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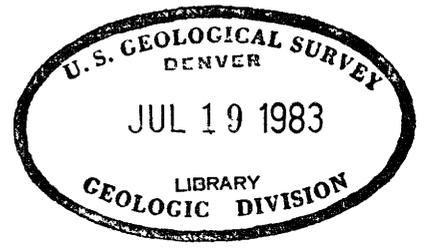
UNITED STATES DEPARTMENT OF THE INTERIOR
GEOLOGICAL SURVEY

PROGRESS REPORT ON SAMPLING OF LEACHED ZONE MATERIALS
OF FLORIDA FOR MINERALOGIC AND METALLURGICAL STUDY*

By

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August 1952



Trace Elements Memorandum Report 478

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PROGRESS REPORT ON SAMPLING OF LEACHED ZONE MATERIALS
OF FLORIDA FOR MINERALOGIC AND METALLURGICAL STUDY

By Z. S. Altschuler

ABSTRACT

With the recently heightened interest in the exploitation of leached zone rock of Florida for uranium production, laboratory work on southeast phosphates will become increasingly devoted to problems of mineral variation and uranium concentration in this rock. Efforts are now underway to establish a cooperative investigation of mineralogy.

Owing to the fact that lithologic zones of secondary alteration vary greatly in composition, it is necessary to establish a pattern of sampling control for the programs of metallurgical and chemical investigation of leached zone rock that are now underway. It is suggested that the most practical approach to this sampling problem would be to study in detail the mineralogy and extraction characteristics of end-member types of ore to the extent that these can be defined. Studies are now in progress of samples of pseudowavellite-rich rock. Magnetic, size, and gravity separations have been made, and chemical leaching experiments are now in process.

The first three weeks of May 1952 were spent in field work by Z. S. Altschuler and Robert Berman in the Florida phosphate field. During this period conferences were held with Messrs. Gillingham, Hosted, Ruch, and Wimpfen of the Atomic Energy Commission and Cathcart and McKelvey of the Geological Survey. In view of the current interest in process development for extraction of uranium from leached-zone rock, it was arranged at that time to plan a cooperative program of investigation with the group working under the direction of Keith Brown at Oak Ridge National Laboratory.

Oak Ridge National Laboratory, TVA Laboratory at Muscle Shoals, and several other research establishments are now engaged in chemical studies on leached-zone rock for the purpose of testing and designing uranium-extraction processes. Whereas it is possible to concentrate uranium after

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total solution of quartz-free leached-zone rock, there are many commercial factors which make desirable the search for a method of selective or preferred extraction of uranium. This is particularly true because there are geologic-chemical indications that selective extraction may be possible. Some of these are:

1. The calcium uranyl phosphate, autunite, has been found in one locality.
2. The uranium present in the leached zone has been secondarily redistributed during weathering of the upper Bone Valley formation. In many other deposits the weathering of primary uranium minerals or sources results in a profuse development of secondary uranium minerals in the form of common salts, sulfates, carbonates, and prominent phosphates.
3. Complexes of the type UO_4HPO_4 or $(UO_4)_3(PO_4)_2$ could be stable under the conditions of formation of wavellite (personal communication, F. S. Grimaldi). In fact, aluminum phosphate is used as a coprecipitant of uranium from nitrate solutions.

Thus, there are reasons to believe that uranium may be present in the leached zone as absorbed complexes or intimately dispersed secondary phases, too small and too sparse quantitatively to be detected except rarely. It is equally possible, of course, that such secondary phases, if present, are effectively trapped in the fine structure of the host minerals. At any rate, experiments in selective extraction should be exploited.

The chemical laboratories engaged in such work are faced with two problems:

- 1) The leached zone varies greatly in its lithology and hence it is almost impossible to define a typical sample or range of composition.

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- 2) Detailed mineralogic and petrographic study should be made of the leached zone in conjunction with chemical experimentation.

Until recently, the groups at Oak Ridge and TVA have made studies of randomly selected samples which have been sent to them from various sources, including the Geological Survey. These samples have all been good samples; however, they are random in the sense that no pattern has been established to govern the selection of materials and hence the significance of conflicting, or merely different, results could not be evaluated.

Three approaches may be thought of in designing a program of metallurgical testing.

1. Large areas of leached zone may be sampled statistically on the basis of a geographic net. Such a program would take too long to fulfill.
2. Continue random selection of "hot" and obviously different materials. This would require a large number of samples and experiments before the patterns or averages would emerge.
3. Define as nearly as possible polar or end-member types and study these in detail, and supplement this with a smaller selection of random samples, or intermediate types, than would be necessary under either of the other two approaches.

This problem was discussed with Keith Brown and his associates during a visit to Oak Ridge National Laboratory and the third of the above approaches has been agreed on. Thus the Geological Survey has undertaken to furnish to Oak Ridge samples of pseudowavellite-rich material, and wavellite-rich material. These samples will be studied in detail petrographically and will be separated and tested for uranium on the basis of various physical properties.

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In addition to the above we shall furnish petrographic descriptions of materials for which Oak Ridge has process-development data.

It is recognized that this program is an over-simplified one and will have to be supplemented by study of additional types of samples. Thus it will be necessary to study ores containing pseudowavellite and kaolinite to ascertain whether the presence of kaolinite, which itself contains practically no uranium, can alter the results of process studies made on ore consisting almost entirely of pseudowavellite.

The following work is now in progress in fulfilling the above program:

1. Study of the petrography and uranium distribution of a sample of pseudowavellite ore, a two-pound split of which has been sent to Oak Ridge for process studies.
2. Mineralogic study of a suite of samples, sent to us by Dr. Coleman of Oak Ridge, and of interest because leaching studies have already been made of them.

The sample of pseudowavellite ore mentioned under (1) above came from the Homeland mines of the Virginia-Carolina Chemical Corporation. The particular quarry, which was opened in the Spring of 1952, is situated at approximately the NE Cor. SW $\frac{1}{4}$ Sec. 33, T. 30 S., R. 25 E. The material is a light-yellow coarsely porous quartz sand with about 25 percent of fine-grained clay-like phosphatic matrix. The material was dried at 100 C., stage-ground to three fractions, plus 120 mesh, 120 mesh to 200 mesh, and minus 200 mesh. Of these, the plus 120 mesh material contained most of the quartz, the 120 to 200 mesh material had roughly equal amounts of quartz and phosphate and the minus 200 mesh material had only minor quartz. There were no compositional differences among the phosphatic materials of the different size fractions.

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The sample consists, in addition to quartz, of pseudowavellite, apatite, and minor amounts of an unknown material represented by the following spacings in X-ray patterns (all measured in degrees 2θ for copper radiation):

--- 18.5° , 18.8° , 22.8° , 26.1° , and 29.1° . Although the sample was further separated magnetically, by heavy liquids, and by sieving at 400 mesh, none of the resultant fractions showed a concentration of the unknown material.

The phosphatic material is light brown in color (microscopically) and has a mean index of $1.620 \pm .003$. Disseminated in it are fiber-like areas of birefringent material. Various gradations of color and birefringence can be seen in all fractions. However, discrete mineral types are not evident. From these facts and the lack of different X-ray results among different size, magnetic, and gravity separates, it is believed that the pseudowavellite occurs as a replacement of apatite and that the unknown material also occurs in the same manner -- as an intimately dispersed secondary material. On the basis of the relative intensities of the "extra" lines in the X-ray patterns, it is assumed that both gibbsite ($Al_2O_3 \cdot 3H_2O$) and phosphosiderite ($Fe, Al PO_4 \cdot 2H_2O$) are present. These identifications are, however, far from conclusive.

Minor amounts of kaolinite and trace amounts of goethite, zircon, rutile, sillimanite, tourmaline, ilmenite, and kyanite are also present in this sample.

The phosphatic material is soft, highly porous, and most of it has a specific gravity in the range 2.81 to 2.83. The extremes in specific gravity are 2.76 and 2.86.

Material in the 120 and 200 mesh fraction was separated magnetically by use of the Frantz Isodynamic separator and an induced roll magnetic separator.

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It was found that the phosphates could be fractionated at magnetic intensities usually used for work on garnet and monazite and some material could be separated out at intensities low enough to segregate ilmenite. The following fractions were obtained:

<u>Fraction and Setting</u>	<u>Weight (Gms.)</u>	<u>% Eq. U</u>
least magnetic > 0.9 Amp. (Frantz)	50	0.033
0.25 Amp. (roll) to 0.9 Amp. (Frantz)	100	0.032
0.1 Amp. (roll) to 0.25 Amp. (roll)	350	0.043
0.1 Amp. (Frantz) to 0.1 Amp. (roll)	75	0.047
most magnetic < 0.1 Amp. (Frantz)	30	0.049
original sample		0.041

A significant concentration of uranium occurs in the more magnetic fractions. Under the petrographic microscope the more magnetic material is seen to contain a concentration of secondary minerals with slightly higher birefringence than the rest of the rock. However, X-ray study does not reveal any difference mineralogically among these splits. Spectrochemical analysis reveals the presence of iron in the order of 1-10 percent but does not show significant difference in iron content among the various magnetic fractions, probably due to the insensitivity of this type of analysis to small differences.

Chemical analyses are now in progress for determinations of Fe_2O_3 , Al_2O_3 , P_2O_5 , U, and CaO on the whole sample and for U and Fe_2O_3 on various fractions.

After consultation with F. S. Grimaldi, a program of leaching studies on this same pseudowavellite ore has been started. This will involve leaching of samples with two series of acids - one of these a series of complexing acids representing a range of pH values, the other a series of noncomplexing acids of the same pH values on the first series.

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During the past quarter and for the next quarter, work on phosphates has been confined mainly to the above-described study of leached-zone material, and to mineralogic work on deep-well samples and ore samples submitted by Dr. Coleman of Oak Ridge. The last two have not been reviewed in this report but will be forwarded in other reports.

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