2</td>
</tr>
<tr>
<td>6.03</td>
<td>1</td>
</tr>
<tr>
<td>4.826</td>
<td>2</td>
</tr>
<tr>
<td>4.273</td>
<td>2</td>
</tr>
<tr>
<td>3.919</td>
<td>1</td>
</tr>
<tr>
<td>2.908</td>
<td>2</td>
</tr>
</tbody>
</table>

The color remains yellow.

Between 300 and 600°C the material undergoes complete collapse, with the apparent development of an amorphous phase. The X-ray diffraction pattern shows only 4 weak lines all due to quartz. The material after heating to 600°C turns bright orange.

After heating to 800°C the material becomes dark orange and undergoes recrystallization to form a new compound which does not seem to be related to any of the recorded data at hand. It was thought that it might dissociate to form U₃O₈ and wollastonite, CaSiO₃, but this does not appear to be the case. The quartz lines which are omitted from the spacings given below seem to be quite intense. The spacings for the new
A differential thermal curve of uranophane from the upper adit level of the Freedom No. 2 mine at Marysvale, Utah is well defined. Up to 300°C it appears to lose some lattice water and most of the adsorbed water. Above 300°C it undergoes complete collapse, and remains amorphous to 600°C. Above this temperature it recrystallizes to form a new phase.
compound are listed with the hope that future investigation may properly identify them:

<table>
<thead>
<tr>
<th>( dR )</th>
<th>I</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.9</td>
<td>6</td>
</tr>
<tr>
<td>4.2</td>
<td>3</td>
</tr>
<tr>
<td>3.196</td>
<td>10</td>
</tr>
<tr>
<td>2.938</td>
<td>1</td>
</tr>
<tr>
<td>2.851</td>
<td>1</td>
</tr>
<tr>
<td>2.700</td>
<td>5</td>
</tr>
</tbody>
</table>

Table 11 shows the measurements determined for Marysvale uranophane with the Philips X-ray diffractometer using copper radiation and nickel filter.

**TABLE 11**

<table>
<thead>
<tr>
<th>( dR )</th>
<th>I</th>
<th>( dR )</th>
<th>I</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.87</td>
<td>100</td>
<td>2.987</td>
<td>31</td>
</tr>
<tr>
<td>6.58</td>
<td>26</td>
<td>2.902</td>
<td>23</td>
</tr>
<tr>
<td>5.411</td>
<td>23</td>
<td>2.695</td>
<td>17</td>
</tr>
<tr>
<td>4.808</td>
<td>31</td>
<td>2.621</td>
<td>16</td>
</tr>
<tr>
<td>4.267</td>
<td>16</td>
<td>2.548</td>
<td>11</td>
</tr>
<tr>
<td>3.948</td>
<td>53</td>
<td>2.199</td>
<td>14</td>
</tr>
<tr>
<td>3.588</td>
<td>19</td>
<td>2.096</td>
<td>17</td>
</tr>
<tr>
<td>3.504</td>
<td>23</td>
<td>2.056</td>
<td>12</td>
</tr>
<tr>
<td>3.342</td>
<td>23</td>
<td>1.974</td>
<td>13</td>
</tr>
<tr>
<td>3.201</td>
<td>25</td>
<td>1.909</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.884</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.826</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.817</td>
<td>11</td>
</tr>
</tbody>
</table>

Table 12 shows the strong lines of the Marysvale material compared with those in the literature. There appears to be a closer agreement between the spacings of the Marysvale uranophane and those reported by Weeks and Thompson (1953).

As mentioned earlier, Billiet (1936) determined the cell dimensions for uranophane. Using these values an attempt was made to index the Marysvale uranophane, but it appears that the dimensions do not apply to this material exactly, and that the \( b \)-spacing is probably greater than that determined by Billiet. This may result from some Mg substitution in the Marysvale uranophane, as this spacing is larger in the Mg member, sklodowskite.
MITCHELL COUNTY, NORTH CAROLINA URANOPHANE

A specimen of uranophane from the Columbia University mineral collection was examined optically and by X-ray diffraction to serve for comparison with the Marysvale uranophane. The material comes from Mitchell County, North Carolina and is labeled uranophane. Steinocher and Novacek (1939) refer to a specimen from this locality which had been misidentified as schroeckingerite, as well as uranophane. They showed that actually the material was $\beta$-uranotile. Investigation of the Columbia specimen shows that it is uranophane, and therefore it would seem that both uranophane and $\beta$-uranotile occur at this locality. The indices of refraction are: $\alpha = 1.641$, $\beta = 1.664$, $\gamma = 1.671$.

Table 13 gives the interplanar spacings for the Mitchell County uranophane. These were obtained with a debye-scherrer camera using copper radiation and nickel filter. The spacings are close to those for the Marysvale material as shown on Table 11, except for the spacings between $2.623 \AA$ and $2.196 \AA$ which may be due to an unidentified impurity.

THE NATURE OF $\beta$-URANOTILE

Nováček (1935) introduced the term $\alpha$-uranotile which he changed to $\beta$-uranotile for material found at Jáchymov, Czechoslovakia. It is chemically similar to uranophane, but has higher refractive indices. Steinocher and Novacek (1939) found that $\beta$-uranotile is monoclinic, and the indices vary: $\alpha = 1.659 - 1.674$, $\beta = 1.674 - 1.704$, $\gamma = 1.682 - 1.713$. They report that some crushed $\beta$-uranotile corresponds optically to uranophane. Schoep and Scholz's (1931) unknown from Wölsendorf, the original uranotile (uranophane) locality, is shown to be $\beta$-uranotile.
MARYSVALE $\beta$-URANOTILE

$\beta$-uranotile, the dimorph of uranophane, has been found associated with uranophane in the upper adit level of the Freedom No. 2 mine. It occurs as minute veinlets which are disseminated through the oxidized portion of the vein. It is orange-yellow, occurs in small lath-like bundles which have a generally radial orientation, and is non-fluorescent in ultraviolet light. The optical properties are as follows:

<table>
<thead>
<tr>
<th>Indices</th>
<th>Pleochroism</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha$ = 1.665</td>
<td>colorless</td>
</tr>
<tr>
<td>$\beta$ = 1.684</td>
<td>pale greenish yellow</td>
</tr>
<tr>
<td>$\gamma$ = 1.695</td>
<td>greenish yellow</td>
</tr>
</tbody>
</table>

The material is biaxial negative, 2V about 35°, $r < v$ very strong, extinction 30° †, and the birefringence is anomalous. These properties agree closely with those determined by Steinocher and Nováček (1939). The laths are clear with a few inclusions of minute, unidentified black specks which may be pitchblende.
X-ray Diffraction Data

Gruner and Gardiner (1950) give the following key interplanar spacings for $\beta$-uranotile with unfiltered Fe-radiation.

<table>
<thead>
<tr>
<th>(d)</th>
<th>(I)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.65</td>
<td>10</td>
</tr>
<tr>
<td>3.85</td>
<td>7</td>
</tr>
<tr>
<td>2.58</td>
<td>4</td>
</tr>
<tr>
<td>1.80</td>
<td>2</td>
</tr>
</tbody>
</table>

These compare favorably with the Marysvale $\beta$-uranotile which is shown in Table 14. The X-ray diffraction pattern was obtained with a debye-scherrer camera using copper radiation and a nickel filter.

<table>
<thead>
<tr>
<th>(d)</th>
<th>(I)</th>
<th>(d)</th>
<th>(I)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.7</td>
<td>10</td>
<td>2.583</td>
<td>7</td>
</tr>
<tr>
<td>6.67</td>
<td>6</td>
<td>2.368</td>
<td>2 broad</td>
</tr>
<tr>
<td>6.12</td>
<td>6</td>
<td>2.186</td>
<td>3 broad</td>
</tr>
<tr>
<td>5.049</td>
<td>5</td>
<td>2.144</td>
<td>1</td>
</tr>
<tr>
<td>4.870</td>
<td>1</td>
<td>2.122</td>
<td>1</td>
</tr>
<tr>
<td>4.583</td>
<td>6</td>
<td>2.074</td>
<td>2</td>
</tr>
<tr>
<td>4.092</td>
<td>6</td>
<td>2.005</td>
<td>2</td>
</tr>
<tr>
<td>3.855</td>
<td>9</td>
<td>1.956</td>
<td>1</td>
</tr>
<tr>
<td>3.646</td>
<td>8</td>
<td>1.923</td>
<td>3</td>
</tr>
<tr>
<td>3.510</td>
<td>2</td>
<td>1.867</td>
<td>3</td>
</tr>
<tr>
<td>3.342</td>
<td>2</td>
<td>1.837</td>
<td>1</td>
</tr>
<tr>
<td>3.173</td>
<td>7</td>
<td>1.795</td>
<td>1</td>
</tr>
<tr>
<td>3.030</td>
<td>5</td>
<td>1.727</td>
<td>2</td>
</tr>
<tr>
<td>2.957</td>
<td>3</td>
<td>1.693</td>
<td>2</td>
</tr>
<tr>
<td>2.887</td>
<td>1</td>
<td>1.614</td>
<td>2 broad</td>
</tr>
<tr>
<td>2.815</td>
<td>6</td>
<td>1.540</td>
<td>1</td>
</tr>
<tr>
<td>2.765</td>
<td>3</td>
<td>1.518</td>
<td>1</td>
</tr>
</tbody>
</table>

CONCLUSION

Silica is concentrated in the upper portions of veins, and where veins are exposed at the surface. This occurs at many places in the Marysvale area as a result of supergene action. It is not surprising, therefore, to find uranium-bearing silicates such as uranophane and $\beta$-uranotile in the oxidized zone.
REFERENCES


Frondel, Clifford (1951) Studies of uranium minerals, VII, sabugalite, an aluminum-autunite, Contr. from Dept. Mineralogy and Petrology, Harvard University, no. 330.


Gruner, J. W., and Gardiner, Lynn (1950) Tables of uranium and vanadium minerals which are largely of secondary origin, Univ. Minnesota, Minneapolis, Minn.


PHOSPHURANYLITE AT MARYSVALE, UTAH

by

P. K. Hamilton and Paul F. Kerr

ABSTRACT

Phosphuranylite, $\text{Ca}_3(\text{UO}_2)_5(\text{PO}_4)_4(\text{OH})_4\cdot 10\text{H}_2\text{O}$, is associated with alunite at the J. and L. Alunite mine near Marysvale, Utah. This occurrence is of interest as it is one of few places in the Marysvale district where supergene uranium mineralization is associated with earlier hydrothermal alteration which resulted in alunite. The mineral is identified on the basis of X-ray diffraction data.

OCCURRENCE

Phosphuranylite occurs at the end of the alunite tunnel in the J. and L. Alunite mine. A strong fault passes diagonally northward at the end of the tunnel. Accompanying the fault is a zone of fracturing which is exposed along the tunnel some twenty feet from the heading. Uranium mineralization has migrated along small fractures in this zone and resulted in thin disseminated films of phosphuranylite which fluoresce in ultraviolet light. The alunite occurs in blocks between the fractures, and represents earlier hydrothermal alteration, whereas the phosphuranylite appears to be supergene. However, the general occurrence of uranium in the district suggests derivation from a hypogene source. The occurrence is significant since it is one of the few places in the Marysvale district where uranium mineralization may be found in association with the earlier alunite deposits.

X-RAY DIFFRACTION DATA

The X-ray diffraction pattern of the yellow radioactive mineral from the J. and L. Alunite mine yields lines which correspond to phosphuranylite, $\text{Ca}_3(\text{UO}_2)_5(\text{PO}_4)_4(\text{OH})_4\cdot 10\text{H}_2\text{O}$. Frondel (1950) indicates that the lead-bearing isostructural dewindtite, $\text{Pb}_3(\text{UO}_2)_5(\text{PO}_4)_4(\text{OH})_4\cdot 10\text{H}_2\text{O}$, has a similar X-ray diffraction pattern, hence it is necessary to establish the presence of calcium or lead in distinguishing the two minerals. Qualitative tests for lead with acetic acid and potassium iodide are negative. Tests for calcium with ammonium oxalate and phosphate with ammonium molybdate are positive. It seems probable, therefore, that the mineral is phosphuranylite.
The X-ray diffraction measurements compare favorably with those in the literature. A weak extra line appears at 8.58 AU. According to Frondel, in some cases the 7.8 AU line may be a doublet. This may account for the weak line at 8.58 AU in the Marysvale material. A comparison of the strong lines as given by Gruner and Gardiner (1950), Weeks and Thompson (1953), and Frondel (1950) with the Marysvale sample is shown in Table 15. The slight variations noted may result from some undetected substitution of Pb for Ca in the Marysvale material. Table 16 gives the measured interplanar spacings compared with those determined by Frondel.

### TABLE 15

<table>
<thead>
<tr>
<th>Gruner &amp; Gardiner</th>
<th>Weeks &amp; Thompson</th>
<th>Frondel</th>
<th>Marysvale, Utah</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$d \bar{\AA}$</td>
<td>I</td>
<td>$d \bar{\AA}$</td>
<td>I</td>
</tr>
<tr>
<td>7.96</td>
<td>10</td>
<td>7.9</td>
<td>s</td>
</tr>
<tr>
<td>5.85</td>
<td>5</td>
<td>5.83</td>
<td>m</td>
</tr>
<tr>
<td>3.92</td>
<td>5</td>
<td>3.92</td>
<td>m</td>
</tr>
<tr>
<td>2.87</td>
<td>5</td>
<td>2.88</td>
<td>m</td>
</tr>
</tbody>
</table>

### REFERENCES


Gruner, J.W. and Gardiner, Lynn (1950) *Tables of uranium and vanadium minerals which are largely of secondary origin*, Univ. Minnesota, Minneapolis, Minn.

### TABLE 16

**X-ray Powder Spacing Data for Phosphuranylite**  
Cu rad.  Ni fil.  

<table>
<thead>
<tr>
<th>Frondel (1950)</th>
<th>Marysvale, Utah</th>
<th>Frondel (1950)</th>
<th>Marysvale, Utah</th>
</tr>
</thead>
<tbody>
<tr>
<td>$d \bar{R}$</td>
<td>$I$</td>
<td>$d \bar{R}$</td>
<td>$I$</td>
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<tr>
<td>10.163</td>
<td>1</td>
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<tr>
<td>8.58</td>
<td>db</td>
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<tr>
<td>7.830</td>
<td>10</td>
<td>7.88</td>
<td>10</td>
</tr>
<tr>
<td>7.196</td>
<td>1/2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6.326</td>
<td>1</td>
<td>6.35</td>
<td>1/2</td>
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<td>5.829</td>
<td>8</td>
<td>5.873</td>
<td>8</td>
</tr>
<tr>
<td>5.372</td>
<td>1/2</td>
<td>5.361</td>
<td>3</td>
</tr>
<tr>
<td>4.955</td>
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<td>4.973</td>
<td>1</td>
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<tr>
<td>4.720</td>
<td>2</td>
<td>4.711</td>
<td>2</td>
</tr>
<tr>
<td>4.332</td>
<td>3</td>
<td>4.443b</td>
<td>2</td>
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<td>4.250</td>
<td>1/2</td>
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<td>3.969</td>
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<tr>
<td>3.606</td>
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<tr>
<td>3.440</td>
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<td>3.448</td>
<td>1</td>
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<td>6</td>
<td>3.346</td>
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<td>6</td>
<td>3.143</td>
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<td>4</td>
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<td>2.596</td>
<td>1</td>
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<tr>
<td>2.455</td>
<td>1</td>
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<td>1</td>
</tr>
<tr>
<td>2.430</td>
<td>2</td>
<td>2.439b</td>
<td>1</td>
</tr>
<tr>
<td>2.352</td>
<td>1/2</td>
<td>1.209</td>
<td>1/2</td>
</tr>
<tr>
<td>2.243</td>
<td>1/2</td>
<td>2.272</td>
<td>1/2</td>
</tr>
<tr>
<td>2.212</td>
<td>1</td>
<td>2.213</td>
<td>1</td>
</tr>
<tr>
<td>2.156</td>
<td>1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.099</td>
<td>1/2</td>
<td>2.119b</td>
<td>1</td>
</tr>
<tr>
<td>2.080</td>
<td>2</td>
<td>2.087b</td>
<td>1</td>
</tr>
</tbody>
</table>

db - doublet  
b - broad
PITCHBLENDE AND MANGANOCALCITE AT THE DEER TRAIL MINE
MARYSVALE, UTAH

by
Paul F. Kerr and P. K. Hamilton

ABSTRACT

Pitchblende has been isolated and identified as the principal radioactive mineral in the Deer Trail mine. It occurs on the border of a replacement ore body of lead, zinc, and silver, and is intimately associated with gray-white chlorite and manganocalcite. On the basis of optical and X-ray diffraction data the manganocalcite is estimated to contain between 7 and 16 percent MnCO₃. This occurrence of pitchblende, chlorite and manganocalcite is non-commercial, but it seems worthy of attention since uranium is found in limestone rather than volcanics, and lies on the margin of the Marysvale uranium district.

PITCHBLENDE

A non-commercial but mineralogically interesting uranium occurrence in the Deer Trail mine was discovered by Kerr and Simpson in 1952. Because of its finely divided nature, the mineral responsible for the radioactivity was not determined at that time. The uranium is found in a chloritized zone bordering a lead-zinc-silver ore body (Kerr, Simpson and Hamilton, 1953). Since the mineralization occurs near the Marysvale uranium area, and is present in carbonate rock rather than in altered volcanics or intrusives, it may have considerable significance from the standpoint of genesis.

At the time of the above mentioned report a fraction less than 200 mesh in size was found to contain 0.137% U₃O₈. Further investigation has shown that a fraction less than 300 mesh and greater than 200 mesh actually is the more radioactive. After various settling techniques with water as well as bromoform failed to yield a significantly radioactive concentrate, the fraction was run through the Franz isodynamic separator.

A few black grains and flake-like particles were noted in the concentrate. These were isolated by hand picking, and X-ray diffraction patterns made of both the flake-like material and the small black grains. Both proved to be pitchblende. This material has been identified as
pitchblende rather than uraninite, since the grains as exposed to X-rays before grinding show debye-scherrer diffraction rings rather than Laue-type diffraction spots. As noted by Cohen (1953) these diffraction rings suggest pitchblende crystallites on the order of 10⁻³ cm. or smaller, rather than the megascopically crystalline uraninite. The interplanar spacings for this material are shown on Table 17.

**TABLE 17**

<table>
<thead>
<tr>
<th>dR</th>
<th>I</th>
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</thead>
<tbody>
<tr>
<td>3.170</td>
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<tr>
<td>2.736</td>
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<td>1.933</td>
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</tr>
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<td>1.646</td>
<td>6</td>
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<tr>
<td>1.574</td>
<td>1</td>
</tr>
<tr>
<td>1.363</td>
<td>2</td>
</tr>
<tr>
<td>1.251</td>
<td>2</td>
</tr>
<tr>
<td>1.223</td>
<td>1</td>
</tr>
<tr>
<td>1.113</td>
<td>2</td>
</tr>
<tr>
<td>1.047</td>
<td>1</td>
</tr>
</tbody>
</table>

This fraction also contains traces of a bright yellow, non-fluorescent material which may be an unidentified uranium oxidation product. However, pitchblende is believed to be the principal source of the radioactivity.

**MANGANOCALCITE**

The carbonate associated with the pitchblende and chlorite in the Deer Trail mine was tentatively identified by Kerr, Simpson and Hamilton (1953) as a mixture of magnesium and calcium carbonates. However, the carbonate most intimately connected with the uranium mineralization has been found, on further study, to be manganocalcite. However, the country rock is limestone with some dolomitic phases. In a study of manganocalcite, Krieger (1930) has shown that there is a series relationship between indices of refraction, the X-ray diffraction pattern and the MnCO₃ content. Table 18 shows the relation of the indices of refraction of the Deer Trail manganocalcite to those of Krieger's reference samples. The presence of manganese has been further confirmed by qualitative chemical tests, and on the basis of these indices the Deer Trail material seems to contain between 7 and 16 percent MnCO₃.
Table 19 shows the effect of manganese substitution on the inter-planar spacings of the X-ray diffraction pattern. The table gives the spacings of the most prominent lines based on Krieger's work compared with those of the Deer Trail material. Again, it appears that this material contains somewhere between 7 and 16 percent MnCO₃.

TABLE 19

<table>
<thead>
<tr>
<th></th>
<th>dₐ</th>
<th>dₐ</th>
<th>dₐ</th>
<th>dₐ</th>
<th>dₐ</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcite</td>
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<td>2.522</td>
<td>2.300</td>
<td>2.110</td>
<td>1.935</td>
</tr>
<tr>
<td>7.00% MnCO₃</td>
<td>3.020</td>
<td>2.491</td>
<td>2.268</td>
<td>2.085</td>
<td>1.903</td>
</tr>
<tr>
<td>Deer Trail</td>
<td>3.000</td>
<td>2.473</td>
<td>2.262</td>
<td>2.078</td>
<td>1.889</td>
</tr>
<tr>
<td>15.4% MnCO₃</td>
<td>3.005</td>
<td>2.472</td>
<td>2.257</td>
<td>2.070</td>
<td>1.883</td>
</tr>
<tr>
<td>32.34% MnCO₃</td>
<td>2.975</td>
<td>2.455</td>
<td>2.250</td>
<td>2.060</td>
<td>1.866</td>
</tr>
<tr>
<td>Rhodochrosite</td>
<td>2.850</td>
<td>2.389</td>
<td>2.180</td>
<td>1.990</td>
<td>1.809</td>
</tr>
</tbody>
</table>

Manganese was probably a significant constituent of the solutions responsible for the uranium mineralization in the Deer Trail deposit. Further, it is secondary to the associated limestone. In this connection
it is interesting to note the prominent black manganese staining found on quartz crystals in limestone cavities encountered in the mine workings. Clusters of quartz crystals up to a foot in length have been found lining cavities which in turn are filled with black sooty manganese oxide.

REFERENCES


Euhedral quartz crystals have been found in the Todilto limestone at Grants, New Mexico. These crystals exhibit the characteristic forms of quartz which has formed below 573°C. Since temperature criteria are meager for the Todilto at this locality, the occurrence of secondary quartz is of interest.

LOW AND HIGH TEMPERATURE QUARTZ

Quartz crystals are a rarity in the Todilto limestone at Grants, New Mexico. Hence a number of small crystals collected by Mr. Forrest R. Fincher* and submitted for examination by Dr. Thomas W. Mitcham** are of more than ordinary interest. Since temperature criteria in the Todilto limestone are meager, the crystals were examined in order to determine whether they might represent the low or high temperature phase.

It is well known that low temperature quartz forms below 573°C, whereas the high temperature form exists between 573°C, the inversion point, and 870°C. Larsen and Wright (1909) have outlined criteria for differentiating between low and high temperature quartz on the basis of crystal habit, twinning, intergrowths, and shattering.

Low-quartz belongs to the trigonal trapezohedral class. This is characterized by one axis of threefold symmetry, and three axes of twofold symmetry which are perpendicular thereto and separated by angles of 120°. High-quartz belongs to the hexagonal trapezohedral symmetry class which is characterized by one sixfold axis of symmetry, and six twofold axes which are perpendicular thereto and separated by angles of 30°. High-quartz differs in habit from low-quartz. In this phase the pyramidal faces are equally developed, trigonal trapezohedrons are

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*Geologist, Field Office, Grants, New Mexico.
**Assistant Chief Geologist, Division of Raw Materials, Grand Junction Office.
absent, and the habit of the crystal is, in general, that of simple di-hexahedrons or first-order hexagonal bipyramids. The first-order hexagonal prism also occurs, but much less commonly than in low-quartz. Crystals of low-quartz, on the other hand, are usually prismatic, and often show marked differences in the size and character of the rhombohedral faces.

The form and character of twinning on basal sections of quartz can be used as a criterion in determining whether or not it has formed above or below 573°C. This twinning is best studied by means of etch figures (obtained by immersing plates of quartz in cold hydrofluoric acid) on the basal pinacoid. In high temperature quartz an intricate complex of twins is present, and the twinning lines are usually irregular. This is in contrast to low-quartz which tends to have regular, sharply marked twinning.

Intergrowths of right and left-handed crystals are less frequent, and more irregular in high-quartz than in low-quartz.

High-quartz is often considerably fractured, and frequently crumbles or breaks with acid (HF) treatment. Low-quartz, on the other hand, tends to remain intact after this treatment.

**QUARTZ CRYSTALS FROM THE TODILTO**

The quartz crystals in the Todilto limestone (Plate 2) were separated by leaching in cold dilute HCl. When freed from the matrix, the crystals range in length from about 1 to 5 mm. They occur as clusters as well as euhedral crystals which are either singly or doubly terminated. The crystals appear to vary from a dark muddy brown to a lighter yellow brown, to white.

The accompanying photograph (Plate 2) shows two of these quartz crystals at a magnification of approximately X20. They have the typical forms of well crystallized low temperature quartz. The crystals are prismatic, and show marked differences in the size and character of the rhombohedral faces. This indicates that the crystals formed below 573°C, and therefore are the low temperature form. The larger, doubly terminated crystal in the photograph also seems to contain some minute, unidentified inclusions.

It is of interest to note that clastic quartz has been reported as occurring in the Todilto limestone by Rapaport (1952) as well as by Kerr and Hamilton (1953). However, as Mr. Fincher has observed, this may be the first reported secondary quartz in this formation.
PLATE 2

QUARTZ CRYSTALS FROM THE TODILTO LIMESTONE GRANTS, NEW MEXICO X20

These crystals show the characteristic prism faces and unequally developed rhombodrons of quartz ordinarily considered to be the low temperature phase.
REFERENCES


URANIUM OCCURRENCES IN THE OLDER SEDIMENTARY ROCKS
OF THE MARYSVALE DISTRICT

by
Philip M. Bethke and Paul F. Kerr

ABSTRACT

Uranium occurs in the older sedimentary rocks exposed on the upthrown block of the Tushar fault along the eastern front of the Tushar Mountains to the south and west of Marysvale. Older sedimentary rocks exposed range from probable Carboniferous to middle Jurassic in age. Uranium occurs in association with base and precious metal mineralization in both quartz vein and replacement type bodies. Alunite bodies in the overlying Bullion Canyon Volcanics are believed to be expressions of hydrothermal alteration accompanying base metal and possible uranium mineralization at depth.

ACKNOWLEDGMENTS

In addition to the assistance of Mr. Stephen Steinhauser and his associates of the Richfield Field Office, A. E. C., as mentioned earlier, it is desired to express appreciation for many courtesies on the part of local mine operators in connection with the southwestern Marysvale study. Mr. John Wilhelm, president, and Mr. Jay Sylvester, mine superintendent of Deer Trail Mines; Mr. Scott Cutler and Mr. Robert Glenny, operators of the Shamrock mine; and Mr. Lee Trout, operator of the Great Western mine have all been most courteous in aiding the study and have provided many specimens for laboratory examination. Their cooperation has been most helpful.

INTRODUCTION

An investigation of the general geologic features and uranium occurrences in the older sedimentary rocks near Marysvale, Utah was undertaken during the field season of 1953. Exposed strata may be observed along the eastern front of the Tushar Mountains and in the canyons which cut deeply into the range. An outline map showing the general geologic features of the area of sedimentary outcrops is shown in Figure 7. The eastern front of the Tushar Mountains represents the eroded scarp of the Tushar fault which lies on the western side of the Marysvale valley.
GENERALIZED GEOLOGICAL SKETCH MAP AREA OF OLDER SEDIMENTARY ROCKS

Figure 7
The fault is normal and dips toward the east with a stratigraphic displacement of over 4,000 feet. The Marysvale valley lies in a graben with the Tushar fault on the west and the Sevier fault on the east. Bullion Canyon volcanics (Callaghan, 1939) of post Oligocene (?) age are displaced by the Tushar fault. The strata exposed on the upthrown block forming the Tushar range are inclined to the northwest at about 15° in the lower part of the section and to the southwest at 30-40° in the upper part. The structure of much of the range is complicated locally by minor faulting and occasionally by huge slumps.

At higher elevations on the Tushar range Bullion Canyon volcanics are exposed and overlie the sedimentary strata (Callaghan, 1939). The volcanics are altered in places to form small alunite bodies which may be of either the vein or the replacement type. The alunite is believed to represent deposition along hydrothermal alteration channels which may be synchronous with base and precious metal emplacement in the underlying sedimentary rocks. Small precious metal occurrences have been worked in the Bullion Canyon volcanics themselves.

OLDER SEDIMENTARY STRATA

The older sedimentary strata exposed by the Tushar fault appear to range in age from Carboniferous to upper Jurassic. The younger part of the section may be measured on Deer Trail Mountain just south of the Old Deer Trail mine (Fig. 8). The oldest rocks exposed are seen in the PTH Tunnel in the New Deer Trail mine. These lower strata are almost entirely carbonatites with an aggregate thickness of more than 2,000 feet. The lithologic type varies from pure calcilutite to argillaceous calcisiltite, with small thicknesses of red, gypsiferous shale near the bottom and top of the section. Much of the limestone is silicified, particularly near the areas of base metal accumulation. The uppermost rocks exposed in the tunnel consist of interbedded red shales, carbonatites and quartzites. No fossils were found in the section exposed, but consideration of the stratigraphic sequence in adjoining areas indicates that the interbedded quartzites, limestones and red shales at the top of the sequence may be Permian in age, probably equivalent to the Supai formation, and that the great thickness of underlying carbonatites is of Carboniferous and possibly older age. The Supai has been described by Gregory and Moore (1931) from the Kaiparowits region about 90 miles southeast of Marysvale and by Reeside and Bassler (1922) from Virgin Valley near Zion Park. The top of the Supai is not exposed, but the total thickness is thought to be slightly over 100 feet.
COCONINO FORMATION

The Supai beds are overlain by a brittle, tan, highly indurated quartzite, cross bedded on a grand scale and measuring 534 feet in thickness. These quartzite beds mark the upper limits of talus cones made up of quartzite blocks, some accumulations measuring a hundred or more feet in thickness as shown by drilling. The base of the quartzite is exposed in the lower workings at the Carrizo mine at the mouth of Cottonwood Canyon. On the basis of similar stratigraphic position and lithology this unit may probably be correlated with the Coconino formation described from the San Rafael Swell (Gilluly and Reeside, 1928), Virgin Valley (Reeside and Bassler, 1922) and the Kaiparowits Plateau (Gregory and Moore, 1931).

KAIBAB FORMATION

The Coconino quartzite is overlain by 441 feet of interbedded argillaceous limestone, dolomite, shale, quartzite and sandstone becoming more arenaceous towards the top, all exposed on the steep mountain slope. Above this sequence is a high cliff which exposes 335 feet of argillaceous dolomite and limestone containing occasional chert beds and fossiliferous beds which yield fragmentary remains of spiny brachiopods and pentagonal crinoid stems. McKee (1938) describes a similar section from the west front of the Mineral Mountains some 30 miles west of Marysvale and divides the section into the Toroweap and Kaibab formations. A lithologic break can be observed in the Marysvale section due to the absence of quartzite lenses in the upper, cliff forming unit, but no evidence was found to indicate that the lower unit is the Toroweap equivalent and the upper Kaibab. Sections from the San Rafael Swell (Gilluly and Reeside, 1928) and Circle Cliffs (McKee, 1938) indicate the absence of the Toroweap formation. The writers have tentatively classified the entire sequence at Marysvale as Kaibab, but this classification may be subject to revision. The lower unit, consisting of interbedded limestone and quartzite, is the host rock of the ore bodies in the Old Deer Trail mine and the Lucky Boy mine. In these mines the mineralization is virtually confined to the carbonate lenses where it occurs in replacement bodies.

MOENKOPI FORMATION

The Kaibab formation is overlain disconformably by 104 feet of sands, siltstones and variegated green, red, gray and tan micaceous shale, which makes up the lower red unit of the Moenkopi formation. The contact with the Kaibab formation is marked by shallow scours and 21 feet
of limestone conglomerate and coarse calcarenite with pebbles which are similar lithologically to the underlying Kaibab. The lower red unit forms a steep slope below a high gray cliff of thinly bedded, gray, buff weathering argillaceous calcisiltite with local thin beds of limestone conglomerate. The limestone contains thin, irregular stringers of argillaceous material which give the rock a striped appearance and a knobby surface when exposed along bedding planes. The cephalopod Meekoceras was identified at many horizons in this unit. McKee (1938) describes a Meekoceras-bearing zone in the basal part of the Moenkopi formation in the Mineral Mountains but records no thickness. Gregory (1950) has described Meekoceras from the Virgin limestone member of the Moenkopi at Zion Park. On the above evidence and on the basis of similar stratigraphic position and lithology this limestone unit is correlated with the Virgin limestone member of the Moenkopi formation in adjoining localities.

Overlying the Virgin limestone member are 1,430 feet of variegated red to green shales and siltstones which form a steep slope but the lithology is mainly concealed by soil and vegetation. Gypsiferous beds were not recognized and no lithologic unit similar to that of the Shnabkaib (Gregory, 1950) was found, but the great thickness of this unit appears to indicate that it is composed of the middle red, Shnabkaib, and upper red members of the Moenkopi (Gregory, 1950). The Moenkopi as a whole has an aggregate thickness of 1,893 feet and primary structures such as ripple marks, mudflows, contemporaneous folds and raindrop impressions are beautifully preserved in the variegated shale and siltstone units.

SHINARUMP FORMATION

The top of the Moenkopi is an erosional surface cut by channels filled with a conglomeratic unit consisting of 133 feet of sandstone, quartzite, shale and conglomerate. The thickness of this unit is extremely variable due to channel filling. This conglomerate unit is correlated with the Shinarump formation and consists of interbedded sandstone and shale with some conglomerate lenses in the lower quarter of the unit. The upper part of the formation consists of massive sandy quartzite ranging from fine grained quartz arenite to conglomerate and containing occasional pebbles in the fine grained lenses. Several silicified logs were found, but in the main the plant remains are in the form of "trash," i.e. twigs, leaves, etc. that are either silicified or have been leached leaving only a mold. Fillings of small channels, or scour pools, are evident on examination of most outcrops, and the sandstone lenses are generally cross bedded. The unit forms a massive to medium bedded, resistant ledge, but is not persistent in outcrop.
CHINLE FORMATION

The Shinarump is overlain by 398 feet of variegated sandstone and shale with a color range which includes yellow, gray, red, purple and black. The main part of the unit consists of thin bedded, fissile shale containing calcareous nodules and thin stringers of intraformational conglomerate. The upper one quarter of the unit consists of interbedded sandstone and shale, the sandstone lenses making up 90% of the thickness. The shales and sandstones are a deep red in color with occasional thin white layers. The top 8 feet of the unit consist of green and red sandstone containing clay pellets and a few layers of coarse conglomerate containing shale fragments and fresh water pelecypods. On the basis of similar lithology and stratigraphic position this unit has been correlated with the Chinle formation of adjoining areas.

NAVAJO FORMATION

The Chinle formation is overlain by 2,200 feet of medium grained, tan, buff weathering, cross bedded, highly indurated quartzite with rare lenses of gray siltstone. The bottom 5 feet of this unit are a coarse grained sandstone. The quartzite weathers into massive blocks were measured, but forms a sheer cliff to the north. This unit is believed to represent the Navajo formation, but it may include the Kayenta and Wingate formations in the lower few hundred feet. No changes in lithology were noted on which to base any division of the units but the Wingate formation has been reported from the San Rafael Swell (Gilluly and Reeside, 1928) and Circle Cliffs (Gregory and Moore, 1931) to the east, and from the Kaiparowits plateau (Gregory and Moore, 1931) and Zion Park (Gregory, 1950) to the south; and the Kayenta has been reported from Zion Park (Gregory, 1950). The Todilto (?) formation has been reported from the San Rafael Swell (Gilluly and Reeside, 1928) and Circle Cliffs (Gregory and Moore, 1931), and is probably equivalent to the Kayenta (Baker, Dane and Reeside, 1947).

CARMEL FORMATION

The Navajo formation is overlain by a poorly exposed gray limestone which ranges from calcarenite to calcisiltite with occasional lenses of limestone conglomerate. This formation is thought to be equivalent to the Carmel formation reported from adjoining areas (Gregory and Moore, 1931; Gilluly and Reeside, 1928; Gregory, 1950; Thomas and Taylor, 1946). The top of the Carmel formation represents an erosional surface at Marysvale and probably only the basal parts of the unit are preserved. In places the overlying Bullion Canyon volcanics lie directly on the Navajo formation
making it evident that the Carmel where present represents erosional remnants. The thickness where measured ranges from 0 to 100 feet, but greater thicknesses may exist.

BULLION CANYON VOLCANICS

The sedimentary strata are overlain unconformably by the latites and andesites of the Bullion Canyon Volcanics (Callaghan, 1939). A generalized stratigraphic section is given in Figure 9, and a correlation chart showing the relationship of the rocks at Marysvale to those of adjoining areas is given in Figure 10.

The Bullion Canyon Volcanics at the top of the Tushar Range are altered to form alunite bodies of both the vein and replacement type. This alteration is believed related to base metal and possible uranium mineralization at depth, but further study will be required to establish this relationship. The general structural pattern and lineation of the alunite and base metal occurrences are highly suggestive of this relationship. Reference is made to Figure 7.

URANIUM MINERALIZATION

Uranium occurs in the sedimentary rocks associated with the base and precious metal mineralization. Significant concentrations of uranium have been noted at three localities: the 3,400 foot area in the PTH Tunnel of the New Deer Trail mine, the Shamrock mine, and the Great Western mine. Radioactive material from the PTH Tunnel has been analyzed by Miss P. K. Hamilton and the results are presented on pages 52 and 53 in this report. The radioactive material occurs in an alteration zone on the border of a lead-zinc replacement body. Polished surfaces of the material from the replacement body have been examined and found to contain galena, sphalerite, covellite, chalcopyrite, pyrite and tetrahedrite in a carbonate matrix. X-ray fluorescence analysis shows the presence of selenium in the ore material slightly in excess of 1%. Recent workings have developed a small amount of fluorescent, radioactive material believed to be autunite, according to the mine operators.

Radioactive specimens from the Great Western mine have been analyzed by the A. E. C. ore buying station at Marysvale and found to contain commercial grade uranium. Mineralogical examination of several specimens using X-ray diffraction and polished surface methods has led to the identification of pitchblende as the radioactive material. X-ray diffraction data are given in Table 20. Examination of polished surfaces indicates that the pitchblende occurs in minute grains disseminated in the base metal ore minerals which include galena, sphalerite, covellite, chalcopyrite, pyrite, malachite, azurite and cerussite in a quartz matrix. The
material examined was taken from the top of the inclined slope driven along a metal-bearing quartz vein in the westernmost part of the workings. The samples assayed at the ore buying station were found in a pile of loose material at the bottom of an inaccessible vertical raise at the juncture of the main north-south drift and the east-west crosscut. None of this material was accessible for collection in place. The Great Western mine is near the contact of the Navajo quartzite and the Bullion Canyon volcanics. Near and in the crosscut the volcanics have been infaulted and are cut by the mine workings. In places the volcanics are highly altered. The ore bodies that have been developed consist of base metal accumulations in quartz veins which strike generally north-south and dip to the west. The quartzite shows no apparent alteration near the veins.

Material shipped to the authors by the operators of the Shamrock mine and said to have been collected on a tunnel face in the southernmost part of the workings has been examined by X-ray diffraction methods and under the binocular microscope and found to contain tiny, well developed crystals of meta-autunite. The meta-autunite appears to be disseminated along fractures in the base metal-bearing quartz vein. Base metal minerals identified include galena, sphalerite, covellite and chalcopyrite. The meta-autunite appears to be in close association with the covellite with which it has been found to be intergrown in several instances. X-ray data on the meta-autunite from the Shamrock mine are given in Table 21. The Shamrock mine is similar to the Great Western mine in mineralogy and ore emplacement with the exception of the uranium mineralization. In all three occurrences the uranium mineralization is associated with base metal ore bodies, and the uranium minerals appear to be late in the paragenetic sequence.
Fig. 9. GENERALIZED STRATIGRAPHIC SECTION, DEER TRAIL MTN.
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#### X-ray Diffraction Data Pitchblende

**Great Western Mine**

Cu radiation, Ni filter

### Table 21

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#### X-ray Diffraction Data Meta-autunite

**Shamrock Mine**

Cu radiation, Ni filter
Figure 10. CORRELATION OF MARYSVALE SECTION WITH ADJOINING AREAS.
REFERENCES


X-RAY METHODS APPLIED TO THE STUDY
OF THE TIEMANNITE-METACINNABAR GROUP

by
Philip M. Bethke and Paul F. Kerr

ABSTRACT

X-ray methods are applied to the study of the mineral group tiemannite-metacinnabar; mercuric sulfide and selenide minerals crystallizing in the isometric crystal system. X-ray diffraction and fluorescence data indicate that a complete isomorphous series exists between the two minerals. Sulfur and selenium are shown to be dia
dochic in this mineral group, and their complete mutual substitution is indicated by the data. The relationship between unit cell dimension and chemical composition is presented in quantitative form. The presence of onofrite at Marysvale, Utah, previously discredited is re-affirmed.

ACKNOWLEDGMENTS

The writers wish to extend their thanks to Dr. Brian Mason of the American Museum as he provided several mineral specimens, and to Dr. William Foshag of the National Museum who provided the writers with a specimen of tiemannite from Tilkerode. The writers are greatly indebted to both. Special appreciation is extended to Messrs. Ronald Arnott and William Cohen whose suggestions and criticisms were of great assistance in the analysis of various specimens.

INTRODUCTION

The mineral group tiemannite-onofrite-metacinnabar includes the mercuric sulfide and selenide minerals crystallizing in the isometric crystal system. All three minerals are structurally similar to sphalerite, crystallizing in the hextetrahedral class of the isometric system, and exhibiting an F43m space group. On this basis these minerals have been classified as belonging to the sphalerite group (Palache, Berman and Frondel, 1944).

The writers' attention was drawn to this mineral group in connection with their investigation of the occurrence of uranium in the older sedimentary rocks in the Marysvale district during the 1953 field season. In the course
of this investigation the Lucky Boy mine was visited and specimens were collected for laboratory examination. The deposit occurs in a carbonate lense of the lower unit of the Kaibab formation just south of the Old Deer Trail mine and the ore minerals replace the limestone along bedding planes (Butler, 1920).

The deposit unfortunately has been mined out, and the ore-gangue relationships obscured, but specimens were taken from the tailings pile and investigated in the mineralogical laboratory at Columbia University. Butler (1920) reported both tiemannite and onofrite from the deposit in association with cinnabar, barite, calcite and quartz, but Short (1931) has discredited onofrite at this locality, claiming it to be a mixture of tiemannite and sphalerite.

MINERAL DESCRIPTIONS

The mineral tiemannite was first described by Marx (1828) from the area near Clausthal in the Harz Mountains, and was given the name selenqueckselber. Naumann in 1855 renamed the mineral tiemannite after Tiemann who first discovered it. Tiemannite is reported in Dana's System of Mineralogy as a mercuric selenide containing small amounts of sulfur and cadmium. The mineral crystallizes in the hexagonal class of the isometric crystal system, and where found in crystals has a tetrahedral external form. Specimens are steel gray to blackish gray with a black streak, and exhibit no cleavage. The specific gravity varies from 8.19 to 8.47 (Palache, Berman and Frondel, 1944). The space group and unit cell dimensions were reported by Hartwig as F43m and a₀ 6.069 plus or minus .006. Hartwig's analysis was on a specimen from the Charlotte mine near Clausthal, Germany.

Metacinnabar was first described by Moore in 1870 from the Redington mine in Napa County, California, as a black, amorphous mineral with formula HgS. The mineral crystallizes in the hexagonal class of the isometric system and has a tetrahedral habit where found in crystals. Specimens are gray-black with a metallic luster and black streak and exhibit no cleavage. The specific gravity varies from 7.643 to 7.678 and the chemical formula is HgS with small amounts of Zn and Se substituting for Hg and S respectively (Palache, Berman and Frondel, 1944). The space group and unit cell dimensions are reported by Lehmann (1924) as F43m and a₀ 5.854 respectively.

The mineral onofrite was first described by Rose (1839) from San Onofre, Mexico and given the name selenschwefelqueckselber. Haidinger (1845) renamed the mineral onofrite. As described by Hintze (1904) onofrite is a blackish-gray mineral with a hardness of 2 to 3 and a
specific gravity of 7.6 to 8.1. The chemical formula of onofrite is Hg(S, Se) with a selenium:sulfur ratio of at least 1:6. Early authors considered onofrite an isomorphous mixture of tiemannite and metacinnabar on the basis of chemical analyses, specific gravity determinations, and similarity of crystallization. Onofrite is now classed as a selenian variety of metacinnabar containing selenium in substitution for sulfur in a ratio of at least 1:3.9 (Palache, Berman and Frondel, 1944).

**X-RAY DIFFRACTION DATA**

Two specimens of tiemannite from the Harz Mountains and a specimen of metacinnabar from Lake County, California were subjected to X-ray diffraction analysis along with five specimens of tiemannite-onofrite from Marysvale, Utah. The samples were powdered and run on the Norelco X-ray diffractometer at a scanning rate of 1/40° 2θ per minute. The angles of X-ray reflection were recorded and converted to interplanar spacings (d values), and the reflections for each mineral were indexed. Lattice constants were computed for each of the eight specimens. The above data are presented in Table 22.

Reference to Table 22 shows a systematic variation of unit cell dimensions (a₀) between the specimens examined. The two specimens from the Harz Mountains, samples 1 and 2, have similar cell dimensions, greater than those of the other specimens. The specimen of metacinnabar from Lake County, California, sample 8, has the smallest cell dimensions. The specimens of tiemannite-onofrite from Marysvale, Utah fall between these two extremes.

**X-RAY FLUORESCENCE DATA**

X-ray diffraction data suggest that the variation in unit cell dimensions is a function of the amount of selenium present in the various lattices. In order to verify this relationship six of the mineral specimens were analyzed for selenium using the X-ray spectrometer. In order to set up a standard of comparison, eleven mixtures of mercuric oxide, selenium metal and sulfur were prepared to simulate mineral compositions of hypothetical minerals within this group containing various amounts of selenium. These prepared mixtures were run on the X-ray spectrometer and the peak heights of the mercury L alpha₁ and selenium K alpha₁ lines were recorded. In order to avoid any error due to variation in sample volumes, the ratio of selenium K alpha₁ to mercury L alpha₁ was calculated and plotted against selenium content of the samples. The resultant curve is presented in Figure 11. After a calibration curve had been prepared, six of the samples investigated by X-ray diffraction methods
### TABLE 22

**X-ray Diffraction Data**

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were analyzed using X-ray fluorescence. The ratio of selenium K \( \alpha_1 \) to mercury L \( \alpha_1 \) was computed and plotted on the calibration curve for each sample to determine the selenium content of the mineral. The data for the calibration of the instrument and analysis of the various samples is presented in Table 23.

SYNTHESIS OF DATA AND CONCLUSIONS

Analysis of the X-ray diffraction data presented in Table 22 shows that there is a systematic increase in unit cell dimensions from metacinnabar to tiemannite from the Harz Mountains. The X-ray fluorescence data show a progressive increase in the amount of selenium in the various samples from metacinnabar to tiemannite from the Harz. When the selenium content is plotted against unit cell dimensions the resultant curve approximates a straight line within the limits of error, and shows that unit cell dimension varies in direct proportion with selenium content in this mineral group. The relationship is shown graphically in Figure 12.

Tiemannite and metacinnabar are structurally similar, both exhibiting an F\(4\bar 3m\) space group. The only variant in the lattice constants is the dimension of the unit cell, which has been demonstrated to vary in direct proportion with selenium content. The ionic radius of selenium minus 2 is 1.91 \(\AA\) and that of sulfur minus 2, 1.85 \(\AA\) (Green, 1953). The above data indicate that selenium substitutes for sulfur in the crystal lattice, and that due to the larger size of the selenium ion the dimensions of the unit cell are expanded. The elements sulfur and selenium are diadochic in this mineral group in that their complete mutual substitution in the crystal lattice is indicated by the data. Sulfur and selenium satisfy the requirements for diadochy (Mason, 1952) in that the difference in ionic radii is less than 15% of the smaller, and both ions have the same charge. Tiemannite and metacinnabar satisfy the requirements for structural isomorphism in that they are chemically dissimilar substances exhibiting the same crystal structure. The above data and considerations indicate that this mineral group exhibits ionic substitution in a structurally isomorphous mineral group, and is, in effect, what is commonly termed an isomorphous series. The data indicate that there is a complete series of ionic substitution from pure HgS (metacinnabar) to pure HgSe (tiemannite), but it is possible that there are breaks in this series between the samples analyzed. Further investigations must be conducted before a definite statement can be presented.

On the basis of X-ray data presented above it would appear that onofrite is present at Marysvale, Utah as a mineral species rather than a mixture of tiemannite and sphalerite, contrary to Short (1931). The terminology of this mineral group is exceedingly loose, and it would seem desirable that a new nomenclature be adopted. Present investigations are not sufficient to form a basis for such a terminology.
TABLE 23

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<td>45.6</td>
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X-RAY FLUORESCENCE CALIBRATION CURVE FOR SELENIUM CONTENT

FIGURE 11
FIGURE 12

UNIT CELL DIMENSION vs. SELENIUM CONTENT

Percent Selenium

Unit Cell Dimension (Å units)
REFERENCES


Green, Jack (1953) Geochemical table of the elements for 1953, G. S. A. Bull. v. 64, pp. 1001-1-12.


Lehmann, W. M. (1924) Röntgenographische untersuchungen an natürlichem und synthetischem metacinnabarit (HgS), Zeitschrift für Kristallographie, band 60, pp. 378-413.


