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A STUDY OF SOME METHODS OF CONCENTRATING URANIUM AND RADIUM  
IN CARNOTITE ORE.

by  
HAROLD L. GIBBS

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A  
THESIS

submitted to the faculty of the  
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI  
in partial fulfillment of the work required for the  
Degree of  
MASTER OF SCIENCE IN CHEMICAL ENGINEERING

Rolla, Missouri

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Approved by

W.T. Schrenk

TABLE OF CONTENTS

Acknowledgments . . . . .	1
Object . . . . .	2
Introduction . . . . .	2
Description of the Ore . . . . .	3
Examination of the Ore . . . . .	3
Methods of Analysis . . . . .	7
Flocculation Tests . . . . .	9
Flotation of Carnotite . . . . .	11
Results of Flotation Tests . . . . .	15
Recovery of Radium from Leached Ore by	
Flotation . . . . .	18
Summary . . . . .	18
Bibliography . . . . .	19

LIST OF ILLUSTRATIONS

Wet Screen Analysis . . . . . 4

Selective Grinding Analysis

    for Low Grade Ore . . . . . 5

    for High Grade Ore . . . . . 5

Float and Sink Analysis . . . . . 6

Flocculation Tests . . . . . 10

The Relation of pH to the Flotation

    of Carnotite . . . . . 12

The Effect of Ammonium Chloride on the

    Flotation of Carnotite . . . . . 13

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## OBJECT

It was sought in this work to study the possible methods of concentrating carnotite ore. The valuable metals to be recovered are radium, uranium, and vanadium.

## INTRODUCTION

Carnotite ore is a source of radium and it is the recovery of this element which is chiefly sought in its treatment. The radium results from the radioactive disintegration of uranium which is one of the constituents of the yellow mineral carnotite. The ores are largely low grade. Higher grade pockets occur scattered in the deposits and because of their irregular occurrence are not easily located. The larger low grade deposits which are more easily accessible contain considerable quantities of calcium carbonate and oxide minerals. These cause a high consumption of the acid used in the chemical treatment of the ore. The complexity of the treatment, the high acid loss, the small quantity of uranium and vanadium, and the extremely minute quantity of radium are factors which militate against a cheap and successful extraction. With these factors in mind the general characteristics of the ore were studied.

## DESCRIPTION OF THE ORE

Carnotite ore, as found in Colorado and Utah, may be classed as a fine sandstone in which the large visible grains are quartz and feldspar cemented together with carnotite, clay, and calcium carbonate. The cementing is not tight, and hence, the large lumps may be easily crushed. Crystalline carnotite found in Utah was studied and some of its properties reported by Hess and Foshag. (F. L. Hess and Wm. F. Foshag, Proc. U. S. Nat. Museum 72; Art. 12, 1-6, 1927). Its chemical composition varies, but it agrees roughly with the formula  $K_2O \cdot 2UO_3 \cdot V_2O_5 \cdot (H_2O)_n$ . This corresponds to potassium uranyl vanadate. It has a canary yellow color.

The ores considered in this paper contained considerable quantities of iron and copper. A large number of other metals are reported by other investigators.

## EXAMINATION OF THE ORE

The ore was examined in order to determine in what state the carnotite was present. Microscopically some of the quartz and feldspar grains were seen to be coated with the yellow carnotite. Water washing as in the case of a wet screen analysis fails to remove all of this coating. Dry uranium oxide clings tenaciously to glass and a corresponding condition may exist with respect to the carnotite and quartz.

The microscope indicated that the fine cementing material was unevenly colored with yellow carnotite. However, carnotite could not be differentiated from the clayey material. Hence, two problems must be solved, first, the removal of the carnotite clay mixture coating the quartz and feldspar grains, second, the separation of the carnotite from its intimate association with the clay.

The distribution of the carnotite was studied by means of a wet sizing analysis. There is a concentration of the carnotite in the finest material. The intermediate sizes, which represent the grains that have been rubbed clean are the leanest as would be expected. The fact that there is not a very high concentration of carnotite in the fine material would indicate the presence of a gangue material of the same size as the carnotite. The results are shown in Table 1.



Table Number 1.

WET SCREEN ANALYSIS OF LOW GRADE CARNOTITE

Product	Weight	% Weight	Assay %U <sub>3</sub> O <sub>8</sub>	Total %U <sub>3</sub> O <sub>8</sub>
-10, plus 14 mesh	21.6 g.	2.86	1.05	2.66
-14, plus 20 mesh	51.9	6.88	1.30	7.92
-20, plus 28 mesh	27.6	4.98	1.19	5.23
-28, plus 35 mesh	40.2	5.32	1.10	5.17
-35, plus 48 mesh	108.4	14.3	0.80	10.17
-48, plus 65 mesh	122.5	16.2	0.77	11.07
-65, plus 100 mesh	132.0	17.5	0.68	10.54
-100, plus 150 mesh	75.0	9.93	0.93	8.18
-150, plus 200 mesh	41.0	5.43	1.12	5.38
-200 mesh	124.9	16.53	2.30	33.60
Composite	755.1 g.	100.00	*1.13	100.00

\*Calculated.

Heads = 1.33% U<sub>3</sub>O<sub>8</sub>

Selective grinding was tried as a means of mechanically rubbing the quartz grains free of carnotite.

The low grade ore contained two per cent uranium oxide and a high percentage of sand and hence, should be most highly beneficiated by this type of treatment. After twenty minutes of wet grinding with rubber rods, eighty-three per cent of the carnotite was in the fines. After forty minutes, ninety-one per cent of the carnotite was in the fines and sixty-eight per cent of the original weight was sand containing only two tenths of one per cent of uranium oxide. The result of these experiments for a low grade ore are tabulated in Table 2, and for a high grade ore in Table 3.

Table 2

SELECTIVE GRINDING ON RUBBER COVERED ROLLS  
OF LOW GRADE CARNOTITE

Product	Weight	% Weight	%U <sub>3</sub> O <sub>8</sub> Assay	%U <sub>3</sub> O <sub>8</sub> Total
-325 mesh slimes				
Without Grinding	118.7 g	11.9	4.98	40.3
-325 mesh slimes				
With Initial 10 min. Grind	116.2	11.7	4.19	33.4
-325 mesh slimes				
With second 10 min. Grind	42.2	4.2	3.38	9.7
-325 mesh slimes				
With added 20 min. Grind	38.7	3.9	2.76	7.3
plus 325 mesh coarse				
Residue 40 min. Grind	682.0 g	68.3	0.20	9.3
Composite	997.8	100.0	*1.47	100.0

\*Calculated

Heads = 1.98% U<sub>3</sub>O<sub>8</sub>.

Table 3.

SELECTIVE GRINDING ON RUBBER COVERED RODS OF  
HIGH GRADE CARNOTITE

Product	Weight	%Weight	%U <sub>3</sub> O <sub>8</sub> Assay	%U <sub>3</sub> O <sub>8</sub> Total
-325 mesh slimes	209 g.	41.8	13.63	56.90
Before Grinding				
-325 mesh slimes	176	35.2	12.50	39.31
After 20 min. Grind				
plus 325 mesh coarse Residue	115	23.0	4.76	9.78
Composite	500	100.0	*11.19	100.00

\*Calculated

Heads = 16.55% U<sub>3</sub>O<sub>8</sub>.

Concentration in the past has been based on the fact that the carnotite can be collected in the fine material. The above experiments confirm this. Where water has been plentiful, the carnotite has been slimed away from the coarse crystalline material. In dry regions dusting has been used.

However, this does not effect a high grade concentrate due to the presence of large quantities of fine gangue material. The elimination of the fine calcareous material is more essential than the removal of the quartz and feldspar. This method has no practical application in the case of the high grade ore studied here, because the coarser sand is present in such small quantities that its removal does not result in an appreciable concentration.

Float and sink tests were made to ascertain whether or not a separation could be made on the basis of specific gravity. Acetylene tetrabromide and benzene were mixed to give a heavy liquid of 2.70 specific gravity. A minus 65 to plus 200 mesh sample of low grade ore analyzing 0.64% uranium oxide was treated with this liquid. The sink product contained 6.8% uranium oxide. The float assayed 0.38% uranium oxide. However, the recovery represented by the sink product was only 31%. This indicates the high specific gravity of the carnotite but it also indicates that the carnotite is intimately associated with gangue material. The results are given in Table 4.

Table 4.

FLOAT AND SINK ANALYSIS OF minus 65, plus 150 MESH

LOW GRADE CARNOTITE

Heavy Liquid Specific Gravity 2.70

Acetylene Tetrabromide and Benzene.

Product	Weight	% Weight	%U <sub>3</sub> O <sub>8</sub> Assay	%U <sub>3</sub> O <sub>8</sub> Total
Sink in 2.70	.216 g.	2.46	6.80	31.04
Float on 2.70	8.58	97.54	0.38	68.96
Composite		100.00	*0.54	100.00

\*Calculated

Heads = 0.64% U<sub>3</sub>O<sub>8</sub>

In order to gain a further insight into the size of the carnotite particles, flocculation tests were made. The high grade ore was used and a suspension was made in distilled water. The uranium content of that material which did not form a suspension, of that which settled almost immediately, and of that which remained in suspension for ten minutes did not vary from the original material more than 2%. The uranium oxide content of this ore was 16%. This indicates that a separation of carnotite from such an ore involves the separation of particles of colloidal size.

#### METHODS OF ANALYSIS

In order to study the concentration of this ore some methods of analysis must be employed. Large differences in values could be roughly recognized by means of the peculiar yellow color of the carnotite. This method is used in hand sorting at the mine. It has been found that this sorting must be done in natural light as artificial light gives very unreliable results. Where the change in concentration is comparatively small such a method fails entirely.

There are two other possible methods, one chemical analysis and the other radioactive determination. Uranium and vanadium form a definite compound in the ore and hence have a definite relation to each other. Also, the radium and its emanation bear a definite relation to the uranium content.

Hence determination of the radium, the uranium, or the vanadium would determine the concentration of the carnotite. Throughout this work all concentrations are expressed in terms of the per cent  $U_3O_8$ , uranium oxide. This is usually the case in such determinations as the ignition of the final precipitate of ammonium uranate in the chemical analysis results in a residue of  $U_3O_8$ . Pure carnotite is approximately 53%  $U_3O_8$ , depending on the amount of water of crystallization. Hence the per cent of carnotite is roughly twice that of the given assay per cent of  $U_3O_8$ .

In the chemical method, the determination of uranium was chosen as being the simplest. A modified Scholl's method of analysis recommended in the Bureau of Mines Bulletin 212 was used. Low results were obtained in ores containing less than three to four per cent of  $U_3O_8$ . The large iron hydroxide precipitates and the difficulty of keeping the solution free of carbon dioxide in the sodium uranate precipitation caused the chief trouble. The concentration of carbon dioxide in the burnt gases from a flame are ample to completely prevent the precipitation of sodium uranate in low grade ores.

The second method, that of radioactive determination by means of a gold leaf electroscope was used to determine nearly all the data here presented. This method is much quicker and is accurate provided the ore is not leached or unduly heated. The radioactivity as measured is due largely to the short-lived radioactive gas, radium emanation, which is occluded by the



solid. Solution or heating will cause the loss of this gas. However, the drying of concentrates on a sand bath did not materially affect their radioactivity. In case of solution and reprecipitation, the recovery of the radioactivity with time can be calculated by the use of the Kolowrat tables or formula. (1e radium, Vol. 6, 195, 1910.)

$$\% \text{ regenerated} = 1 - e^{-Kt}$$

where K = regeneration constant

t = time in hours

In all the cases where the ore came in contact with water a loss of some of the radioactive material occurred. Such losses were thought to be due to a leaching of a small amount of the radium content.

#### FLOCCULATION TESTS

As the ore formed a partial stable suspension in water, it was thought that flocculation tests might lead to some results useable directly or in connection with flotation. These tests were made on ten grams of finely ground ore suspended in twenty cubic centimeters of water. The ore in distilled water represents a condition of nearly maximum stability of suspension. In particular, the bases, sodium hydroxide, sodium carbonate, and ammonium hydroxide were found to leave the stability the same or increase it slightly in very low

concentrations. On further additions of these bases and on the initial addition of all the other salts used a decrease in stability was produced. Sodium and ammonium nitrates were much more active flocculating agents than the corresponding hydroxides. This shows the effect of change in hydroxide ion concentration. The case of sulfuric acid is interesting in that at first an increase of amount added decreased the stability whereas further addition increased the stability. Saturating the solution with sodium oxalate caused a black mineral present to remain in suspension. However, testing of the settled and the suspended material showed that the radium content of each was the same as that of the original ore. The results of these tests are given in table 5.

Table Number 5.

FLOCCULATION TESTS ON 10 GRAMS OF FINELY

DIVIDED GARNOTITE ORE

Salt Added	Molal Conc.	Time of Flocculation and Remarks
None		2 Days - Incomplete precipitate.
NaOH	.0025	2 Days - Slightly more stable than #1.
"	.0250	3 Minutes - Black settled.
"	.125	1 Minute - Clear liquid.
Na <sub>2</sub> CO <sub>3</sub>	.00128	2 Days - Same as #1.
"	.0128	2 Hours - Black flocculated.
"	.125	25 Minutes - " "
"	.50	10 Minutes - " "
NH <sub>4</sub> OH	.0019	2 Days - Same as #1.
"	.0038	2 Days - Slightly less stable than #1.
"	.025	2 Hours - Slight settling.
NH <sub>4</sub> NO <sub>3</sub>	.00125	10 Minutes - Complete flocculation.
"	.00625	7 Minutes - " "
NaNO <sub>3</sub>	.00042	1 Day - " "
H <sub>2</sub> SO <sub>4</sub>	.00125	10 Minutes - Nearly complete.
"	.00312	5 Minutes - Complete flocculation.
"	.0125	4 Minutes - " "
"	.0625	14 Minutes - Partial brown suspension.
"	.1000	12 Minutes - Brown suspension marked.

Table Number 5 (Cont.)

Salt Added	Molal Conc.	Time of Flocculation and Remarks
$\text{Na}_2\text{C}_2\text{O}_4$	.012	1 Day - Complete separation, yellow-black layer covered by black layer.
$\text{KNO}_3$	.0025	3 Hours - Black separation.
"	.0116	$7\frac{1}{2}$ Minutes - Complete.
$\text{ZnNO}_3$	.00153	7 Minutes - "
"	.00306	4 Minutes - "
$\text{SbCl}_3$	.00082	10 Minutes - Complete, milky.
"	.0041	7 Minutes - Complete.
"	.0123	4 Minutes - Complete.

## FLOTATION OF CARNOTITE

Flotation tests were made on two carnotite ores. The low grade ore in which there was considerable quartz and feldspar assayed 1.98%  $U_3O_8$ . The high grade ore which was almost entirely fine material assayed 16.55%  $U_3O_8$ .

In these tests the procedure was as follows: 500 grams of low grade ore were ground in a pebble mill for ten minutes. The slimes were then removed and the coarse material ground for an additional twenty minutes. In the case of the high grade ore, 100 grams were used and the grinding time was reduced to ten minutes. The flotation tests were made in a 500 gram mechanically agitated flotation machine.

Whereas the ore is highly oxidized and as such should be amenable to flotation by fatty acids, nevertheless it was thought that it would be wise to try sulphidizing the mineral and to use customary sulphide flotation reagents such as xanthate. However, no material in the ore was floated either with potassium ethyl xanthate alone or with the xanthate after the ore had had a preliminary treatment with sodium sulfide.

It was found that oleic acid would produce a slight enrichment of either ore when floated in distilled water. The two per cent ore in one test yielded a concentrate assaying 3.68 per cent  $U_3O_8$ .

The effect of acidity on flotation with oleic acid was studied. Sulfuric acid was used to increase the hydrogen

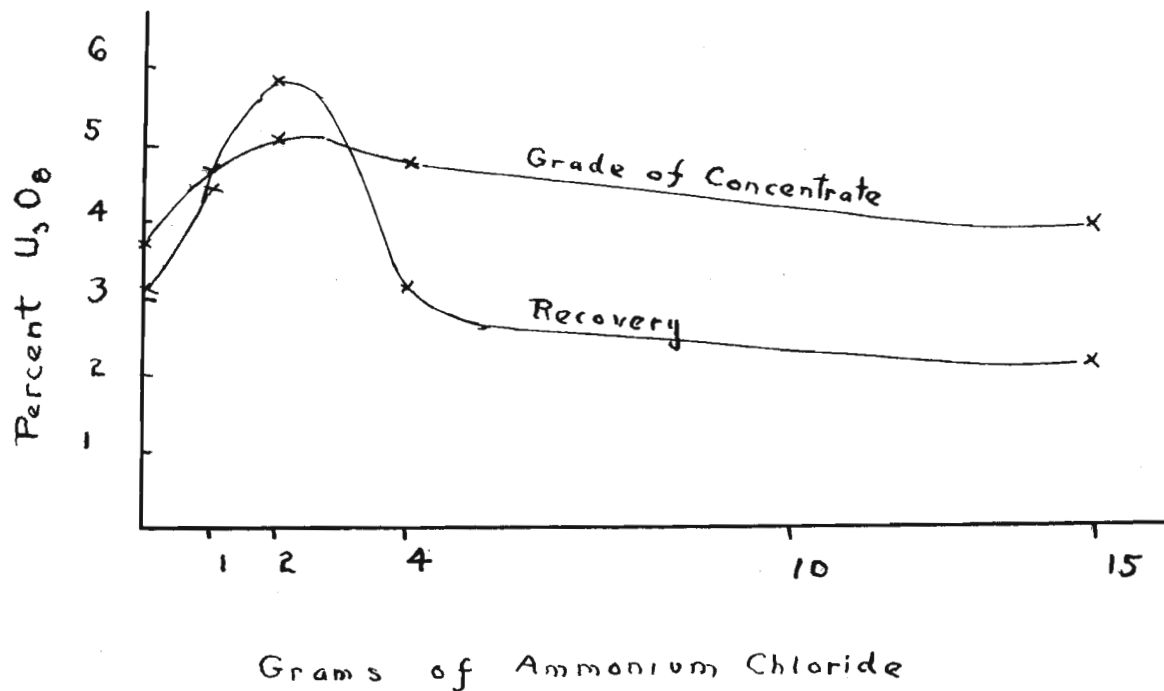
ion concentration and sodium carbonate was used to increase the hydroxide ion concentration. Additions of sodium carbonate caused an increase in the total amount of material floated and also an increase in the amount of carnotite recovered. However, the grade of the concentrate was lowered. This increase in recovery is probably due to an increase in the ionization of the sodium oleate. The results of these tests, which range from a pH of 6.2 to a pH of 9.6 are given in Table 6, and the results are plotted in the accompanying graph.

The ore has a strong buffering action either for acids or for alkalis. In order to obtain a pH of 10.6 or 10.8 it was necessary to use sodium hydroxide in place of sodium carbonate. The concentrates resulting from the use of sodium hydroxide are of higher grade than those of sodium carbonate at the same pH. The carbonate ion prevents precipitation of sodium uranate during analysis. The lowering of the grade of the concentrate in flotation caused by sodium carbonate may be caused by the same reaction.

In an attempt to eliminate possible interfering ions, ammonium chloride was added to form a complex with any copper ions present. Additions of small amounts of ammonium chloride increased the grade of concentrates to the highest value reached on all the tests made on this ore. It is also of interest that the use of aniline hydrochloride in flotation gave practically the same grade of concentrate. The basic nitrogen in the compounds ammonium chloride and aniline hydrochloride seems to have this beneficial effect. The effect of acidity in the presence of ammonium chloride was not investigated. These interesting results are plotted in Table 7, and the grade concentrates and per cent recovery are plotted in the accompanying graph.

Table 7.

EFFECT OF AMMONIUM CHLORIDE ON FLOTATION OF CARNOTITE ORE



Flotation of 500 Grams of Ore With 8 Drops of Oleic Acid,

5 cc. 5% Na<sub>2</sub>CO<sub>3</sub> at pH of 7.8.

Test	Grams NH <sub>4</sub> Cl	% U <sub>3</sub> O <sub>8</sub> Concentrate	% Recovery of U <sub>3</sub> O <sub>8</sub>
#1	0	3.68	3.02
#22	1	4.44	4.62
#17	2	5.09	5.82
#23	4	4.79	3.12
#19	15	3.92	2.16



Sodium oxalate was added during flotation and its effect observed. The addition was found to be very detrimental and a lower grade of concentrate resulted. This effect may be similar to that of carbonate ion.

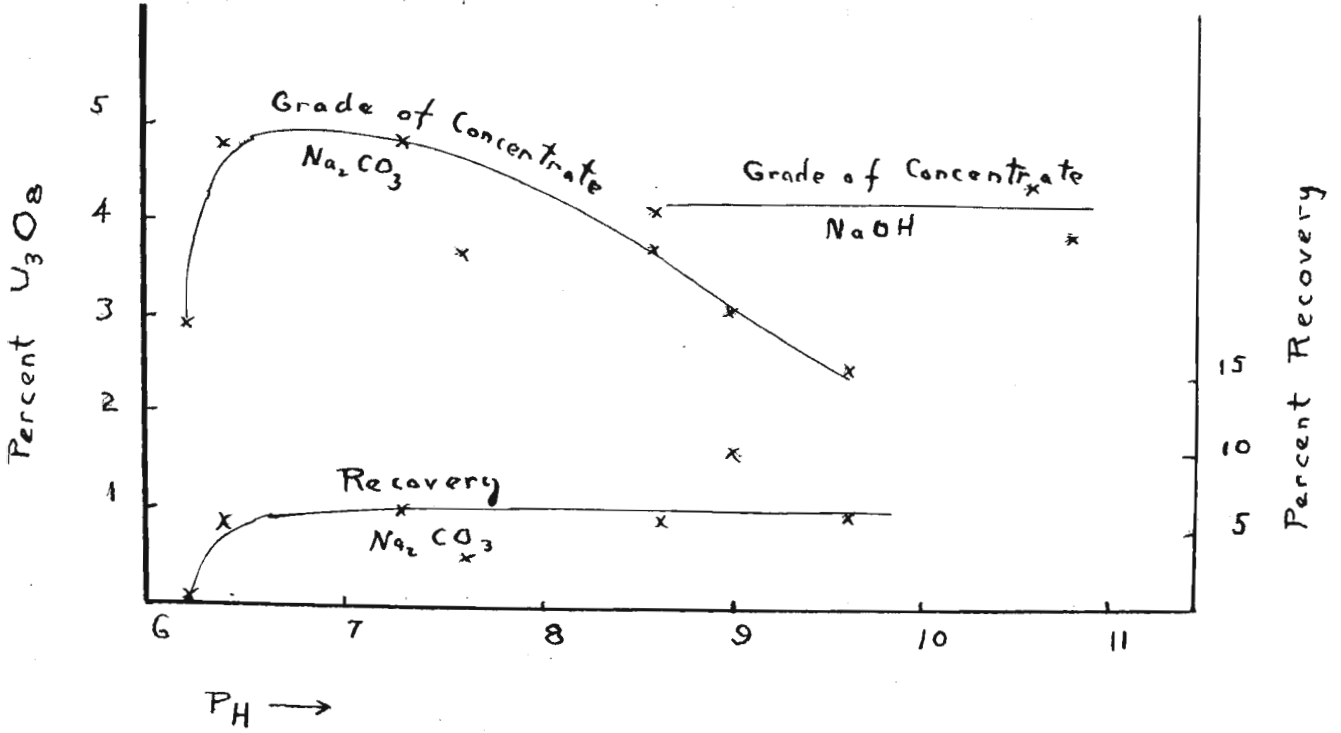
Sodium silicate is known to improve the grade of concentrate in ores containing finely divided clay gangue. This was found to be true in the case of the high grade carnotite. The sixteen per cent ore gave a concentrate of 24.6 per cent uranium oxide as compared with 21 per cent at the same pH, 7.8, without sodium silicate. The recovery of uranium was very low in this test. The effect of acidity was not studied in relation to addition of sodium silicate.

The effect of an acid having a higher dissociation constant than oleic acid was studied. It was found that decylic acid gave a low grade concentrate and the reagent consumption was very high.

The addition of potassium ferrocyanide had no effect at a pH of 9.6. The addition of sodium cyanide increased the grade of the concentrate from 2.52 to 3.22 per cent and lowered the per cent recovery. Copper is present in the ore and the additions of cyanide and ammonia were made to remove any possible copper ions. Whether this represents what actually took place is not known. In both cases the grade of concentrate was improved.

Table 6.

RELATION OF pH TO FLOTATION OF A 2% CARNOTITE ORE



FLOTATION OF 100 GRAMS OF ORE USING  
8 DROPS OF OLEIC ACID

P <sub>H</sub> =	6.2	6.4	7.3	7.6	8.6	9.0	9.6	10.6	10.8
<u>Na<sub>2</sub>CO<sub>3</sub></u>									
Grade of Conc. % U <sub>3</sub> O <sub>8</sub>	2.9	4.78	4.85	3.70	3.76	3.13	2.52		
Recovery % U <sub>3</sub> O <sub>8</sub>	0.9	5.83	7.85	3.04	6.77	10.96	7.14		
<u>NaOH</u>									
Grade of Conc. % U <sub>3</sub> O <sub>8</sub>					4.17			4.42	3.97
Recovery % U <sub>3</sub> O <sub>8</sub>					----			15.0	13.7

## RESULTS OF FLOTATION TESTS

While it was found possible to increase the per cent uranium oxide by flotation the results obtained are not practical as they now stand. This is due to the low recovery. The presence of large amounts of calcareous material and clay makes the flotation of the carnotite ore difficult. The clay and carbonates tend to segregate with the carnotite and give low grade concentrates. The dissolved salts use up large quantities of the oleic acid giving low recoveries. The effect of ammonia, sodium cyanide, and sodium silicate have not been investigated in relation to acidity nor have they been investigated in relation to their combined effect.

## RECOVERY OF RADIUM FROM LEACHED ORE BY FLOTATION

The filtration of an immense amount of fine material could be avoided if it were possible to float away from the leached material the radium content. With this in view the flotation of radium-barium sulphate was studied. Naturally occurring barium sulphate or barite floats readily with oleic acid. Fifty grams of barium sulphate precipitated from barium chloride solution with a slight excess of sodium sulphate gave complete recovery when mixed with 450 grams of fine sand.

A test on the high grade ore was made by leaching 100 grams for twelve hours with 100 cubic centimeters of 3 N. nitric acid on a sand bath. The leached solution was filtered

off to allow for analysis of the residue and thus a determination of the efficiency of the leach could be obtained. Three grams of barium chloride were added to this solution. The excess acid was neutralized just to the point of precipitation of calcium uranate. Then the radium-barium sulphate was precipitated by the addition of an equivalent amount of sodium sulphate. This was placed in a 50 gram flotation cell. On addition of oleic acid, the PH was changed sufficiently to produce the precipitation of the calcium uranate. A complete flotation of the sulfate precipitate away from the calcium uranate was accomplished.

The leach was found to be only twenty-five per cent efficient, however. In the next test stronger acid was used and the leach was boiled. The result of this stronger leach was a brown solution from which it was impossible to separate barium sulfate or natural barite by flotation. On testing this leach water, it was found to carry a large amount of ferric iron. The leach with dilute acid and low temperature did not give a test for iron.

This lead to tests of the flotation of barite in the presence of ferric iron. Flotation was found possible between a pH of 5.0 and a pH of 7.8. The flotation gradually increases from a very little at a pH of 5.0 to a maximum at a pH of 7.6. There was an abrupt stopping of flotation at pH 7.8. A colored precipitate appears at this pH in the presence of oleic acid.

A study was made of the flotation of barium sulfate in the presence of uranium. Flotation was not successful at any of the pH values investigated. As time was not taken to investigate the pH range in detail, it is possible that the optimum point was overlooked. It is interesting that calcium, which causes the uranium to precipitate as calcium uranate, lowers the concentration of that element sufficiently to allow the barite to be floated. The precipitation of sodium uranate does not suffice, probably due to the presence of small amounts of carbon dioxide.

This results in the following condition, that by leaching the ore in dilute acid and by not overheating it during the leach the iron mineral will not be dissolved and as the calcium takes care of the uranium as calcium uranate the flotation of barium sulfate is possible.

It remains to find whether the leach can be made more efficient without dissolving the iron.

The low acid leach gave tailings assaying 9%  $U_3O_8$  and a barium-radium sulfate concentrate analyzing 21.0%  $U_3O_8$ .

## SUMMARY

The tests performed indicate that ores having a high percentage of coarse sands may be readily concentrated by selective grinding. These sands can be cleaned to a point where they may be discarded with a very low loss in mineral content.

Two possible methods of concentrating the valuable minerals from the bonding material have been investigated. These methods are first, direct flotation of the carnotite away from the gangue and second, the flotation of radium-barium sulfate precipitated from the ore leach.

Direct flotation tests have yielded indications of methods which permit the preparation of maximum grade concentrates by the use of such reagents as ammonium chloride, aniline hydrochloride, and sodium silicate. The detrimental influence of such substances as sodium oxalate and carbonates has been noted. The concentrates obtained represent low recoveries and this difficulty must be overcome before this method can have any practical significance.

The recovery of radium from the ore leached with nitric acid has been encouraging and has led to a number of interesting facts such as the possibility of floating barite in the presence of uranium and iron salts. The difficulty as it now stands is to obtain an efficient leach and still retain the floatability of barium sulfate.

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**41419**

## I N D E X

Acknowledgments . . . . .	1
Analysis	
Color Separation . . . . .	7
Radioactive Determination . . . . .	8
Chemical . . . . .	8
Carnotite, crystalline composition of . . . . .	3
Description of Ore . . . . .	3
Drying of Concentrates and Radioactivity . . . . .	9
Examination of the Ore . . . . .	3
Flotation of Ore	
Ammonium Chloride, effect . . . . .	13
Table . . . . .	13
Sulphidizing Treatment . . . . .	11
Hydrogen Ion Concentration . . . . .	11
Table . . . . .	12
Aniline Hydrochloride . . . . .	13
Other Reagents . . . . .	14
Sodium Carbonate and Oxalate, effect . . . . .	14
Sodium Silicate, effect . . . . .	14
Flotation of Barite and Radium-Barium Sulfate . . . . .	15
Flotation in the presence of iron . . . . .	16
Flotation in the presence of uranium . . . . .	17
Effect of Method of Leach . . . . .	17

I N D E X

Flocculation Tests . . . . .	9
Table . . . . .	9
Introduction . . . . .	2
Microscopic Appearance of Ore . . . . .	3
Object . . . . .	2
Past Methods of Concentrating Carnotite . . . . .	6
Screen Analysis . . . . .	4
Table . . . . .	4
Selective Grinding . . . . .	5
Table . . . . .	5
Specific Gravity Separation by Heavy Liquids . . . . .	6
Table . . . . .	6
Water Loss of Radioactive Material . . . . .	9