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Series A.

CATEGORY (Special)

UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

SUMMARY OF THE RESEARCH WORK OF THE TRACE ELEMENTS SECTION, GEOCHEMISTRY AND PETROLOGY BRANCH, FOR THE PERIOD JANUARY 1 - JUNE 30, 1951

by

John C. Rabbitt

September 1951

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Trace Elements Investigations Report 167

USGS-TEI REPORT 167

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by

John C. Rabbitt

INTRODUCTION

This report summarizes the research work of the Trace Elements Section, Geochemistry and Petrology Branch, for the period January 1 - June 30, 1951. Two earlier summaries have been issued.[1,2]

The research work of the Section consists of the following laboratory and related field studies:

1. Mineralogic and petrologic investigations of radioactive rocks, minerals, and ores.
2. Investigations of chemical methods of analysis for uranium, thorium, and other elements and compounds in radioactive materials, and related chemical problems.

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[1] Rabbitt, John C., Summary of the research work of the Trace Elements Section, Geochemistry and Petrology Branch, for the period April 1, 1948 - December 31, 1950: U. S. Geol. Survey Trace Elements Investigations Rept. 148-A, June 1951.

[2] Rabbitt, John C., Summary of the research work of the Trace Elements Section, Geochemistry and Petrology Branch, for the period January 1 - March 31, 1951: U. S. Geol. Survey Trace Elements Investigations Rept. 151, May 1951.

3. Investigations of spectrographic methods of analysis for a wide variety of elements in radioactive materials.
4. Investigations of radiometric methods of analysis as applied to radioactive materials.

It should be emphasized that the work undertaken so far is almost entirely in the nature of investigations supporting the field appraisal of known uraniferous deposits. A program of more fundamental research, particularly in the geology and geochemistry of uranium has been approved by the Division of Research of the Atomic Energy Commission and will be started in fiscal year 1952.

This report does not deal with the routine analytical work of the Section nor with the public-sample program. The analytical work and the public-sample program have been described in separate reports.3,4/

Special thanks are due members of the Section engaged in the research work who have supplied material for this report and who are referred to in the text and to Earl Ingerson, Chief of the Geochemistry and Petrology Branch for his critical review, to Jane Titcomb of the editorial staff of the Section for editing the report, and to Virginia Layne of the same staff, for typing the manuscript and the multilith mats.

3/ Rabbitt, John C., Numerical summary of the analytical work of the Trace Elements Section, Geochemistry and Petrology Branch, for fiscal year 1951: U. S. Geol. Survey Trace Elements Memorandum Rept. 250, August 1951.

4/ Deul, Maurice, Summary report on public samples received 1949-1950, Trace Elements Section Washington Laboratory, Geochemistry and Petrology Branch: U. S. Geol. Survey Trace Elements Memorandum Rept. 124, June 1951.

MINERALOGIC AND PETROLOGIC INVESTIGATIONS

Introduction

The mineralogic and petrologic investigations of the Section, under the direction of Theodore Botinelly, are organized in the following projects:

1. The phosphate project
2. The Colorado Front Range project
3. The Colorado Plateau carnotite project
4. The properties of uranium minerals project
5. The lignite project
6. Miscellaneous projects

The laboratory work is supplemented by field work by members of the Section, and it is closely coordinated with the work of the resident field geologists in the areas studied.

The phosphate project

One of the principal aims of the work on the Florida phosphate deposits in recent months has been to find stratigraphically equivalent leached and unleached material in the Bone Valley formation because only by knowing the characteristics of the unleached rock can a full comprehension of the subsequent leaching be obtained. This goal has now been attained by the study described in "The stratigraphy of the upper part of the Bone Valley formation and its relation to the leached zone," by Z. S. Altschuler, Elizabeth

B. Jaffe, and Edward Dwornik (U. S. Geol. Survey Trace Elements Memorandum Rept. 237, May 1951). The abstract follows:

> The two topmost zones of the Bone Valley formation at the Homeland mine are a phosphate-cemented quartz sandstone and an underlying pebbly and clayey quartz and phosphate sand characterized by graded bedding. This sequence is found in every other area of the land-pebble field. Therefore, these zones are considered to be stratigraphic units and the transition between them to be a key horizon.
>
> The leached zone of the Bone Valley formation encompasses either the upper or both of these units. As a result, rock from the leached zone may have two different aspects. The upper unit is not obviously porous and in places has the appearance of a pure quartz sand. Large cavities have been developed in the lower unit during leaching. It is emphasized that the leached zone is a single secondary feature that was produced in two different stratigraphic units which respond differently to leaching, and that it is not inherently composed of two subzones.

Now that this marker horizon has been established a chemical study is in progress comparing stratigraphically equivalent leached and unleached material. Several suites of stratigraphically matched samples are being analyzed for CaO, P_2O_5, and Al_2O_3. Analyses for uranium have been completed.

Each suite of samples represents a stratigraphic section, and the uranium distribution in each section is found to vary considerably. Until the CaO and P_2O_5 analyses are completed, the variation of uranium content can best be compared with field observations. When the analyses are completed, correlation with P_2O_5 content will be made. It is noticeable that two types of variation occur within these suites, each suite embracing the leached zone and several feet of underlying unleached material. First, there is a variation in the amount of phosphate minerals present. Thus, in the base of one section the uranium contents (in one-foot intervals up the section) are as follows: 0.014, 0.007, 0.006, and 0.014 percent. This is in good agreement with the field observations on the relative amounts of clay and phosphate minerals. Second, in the next 2 feet of section the uranium content goes up to 0.020 percent. This increase is due to the secondary enrichment caused by leaching. It coincides with the occurrence of pebbles that are softened and altered in the outer parts. The altered pebbles and the increased uranium contents are found below that part of the section showing secondary porosity.

This chemical study is being complemented by a study of the mineralogic and textural changes within the rock. In addition, field work during March carried forward a study of the leached zone and its relation to the overburden sands. There are many places where the overburden sands seem to grade into one another. The questions raised by these occurrences concern the original thickness of the leached zone and the overburden sands, their origins, and the amount of material originally available for the concentration of the uranium in the present leached zone. Samples have been collected for the investigations of these problems.

It is desirable to have some information on the ultimate textures of the different phosphorites. The physical and chemical alterations undergone by many phosphorites or the clouding effect of included organic matter make such studies difficult. Many of the apatite nodules from the Hawthorn formation, underlying the Bone Valley formation, are mineralogically pure and texturally simple. This is in contrast to most of the nodules and pebbles of the Bone Valley because the latter have gone through a history of successive reworkings. A study of the Hawthorn nodules involving thin-section and electron-microscope investigations, is now in progress.

Samples were collected in May from representative localities in the "hardrock" phosphate belt of Florida and a study of these samples is in progress.

Work in the synthesis of apatite by Sol Silverman, Jeanne Weiser, and Ruth Fuyat has resulted in the report "The quantitative determination of calcite associated with carbonate-bearing apatites," by Sol Silverman, Ruth Fuyat, and Jeanne Weiser (U. S. Geol. Survey Trace Elements Investigations Rept. 118, in preparation). The abstract follows:

> The CO_2 combined as calcite in carbonate-bearing apatites has been distinguished from that combined as carbonate-apatite, or present in some form other than calcite, by use of X-ray powder patterns, differential thermal analyses, and differential solubility tests. These methods were applied to several pure apatite minerals, to one fossil bone, and to a group of phosphorites from the Phosphoria formation of Permian age from Trail Canyon and the Conda mine, Idaho, and the Laketown district, Utah. With the exceptions of pure fluorapatite, pure carbonate-fluorapatite, the fossil bone, and one phosphorite from Trail Canyon, these substances contain varying amounts of calcite, but in all the samples an appreciable part of the carbonate content is not present as calcite. The results of solubility tests, in which the particle size of sample and the length of solution time were varied, imply that the carbonate content is not due to shielded calcite entrapped along an internal network of surfaces.

In addition to the synthesis of fluorapatite and chlorapatite by dry fusion (see TEIR 148-A, p. 15), a strontian chlorapatite,

$3Ca_3(PO_4)_2 \cdot SrCl_2$, has been prepared by the same method. All three apatites show typical apatite X-ray patterns. Chlorapatites containing 1.6 percent and 2.2 percent uranium had previously been prepared by dry fusion from a mixture of tricalcium phosphate. These products have now been X-rayed and show typical fluorapatite powder patterns. Apparently the presence in these apatites of the uranium mentioned above does not alter the fluorapatite X-ray pattern.

In an attempt to distinguish by X-ray means between hydroxylapatite and tricalcium phosphate it was found that the typical X-ray pattern given by hydroxylapatite was unaltered after heating the sample to 1200°C. Synthetic tricalcium phosphate, after heating to 800°C, showed significant deviations from the typical apatite pattern. The pattern after the change does not fit the beta tricalcium phosphate pattern as established for whitlockite by Clifford Frondel of Harvard University. There is enough difference in the X-ray pattern of tricalcium phosphate after heat treatment to permit discrimination between this material and hydroxylapatite.

Another approach is being developed by Silverman and his colleagues for determining the nature of the carbonate content of certain apatites. This consists of removal of the entire CO_2 content of the sample at 1200°C in vacuum (10^{-3} mm Hg). The ignited product is then mixed with water and the mixture titrated for hydroxyl ion content with a standard HCl solution. If the CO_2 is present as calcite, then each molecule of CO_2 lost will result in one molecular weight of CaO in the residue. After addition of water the $Ca(OH)_2$ formed will, under these conditions, be stoichiometrically equivalent to the CO_2 lost.

Apparatus for this determination has been assembled, and test runs have been made on artificial mixtures of pure fluorapatites and calcium carbonate. The method was found to be reliable in detecting the amount of calcite originally present to within 1 percent. Optically homogeneous carbonate-apatite ("staffelite") will be analyzed by this method and the results compared with all available data (from solubility tests and differential thermal analysis) on the same material.

Studies of different apatites were also made by Edward Dwornik by electron-microscope methods. Polished surfaces were prepared on specimens of "carbonate"-apatite (Staffel, Germany); replicas were made of these surfaces by standard techniques and the replicas were studied under the electron microscope. The polished surfaces were then etched with triammonium citrate to leach selectively any calcite present and replicas were made of the leached surface and studied under the microscope. This method might show the presence of calcite as inclusions too small to be detected under the optical microscope. No evidence of calcite inclusions was seen but the study has not progressed far enough yet to rule out the presence of such inclusions.

Further work on the distribution of uranium and its relation to other constituents of the rich phosphate beds of the Phosphoria formation of the northwest is reported in "Distribution of uranium in rich phosphate beds of the Phosphoria formation," by M. E. Thompson (U. S. Geol. Survey Trace Elements Investigations Rept. 142, February 1951). The abstract follows:

> Five sets of "close" samples were analyzed radiometrically for uranium, and chemically for P_2O_5, CaO, organic matter, and loss on ignition. A Rosiwal analysis was made of thin sections of one set of samples. The results of the analyses are plotted on graphs and on scatter diagrams, and coefficients of correlation are given for uranium with CaO, P_2O_5, organic matter, and loss on ignition. Preliminary studies seem to indicate that the concentration of uranium in these samples of phosphate rock is not due wholly to phosphate content, but may depend in part on organic matter or on other components. The correlations of uranium with P_2O_5 are poor in the groups of samples with smaller amounts of uranium but are good in the groups of samples containing more uranium.

Pellets from the Phosphoria formation are being studied for variations in size and chemical composition. Two samples from widely separated areas but from approximately the same stratigraphic position were found to have almost identical size characteristics. Spectrographic analyses of 10 samples from a single bed show that there is little lateral variation in elements reported spectrographically.

The adsorption of uranium by apatite in the pellets is being studied by Robert S. Jones by immersing crushed and sized pellets in solutions of uranyl salts. The best results have been obtained by using uranyl sulfate in concentrations of 0.010 to 0.025 percent. It has been noted that marked increase in the uranium content of the pellets occurs and that the calcium content of the solutions is also increased. It is possible that this signifies the replacement of calcium ions in the apatite by uranium ions. A report on this study is in preparation by Jones.

Differential thermal analysis of Phosphoria material is in progress by Silverman and Weiser. It is necessary to obtain curves for standards representing minerals commonly found in the Phosphoria formation or minerals that would be useful in the study of the Phosphoria. These include clay minerals, staffelite, selenite, calcite, tricalcium phosphate, bone, and quartz for the quartz inversion point. The study of the organic matter in the phosphate beds of the Phosphoria formation poses a special problem because the composition is not known and it is impossible to obtain a pure sample. To overcome this difficulty the organic matter from several types of rocks found in the Phosphoria was concentrated. Differential thermal curves of the organic concentrates of samples for which an exothermic reaction was observed at 1050°C clearly indicate that this reaction is due to the organic matter present. In the same manner, organic concentrates from samples giving a broad exothermic reaction at 800° to 900°C produced a similar reaction at 800° to 900°C. The difference seems to be due to two kinds of organic matter which at present can be called only the "black" organic matter and the "brown" organic matter. Identification of these materials is being attempted.

Differential thermal analysis has also resulted in data for samples representing different mineralogic combinations commonly found in the phosphatic shale member of the Phosphoria formation in southeastern Idaho. These combinations are (1) predominantly apatite, (2) apatite and calcite, (3) apatite and organic matter, (4) apatite and detritals (mainly quartz), (5) detrital material (mainly quartz with some clay), and (6) detrital material with organic matter. Each of these samples is already chemically and petrographically documented. Further work on these samples is in progress.

The Colorado Front Range project

Relation of pitchblende deposition to radioactive bostonite dikes in the Central City district, Colorado

Mineralogic and chemical studies of the fine-grained radioactive bostonite and bostonite-porphyry dikes of the Central City district, Colo., were continued by George Phair and Kiyoko Onoda in an effort to determine the radioactive constituents. It has not been possible so far to separate the radioactive constituents by means of heavy liquids or magnetic separators because (1) the small grain size of the groundmass necessitates crushing the material to -200 +325 mesh, and (2) the desired accessory minerals, although widely distributed, are scarce in these rocks.

To find out where the uranium is concentrated, hand-picked separates of the feldspar phenocrysts and of the groundmass from three samples taken from three different dikes were analyzed for uranium; the uranium content of the bulk samples was known from previous analyses to range from 0.004 to 0.007 percent. As was expected the results showed a two- to threefold concentration of uranium in the groundmass as compared to the phenocrysts.

A sample of bostonite porphyry from the same region (Red Lyon Lodge, Idaho Springs, Colo.) had earlier been shown by Waldemar Schaller of the Geological Survey to have a ZrO_2 content of 0.11 percent, and it seemed possible that much of the uranium and thorium in the bostonites under study might be tied up in zircon. Twenty samples (16 from the Cental City district and 4 from bordering areas) were analyzed chemically for ZrO_2. The results ranged from 0.02 percent to 0.22 percent ZrO_2 and showed that most of the bostonites are enriched in ZrO_2, some highly so when compared to the average alkalic rock. The figure of 0.22 percent ZrO_2 represents a ZrO_2 content about 10 times that of a normal granitic rock.

The bostonites high in uranium tend to be high in ZrO_2. However, the ZrO_2 contents are not high enough to account for more than a small part of the total uranium by solid solution in zircon even when maximum allowance of 1 percent uranium in zircon is invoked (as postulated by E. S. Larsen, Jr., personal communication). It follows, therefore, that most of the uranium in the rock is not in the zircon and that the ZrO_2 and most of the uranium are concentrated in separate mineral phases.

Study of thin sections and of heavy-mineral separates shows that interstitial fluorite is one of the most abundant accessory minerals in samples from the three bostonite dikes with the highest radioactivity. The high ZrO_2 content and the presence of interstitial fluorite in these dikes suggest that volatiles were active during the late stages of emplacement of the dikes. In this connection it is interesting to note that both ZrF_4 and UF_6 are volatile at only slightly elevated temperatures at atmospheric pressures. Moreover, uranium is associated with many fluorite deposits, including those at Jamestown, Colo., in the Thomas Range, Utah, and at Wölsendorf, Bavaria. The question as to the role of these postulated volatiles in uranium deposition is under study.

Additional thorium analyses have been made on samples of the bostonites, and a comparison of these analyses with the chemical uranium analyses and with the radiometric results shows that both the uranium and thorium in these rocks are in equilibrium with their daughter products.

The radioactivity of fluorite from the Blue Jay mine, Jamestown, Colorado

The work on hydrothermal uranothorite is described in "Hydrothermal uranothorite in fluorite breccias from the Blue Jay mine, Jamestown, Boulder County, Colorado," by George Phair and Kiyoko Onoda (U. S. Geol. Survey Trace Elements Investigations Rept. 144, March 1951). The abstract follows:

> In the course of thin-section and heavy-mineral studies of fluorite breccias from Jamestown, Colo., the mineral thorite, variety uranothorite, was identified on the basis of chemical, spectrographic, optical, and X-ray data. This uranothorite is compared with uranothorite from South Westland, New Zealand, described by Hutton (1950), and with similar material from an Alaskan placer sample studied by the writers. These uranothorites are in turn contrasted with the uranothorites described in the older literature all of which represent hydrated material. Thorium halos in fluorite are described, probably for the first time. The uranothorite associated with the fluorite breccias is clearly of hydrothermal origin--unlike all previously described thorites and uranothorites with one very doubtful exception.

The authors believe that uranothorite will be found as a constituent of other hydrothermal veins now that the physical properties of the unaltered material are known.

A study of the radioactive constituents in the cerite deposit near Jamestown was begun late in June.

The use of the Ra/U ratio of pitchblende as a quantitative measure of the recent leaching

No work was done on this project during the last six months. A report on this work "Some factors in the differential leaching of uranium and radium in pyritic mines and mine dumps," by George Phair is in preparation but is being held up pending more laboratory data.

The Colorado Plateau carnotite project

Preliminary graphs and charts based on the lead isotope ratios from lead extracted from carnotite ores of the Colorado Plateau have been completed. These will form part of a report "A preliminary report on the lead-isotope ages of the Colorado Plateau carnotite ores," by Lorin Stieff and T. W. Stern which was nearing completion at the end of June. This isotope work has been done on 45 Plateau samples from different geographic and stratigraphic localities.

Dr. Hans Suess who recently joined the staff of the Geological Survey has reviewed the calculations with Stieff and Stern and has made many helpful suggestions as to the calculations and graphs and the manner of their presentation.

The calculations indicate that, so far as the geologic age of the deposits can be determined from the isotope ratios, the deposits are Tertiary. Suess, Stieff, and Stern are in agreement on this point. Additional samples to fill in gaps in the record will be collected during July and August, and data from them will be added to the report.

Work on the clays underlying the carnotite deposits is reported in "Red and gray clay underlying ore-bearing sandstone of the Morrison formation in western Colorado," by Alice Dowse Weeks (U. S. Geol. Survey Trace Elements Memorandum Rept. 251, May 1951). The abstract follows:

> A preliminary study of the clays underlying the ore-bearing sandstone of the Morrison formation, Colo., has tentatively identified the chief clay mineral as hydrous mica. Complete and partial chemical analyses show that the red clay contains more total iron and ferric iron than the gray clay. Spectrographic analyses of minor constituents show no significant difference between the red and gray except in iron content. Quartz and carbonate have a wide range in quantity that is not related to the color of the clay. Insufficient evidence is available from these specimens to indicate whether the gray altered from the red clay.

Further work on these clays by Mrs. Weeks included mineralogic study of six samples of clay from directly beneath the ore-bearing sandstone at Cougar mine, Lower Group, Slick Rock area, Colo. Three gray clay samples, taken 2, 5, and 17 inches below the sandstone, contained both montmorillonite and hydromica. Gray clay at 30 inches, mottled red and gray clay at 33 inches, and red clay at 48 inches below the sandstone contained hydromica and no montmorillonite. All six clays contained quartz and some calcite and halite. These samples are the first to show even a slight suggestion of different mineralogy in gray clay directly under the ore from that of clay farther from the ore. This problem will be studied further.

A set of 50 clay and siltstone samples representing six members of formations from eight localities in western Colorado and eastern Utah are also being studied by Mrs. Weeks. The purpose of this work is to look for distinctive mineralogic features in each formation and to detect regional differences and other evidence bearing on the origin of the clays and siltstones. It was agreed that size analyses would be done on the clay samples by R. A. Cadigan of the Geological Survey's Mineral Deposits Branch in the sedimentation laboratory in Grand Junction, Colo. So far the work has entailed 150 X-ray powder photographs, 130 spectrographic analyses, differential thermal analysis, electron microscope work, radiometric and chemical analyses, and optical crystallographic examination.

A chart summarizing the mineralogy of the samples by formations and localities has been prepared for the field stratigraphers at Grand Junction.

Although the number of samples from each formation is small, certain characteristic are suggested by this study:

1. The clay mineral of the Summerville formation (Upper Jurassic) is chiefly hydromica with some kaolinite and no montmorillonite. Gypsum seems to be much less common than is indicated in published descriptions of the Summerville.
2. The Salt Wash sandstone member of the Morrison formation (Upper Jurassic) also is characterized by hydromica. A small part of the samples also contains some kaolinite and montmorillonite.
3. The Brushy Basin shale member, upper part of the Morrison formation, consists chiefly of montmorillonite

clay in the Utah samples, but the Colorado samples have appreciable amounts of hydromica and kaolinite.

4. The Burro Canyon formation (Lower Cretaceous) contains chiefly hydromica with some kaolinite and montmorillonite.
5. The Cedar Mountain formation (Lower Cretaceous) of the Woodside anticline, Utah, which may be the equivalent of the Burro Canyon, was represented by only two samples; one of highly swelling montmorillonite clay and one of nonswelling hydromica clay.
6. Two samples of the Dakota formation of Upper Cretaceous age consisted chiefly of kaolinite with some hydromica.

The montmorillonite or bentonite of the Brushy Basin shale member is thought to be volcanic in origin because of the presence of euhedral igneous rock minerals and several water-soluble minerals such as thenardite, burkeite, trona, soda niter, and halite; there is a lack of detrital minerals. The kaolinite clays of the Dakota formation were probably derived from residual clay on a land surface eroded during the interval represented by the regional unconformity at the base of the Dakota.

Heavy-mineral separations have been made on the sands in certain of these samples: barite and pyrite have been found in one sample; detrital biotite, zircon, and tourmaline have been found in some; euhedral zircons, apatite, magnetite, and biotite have been found in others. Only one sample contained fossils which seem to be the microscopic calcareous algae, Chara. These have been submitted for identification to the Paleontology and Stratigraphy Branch of the Survey.

A report on this work by Mrs. Weeks was nearing completion at the end of June.

Chemical analysis by A. M. Sherwood of the Section made on a 4-g sample of a red-brown iron-vanadium mineral from the Jo Dandy mine, Montrose County, Colo., showed that the sample was too impure to determine the correct mineral formula. This mineral is thought to be new, and attempts to obtain a pure sample will continue. The material occurs with hummerite and pyrite in gray clay.

A single-crystal X-ray study is being made by Evelyn Cisney and Mrs. Weeks of an unusual specimen of hewettite from Utah collected by Leonard B. Riley of the Section. Previously available hewettite has been too fine-grained for single-crystal study and was probably dehydrated. The material occurs as fibers, and care has been taken to keep the material hydrated. The new material, in X-ray powder patterns, seems to be monoclinic instead of orthorhombic which is the crystal system assigned to hewettite by earlier workers.

Attempts have been made by Eleanor Thompson to separate the minerals in a so-called "corvusite" ore from the Bitter Creek mine, Montrose County, Colo. Concentrates of montroseite and hydromica were separated but no mineral giving an X-ray pattern similar to "corvusite" was found. It is suggested that "corvusite" is a mixture of vanadium minerals or is amorphous.

Two unusual heavy minerals that had been separated from the Salt Wash sandstone member (Morrison formation) in the sedimentation laboratory at Grand Junction were submitted by Riley for identification. These have been identified as staurolite and "carbonate"-apatite.

A black iron-vanadium mineral from Saskatchewan, Canada, sent in by H. V. Ellsworth was compared with montroseite. The Canadian material is similar but contains more iron and has a higher density.

During the period covered by this report 50 thin sections of material from the Colorado Plateau were covered with nuclear stripping film by Stieff and Stern as part of the general survey of radioactive minerals in the ores of the Plateau. Fifty companion alpha plates were also made. The technique for permanently unsupported nuclear emulsion on uncovered thin sections has been studied in some detail. Completely satisfactory results have not been obtained yet but it is believed that solution of this problem is near. Such mountings should provide high resolution for nuclear tracks, and the use of such high resolution for low-grade material should aid in demonstrating whether uranium is systematically distributed throughout the rock.

The properties of uranium minerals project

A meeting was held at Harvard University on May 29, attended by John C. Rabbitt, Theodore Botinelly, and Judith Weiss Frondel of the Survey, and Clifford Frondel of Harvard, on how to complete the work on the proposed monograph on the properties of uranium minerals.

Because of urgent business, George Switzer of the National Museum was unable to attend. It was decided that enough data have been obtained so that work on writing the monograph could begin. It was further decided that the title of the work will be "The Mineralogy of Uranium" and that an endeavor will be made to publish it as Monograph No. 1 by The American Mineralogical Society. A tentative outline for the work is as follows:

- Introduction
- Occurrence and association of uranium minerals
- Geographic distribution of uranium minerals
- Description of uranium minerals
- Methods of identification
- Determinative tables
- References

The four people mentioned above together with George Switzer will write the different chapters and the work will be under the editorship of Rabbitt. It is expected that preparation of the monograph will begin in the fall of 1951.

Work on the metamict minerals by Joseph Berman continued. Heat treatment has now been standardized, and a file has been compiled of X-ray patterns from such treated minerals.

In the standard method the mineral is heated in a Dietert tube furnace with sillimanite tubes in an atmosphere of water-free helium. Duplicate samples are heated in air for comparison purposes. Temperatures used range from 850° to 1200°C and variations in heat for certain minerals are necessary. Certain anomalous results have been found, notably with thorites and related minerals.

The atmosphere necessary for recrystallization of metamict minerals by heat treatment depends on the composition of the mineral; some crystallize at low temperatures (allanite at 600°C) in air, others crystallize only at temperatures of 1200°C or higher. Some minerals on heating in air over a Meeker burner will recrystallize sufficiently to give an X-ray pattern that will identify the specimen. Others apparently oxidize if even small amounts of water vapor are present in a helium atmosphere, and the heated specimen then gives an X-ray pattern that does not serve to identify the mineral.

Table 1 summarizes some of the work to date on different metamict minerals. The work is continuing, and a report on the work to date is in preparation by Berman.

A selected annotated bibliography on metamict minerals is being prepared by Theodore Botinelly.

The lignite project

Work started late in March by Maurice Deul on a study of the uraniferous lignites of the Dakotas. It is planned to investigate the lithologic and chemical composition of this material with particular reference to the manner of occurrence of the contained uranium. In a visit to the Fuels Branch Laboratory of the Survey

Table 1.--Results of heating metamict minerals in different atmospheres and at different temperatures

Mineral	Atmosphere	Temperature (°C)	Results
Zircon	air inert	1200	Sharp zircon pattern
Thorite (completely metamict)	air	900	ThO_2 pattern
Thorite (completely metamict)	air	1200	Huttonite pattern
Thorite (partially metamict)	air	1200	Huttonite and ThO_2 patterns
Thorite (partially metamict)	inert	1300	Sharp thorite pattern and ThO_2 pattern
Euxenite	air	below 1000	Poor patterns
Euxenite	air	1200	Identifiable euxenite pattern
Euxenite	helium	1100	Identifiable euxenite pattern
Euxenite	helium	1250	Material dissociates
Brannerite	air	750-900	Sharp brannerite pattern
Fergusonite	air	1000-1300	Fergusonite pattern
Fergusonite	helium	1000-1300	Fergusonite pattern
Fergusonite	air	below 1000	Poor patterns
Samarskite	inert	1000	Sharp patterns
Davidite	air	900(?) over Meeker burner	Sharp pattern (davidite ?)
Allanite	air inert	red heat(600 ?)	Sharp pattern
Allanite	air inert	over 1000	Fuses

All minerals were heated at atmospheric pressure.

in Columbus, Ohio, Deul arranged with James Schopf, head of the laboratory, for a cooperative study of the lignites. Schopf will suggest proper methods of sampling and will have thin sections prepared in his laboratory according to standard techniques used for coaly material. He will also make petrographic examinations of the material and arrange for certain proximate and ultimate analyses of the samples by the Bureau of Mines. Radiometric, nuclear emulsion, chemical, spectrographic, and related studies will be made in the Section.

Deul also arranged for a three-day field conference in the Buffalo, S. Dak., area in the middle of July. This conference will be attended by Deul, Schopf, Norman Denson of the Survey, in charge of field work, Professor Farrington Daniels of the University of Wisconsin, and Donald Peppard of the Argonne National Laboratory.

The possibility of adsorption of uranium by organic constituents in the lignites will be studied in close cooperation with Professor Daniels. Professor Daniels will be working on some aspects of this problem involving a study of the geochemistry of uranium in peat bogs and in their constituent plants. He will also investigate problems connected with the extraction of uranium from lignites, problems in which Mr. Peppard of Argonne is also interested. The theoretical study of the chemistry of uranium in lignites has been discussed with S. J. Katz of the Argonne National Laboratory.

During June six selected samples of lignites were ashed and the ash completely analyzed both chemically and spectrographically. Results of this work will be given in the next quarterly report.

Miscellaneous projects

There are many small research projects in the mineralogy and petrology of radioactive minerals in progress in the Section at all times. Many of these are "spot" research studies on small suites of samples sent in, for example, by the field geologists of the Reconnaissance Group. Others are studies on small samples submitted by the Mineralogical Laboratory of the Atomic Energy Commission New York office, by the Atomic Energy Commission Washington office, and by the public. The total of this work looms rather large in the over-all work of the Section.

The studies of Professor E. S. Larsen, Jr., are in a special category. He works in the Section as a special consultant and catalyst, especially in the mineralogic and petrologic work, and his advice is constantly sought in carrying on the research program. He is also making many investigations of his own. At present, he is continuing his work on the determination of the geologic age of igneous rocks by investigating the applicability of the isotope-dilution technique developed by Harrison Brown and his colleagues at the Institute of Nuclear Studies, University of Chicago. The technique is being tried on mineral separates obtained by Professor Larsen from Canadian granites, and the results so far are encouraging.

Work is in progress by Z. S. Altschuler on a suite of samples of phosphate rock from Mona Island, B.W.I. The material occurs in limestone caves, and it was desirable to know its uranium content and whether it is a residual product of weathered limestone or alterations of limestone related to guano accumulations. Eight samples containing 11 different rocks were submitted to the Section for study. There were two fresh limestones, four weathered limestones, and five phosphorites. Chemical analyses were made for P_2O_5, fluorine, and uranium, and it was found that the fluorine and uranium contents were extremely low for marine apatites. In addition, a semiquantitative spectrographic survey of the samples did not show any significant enrichment of the minor or trace elements in the phosphates with respect to the limestones. This can be evaluated by comparing the normally less soluble or less active rock elements which accumulate lateritically, such as Fe, Al, Pb.

It was noted that some of the limestones that contain calcite oolites or fossils also contain phosphate. However, any attempt to decide whether the phosphate is undisturbed or replaced, and if replaced, whether the replacement is diagenetic or epigenetic and continental must await further work which is in progress.

CHEMICAL INVESTIGATIONS

Methods of analysis project

Fluorimetry

The design of a fluorimeter for solutions is described in "A fluorimeter for solutions," by Mary H. Fletcher and E. Ray Warner (U. S. Geol. Survey Trace Elements Memorandum Rept. 252, April 1951). The abstract follows:

> A description of and complete drawings for the construction of a fluorimeter for the measurement of fluorescence of solutions are given. The instrument is sturdy and versatile. It may be used with various phototubes and measuring devices. It is constructed so that phototubes and filters may be changed readily. Sensitivity is controlled easily over a wide range by limiting the size of either the ultraviolet or fluorescent light beam with standard apertures.

Work is continuing in this field, particularly in simplifying present instruments to make them more compact, stable, versatile, and less expensive, and in designing new instruments for special applications.

Methods of uranium and thorium analysis

A study of separations obtainable with sodium carbonate fusion for application to the determination of uranium by fluorimetric procedures in shales, lignites, and monazites is reported in "Fluorimetric determination of uranium in shales, lignites, and monazites after alkali carbonate separation," by Norma S. Guttag and F. S. Grimaldi (U. S. Geol. Survey Trace Elements Investigations Rept. 153, in preparation). The abstract follows:

> Comparative data are presented on separation of microgram amounts of uranium from milligram amounts of various metal ions with Na_2CO_3-K_2CO_3, Na_2CO_3-K_2CO_3-H_2O_2, and Na_2CO_3-$NaClO$. The Na_2CO_3-K_2CO_3 separation procedure is applied to the analysis of shales, lignites, and monazites. This method will determine as little as 0.001 percent uranium in shales and lignites and 0.01 percent uranium in monazites.

An investigation was started by Mary H. Fletcher and F. S. Grimaldi during May of colorimetric and fluorimetric reagents for the rapid determination of thorium. Objectives of the study are as follows:

1. To find sensitive colorimetric or fluorimetric reagents for the determination of thorium.
2. To choose among these reagents those that are highly selective in their action towards thorium.
3. To study the most favorable reagents and to select the best for the rapid analysis of thorium.

More than 70 reagents, mostly organic dyes of known composition, have been tested for their reactions with thorium and zirconium, zirconium being tested also because it is an element hard to separate from thorium. Each reagent was studied at six different levels of controlled pH. As a result, six reagents showed promise of being useful. These have been studied intensively at carefully controlled pH levels and dye concentrations. Spectrophotometric curves for the dyes alone, for the dyes plus thorium, for the dyes plus zirconium, and (for one dye) for the dye plus aluminum have been made for several reagents. So far, none of these dyes has proved completely satisfactory, and other dyes for testing are on order.

An investigation on the effect and possible interference of arsenate ion in the volumetric determination of uranium was begun by J. J. Rowe in June. The Bureau of Standards method for volumetric analysis provides for the elimination of arsenic by volatilization prior to the reduction of uranium in the Jones reductor. The objects of the study are to investigate the behavior of arsenate ion in the Jones reductor and to determine whether arsenic interferes in the volumetric determination of uranium if not removed prior to the reduction.

Jones reductors of different amalgam strengths (2, 3, and 10 percent) were prepared, and standard solutions of uranium, arsenic (as arsenate ion), and mixtures of the two, were passed through the reductors and then titrated with permanganate. This study is continuing.

Methods of analysis for aluminum and phosphorus

An investigation of the molybdenum method for the determination of micro amounts of P_2O_5 in sea water in the presence of arsenic was started by Henry Mela, Jr., in June. It is expected that samples of sea water from work of the Fish and Wildlife Service in progress in the Gulf of Mexico will be made available to the Section for uranium determinations. At the same time these samples will be analyzed for P_2O_5. Sensitive methods will have to be employed in this analysis. The colorimetric molybdenum blue method is a highly sensitive one for both phosphorus and arsenic; it is, therefore, necessary to adapt this method for the determination of P_2O_5 in the presence of arsenic, as micro amounts of arsenic might be expected in the sea water. A search of the extensive literature on this procedure is in progress, and several variations of the method are being studied and tested.

Methods of analysis for miscellaneous minerals and compounds

Work is underway by Henry Mela, Jr., and Claude Waring on the chemical-spectrographic determination of small amounts (0.001 percent and up) of individual rare earths in phosphate rocks. Consultation among the chemists and spectrographers resulted in agreement that the best approach to the determination of individual rare earths in phosphate rocks is a combined chemical concentration and spectrographic determination. The investigation is thus a cooperative one. Samples of rare earths with different concentrations in Al_2O_3 have been prepared and spectrographic working curves have been made for Ce, Gd, La, Nd, Pr, Sm, Y, Dy, Eu, Yb, and Th. Preliminary spectrographic determination of composite mixtures of these samples have shown an apparent error of probably less than 10 percent. Other composite mixtures have been prepared and spectrographic work on these is in progress. Chemical separation methods for the rare earths and thorium are now being tested. As part of this project an annotated bibliography of the analytical chemistry of the rare earths is being prepared and will be distributed to interested workers.

The new rapid method for the determination of oil content in shales and phosphates is described in "A photometric method for the estimation of the oil yield of oil shale," by Frank Cuttitta (U. S. Geol. Survey Trace Elements Investigations Rept. 152, May 1951). The abstract follows:

> A method is presented for the distillation and photometric estimation of the oil yield of oil-bearing shales. The oil shale is distilled in a closed test tube and the oil extracted with toluene. The optical density of the toluene extract is used in the estimation of oil content and is converted to percentage of oil by reference to a standard curve. This curve is obtained by relating the oil yields determined by the Fischer assay method to the optical density of the toluene extract of the oil evolved by the new procedure. The new method gives results similar to those obtained by the Fischer assay method in a much shorter time. The applicability of the new method to oil-bearing shale and phosphatic shale has been tested.

In the course of the above study an investigation of the applicability of volumetric methods for estimation of oil in shales and phosphates was made. The closed-tube distillation method proved to be remarkably efficient and it can be made the basis in conjunction with a direct volumetric measurement of the oil yield, of a simple method for the estimation of oil in a sample as small as one gram. This method yields results similar to the Fischer method but is much faster. This method can be used particularly on samples for which photometric standard curves are not available.

In the study of the determination of small amounts of lead (0.1 to 10 ppm) by microchemical methods work went forward on the isolation and determination of lead in zircon. Tests made during May showed erratic recovery of the small amounts of lead in zircon, and it is thought that contamination by airborne dust is a contributing factor to the erratic results. Efforts are being made to correct this condition.

Quality of analysis project

Out of 427 samples of Florida phosphates previously studied statistically 10 samples showed discrepancies between the radiometric and chemical results and these discrepancies persisted on re-analysis. These 10 samples were made the subject of further study to determine whether the differences were due to natural causes (lack of radioactive equilibrium) or to analytical errors. Six chemical determinations were made on each sample, three using the usual routine method and three using one of the most refined methods available. The samples were also counted radiometrically with the samples shielded for longer periods of time after special attention was paid to the preparation of the sample. The results by the two chemical methods were in complete agreement. Nine of the 10 differences between the previous chemical and radiometric measurements were resolved and were found to be due to analytical error (about half chemical, half radiometric).

High-index liquids project

Additional sets of high-index liquids were prepared and distributed, mainly in the Survey. A new series of liquids ranging in refractive index from 1.66 to 2.02 has been prepared by Robert Meyrowitz, and the stability of these solutions is being determined. All of them contain arsenic bromide, α-bromonaphthalene, precipitated sulfur, and arsenic disulfide.

Five of the seven complete sets of liquids prepared in July 1950 were restandardized. The maximum change in index of the liquids in the range 1.74 to 1.81 (methylene iodide, arsenic bromide, and sulfur) was ± 0.001. For the liquids in the range 1.82 to 2.00 (arsenic bromide, sulfur, and arsenic disulfide) the maximum change in index was - 0.004.

A liquid of refractive index 2.074 has been prepared from arsenic bromide, sulfur, and arsenic trisulfide. The stability of this liquid is being determined.

SPECTROGRAPHIC INVESTIGATIONS

Eight elements have been added to the list of elements determined in the semiquantitative method in one arcing of a 10-mg sample, thus bringing the total to 64 elements.

Work is in progress by Charles Annell and Helen Worthing on the determination of impurities in uranium as a means of handling complex refractory oxides with complex spectra. The so-called carrier distillation method has been investigated whereby gallium oxide is used to carry the impurites in uranium into the arc without exciting the uranium atoms. This study uses 100-mg samples of U_3O_8 prepared from $(UO_2)(NO_3)_2 \cdot 6H_2O$, and pitchblende to which 2 percent of Ga_2O_3 has been added. It has been found that the lines of the impurities in uranium were enhanced and those of the uranium were suppressed at 5 to 6 amperes, 260 to 270 volts, interrupted d-c arc. Moving-plate studies indicated that the above conditions lasted about two minutes before uranium entered the arc stream. A 10-second preburn is necessary as uranium oxide is swept into the stream by the ignition spark. Further work indicated that the Ga_2O_3 carrier was not essential for the suppression of the uranium lines when the arc conditions were 25 ohms, 5 to 6 amperes, 260 to 270 d-c volts. Mg and Si, as impurities, showed more sensitivity in material with the carrier added but Ca, U, and Ti showed more sensitivity without the carrier. This study is still in progress.

Work is being done by C. L. Waring, Annell, and Worthing on the detection of trace amounts of thorium in samples without prior chemical concentration, on the determination of thorium in samples high in uranium, and on the detection of trace amounts of lead. Special equipment is to be applied in the study of the determination of thorium in samples high in uranium. A silica glass atmosphere chamber has been received, and a thermometer specially designed for use with the Christiansen filter is on order. Work will be resumed in this study when the thermometer is available.

The study of the determination of trace amounts of lead is described in "The spectrographic determination of trace amounts of lead in zircon and other minerals," by C. L. Waring and Helen Worthing (in preparation). The tentative abstract follows:

> The spectrographic determination of lead in zircon and in some other minerals has been made to provide data in a study of the age of the rocks that contained these particular minerals. Approximately 100 determinations have been made, and one standard deviation test, consisting of 25 observations, has been completed. A standard deviation of 2.06 ppm lead was indicated by the tests conducted on a lead standard of 30 ppm. The method is applicable to samples containing from 0.5 to 1,000 ppm lead, with an estimated accuracy of 6 to 10 percent. The method has also been applied to samples of apatite, sphene, microlite, allanite, and perthite.

Development by Jules Stich of the so-called single-grain qualitative method continued. The procedure is being adapted from use in the 3-m Baird spectrograph for use in the Gaertner prism spectrograph. Slightly greater sensitivity, complete spectrum coverage, and better distribution of the sample load on the instruments, contributed to this decision.

The semiquantitative method has been applied to the analyses of micro samples (1 mg or less) with no changes in the excitation conditions or electrodes.

Work is in progress to explore the possibility of automatically scanning the semiquantitative plates. The wavelength positions of 62 elements have been marked on a test plate and this plate will be run through the Leeds and Northrup Microphotometer. If automatic scanning is successful it will result in a great saving of time and eye fatigue to laboratory personnel.

As part of a continuing evaluation of spectrographic results more than 150 chemical check analyses were made on samples on which routine semiquantitative spectrographic determinations were made. Of these, five disagreements were noted, three of which were borderline cases. The first chemical checks on Cb and Ta (in euxenite) showed no disagreement with the spectrographic results.

Tennyson Myers, in charge of spectrographic work in the Section's Denver Laboratory, has made a study of contamination as affecting spectrographic analysis of samples handled by ordinary grinding procedures. The results of his study show contamination by Mo, Ni, and Cu in general and by Cr, Co, and Mn, in some of the samples. A report on this study giving further details is in preparation by Myers.

RADIOMETRIC INVESTIGATIONS

In the continuing study of phosphors for use in scintillometers by Jim Bracken and William R. Champion, the behavior of anthracene in xylol as an internal phosphor was investigated. The results were unsatisfactory.

A high-gain linear amplifier ~~and~~ power supply was designed and constructed for use with an ionization chamber for the study of absolute alpha activity of low orders of magnitude. The characteristics of this apparatus are being studied.

Also under study are the merits of magnetic, electrostatic,and differential pulse height alpha analyzers with respect to possible geologic applications such as half-life determinations, abundance ratios, and U/Th ratios. Different published circuits for differential pulse analyzers are being evaluated.

A windowless Geiger-Muller tube was constructed so that a sample can be put directly into the tube, thus affording minimum absorption. The tube was also designed for minimum backscattering and nearly 2 π geometry. Experiments are in progress with different combinations of counting gases to obtain the best plateau. This tube will be used for absolute β-counting.

An investigation is in progress of the emanation properties of zircon. An attempt is being made to correlate the emanation rate with the amount of Th present and also to obtain a good approximation (in the order of $\pm$ 3 percent) of the true activity.

Construction of radon-counting equipment to be set up in the Denver Laboratory is in progress.

Control charts for the Tracerlab automatic counter are still in preparation.

Work is in progress on the development of a rapid radiochemical method for the determination of uranium and thorium in monazite by Francis J. Flanagan and Jesse Warr. This method involves the chemical separation of radium isotopes in monazite by sulfate precipitation, using barium as a carrier. The half lives of the two radium isotopes of the thorium series are short as compared to radium in the uranium series, and equilibrium with their daughter products is reached in about 25 hours as compared to 30 days for the radium in the uranium series.

The radium isotopes precipitated in the procedure are Ra^{223}, Ra^{224}, Ra^{226}, and Ra^{228}. The effect of Ra^{223} on the count may be neglected and it is assumed (this remains to be confirmed) that the radium from the U^{238} series does not contribute appreciably to the β-γ count in the time required for the operation of the method. Preliminary separations on monazites supplied by John B. Mertie, Jr., of the Survey indicate that a practical maximum (probably not absolute) in the growth curve of the isotopes from monazite occurs at about 25 hours after precipitation of the sulfates. Test runs seem to confirm this idea.

The method is based on the assumptions that (1) Ra^{226} does not yield, in the amounts found, an appreciable β-γ count in the time required, and (2) that it is possible to obtain 100-percent chemical recovery of $BaSO_4$ in the method. These assumptions are being checked.

If this method proves to be sound, its application to monazites will be most useful. Possibly uranium and thorium could be determined in about 28 hours of elapsed time, with only about three hours being spent in laboratory work on the sample; whereas, with the best present chemical methods of determining these two elements directly in monazite, about three to four days are required. Work on this project was recessed during April, May, and June because of the urgency of other work.

REPORTS

Reports written by members of the Section, or on behalf of the Section by other members of the Survey up to December 31, 1950, are listed in TEIR 148-A, pages 92 to 96.

Reports issued from January 1 - June 30, 1951, in the U. S. Geological Survey's Trace Elements Investigations Report and Trace Elements Memorandum Report series are as follows:

TEIR	Title and author	Date
130	A study of the critical factors in the "direct" fluorimetric determination of uranium, by Mary H. Fletcher.	October 1950 transmitted February 1951
142	Distribution of uranium in rich phosphate beds of the Phosphoria formation, by M. E. Thompson.	March 1951
143	A semiquantitative spectrographic method for the analysis of minerals, rocks, and ores, by C. L. Waring and C. S. Annell.	March 1951
144	Hydrothermal uranothorite in fluorite breccias from the Blue Jay mine, Jamestown, Boulder County, Colorado, by George Phair and Kiyoko Onoda.	March 1951
148-A	Summary of the research work of the Trace Elements Section, Geochemistry and Petrology Branch, for the period April 1, 1948 - December 31, 1950, by J. C. Rabbitt.	June 1951
151	Summary of the research work of the Trace Elements Section, Geochemistry and Petrology Branch, for the period January 1 - March 31, 1951, by John C. Rabbitt.	May 1951
152	A photometric method for the estimation of the oil yield of oil shale, by Frank Cuttitta.	May 1951

TEMR	Title and author	Date
124	Summary report on public samples received 1949-1950, Trace Elements Section Washington Laboratory, Geochemistry and Petrology Branch, by Maurice Deul.	June 1951
236	Preliminary notes on the minor-metal content of Florida phosphate rock, by V. E. McKelvey, J. B. Cathcart, and Helen Worthing.	January 1951
237	The stratigraphy of the upper part of the Bone Valley formation and its relation to the leached zone, by Z. S. Altschuler, Elizabeth B. Jaffe, and Edward Dwornik (Offical use only).	May 1951
251	Red and gray clay underlying ore-bearing sandstone of the Morrison formation in western Colorado, by Alice Dowse Weeks.	May 1951
252	A fluorimeter for solutions, by Mary H. Fletcher and E. Ray Warner.	April 1951

The following report was approved for publication by the Director of the Survey:

> The preparation of nuclear-track plates and stripping films for the study of radioactive minerals, by L. R. Stieff and T. W. Stern, approved June 1951 for publication in The American Mineralogist.

The following report was published:

> Studies of uranium minerals VII: Zeunerite, by Judith Weiss Frondel: Am. Mineralogist vol. 36, nos. 3 and 4, pp. 249-255, 1951.

The following reports were in process as of June 30, 1951:

1. Abstracts of the literature on synthesis of apatites and some related phosphates, by E. B. Jaffe (to be published by the Survey as U. S. Geological Survey Circular 135).

2. Hydrothermal uranothorite in fluorite breccias from the Blue Jay mine, Jamestown, Colorado, by George Phair and Kiyoko Onoda (permission being requested of the Director to submit this for publication in The American Mineralogist).

3. The quantitative determination of calcite associated with carbonate-bearing apatites, by Sol R. Silverman, Ruth K. Fuyat, and Jeanne D. Weiser (permission being requested of the Director to submit this for publication in The American Mineralogist).

4. Bibliography on metamict minerals, by T. Botinelly.

5. Some factors in the differential leaching of uranium and radium in pyritic mines and mine dumps, by George Phair.

6. Further studies of the distribution of uranium in rich phosphate beds of the Phosphoria formation, by M. E. Thompson.

7. Physical and chemical comparison of modern and fossil tooth and bone materials, by E. B. Jaffe and A. M. Sherwood.

8. USGS Bulletin -- Contributions to Geochemistry: Methods of analysis for uranium and thorium developed by the U. S. Geological Survey, edited by F. S. Grimaldi, Irving May, and Jane Titcomb.

9. Contamination of rock samples during grinding as determined spectrographically, by A. T. Myers and Paul R. Barnett.

10. The spectrographic determination of trace amounts of lead in zircon and other minerals, by C. L. Waring and Helen Worthing.

11. Absorption of uranium by pelletal phosphate, by R. S. Jones.

12. Fluorimetric determination of uranium in shales, lignites, and monazites after alkali carbonate separation, by Norma S. Guttag and F. S. Grimaldi.