I

RECOVERY OF URANIUM FROM CLIMAX BRANNERITE CONCENTRATE, C-7

By

D'Arcy R. George

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March 8, 1950

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Hugh Kinney /OSTI
Authorizing Official
Date: *1/16/08*

MIT Mineral Engineering Laboratory
Building 421, Watertown Arsenal
Watertown 72, Massachusetts

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MITG-231
Metallurgy-Raw Materials
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II

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III

RECOVERY OF URANIUM FROM CLIMAX BRANNERITE CONCENTRATE, C-7

By

D'Arcy R. George

A B S T R A C T

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From ninety-five to ninety-nine per cent of the uranium was extracted from brannerite concentrates by digesting the sample with concentrated sulfuric acid and then leaching with water, using one ton of acid per ton of concentrate. Precipitates containing from sixteen to forty-eight per cent U_3O_8 were obtained from the pregnant solutions. Further research on the precipitation of uranium and the recovery of wolfram is needed to determine the proper treatment.

D'Arcy R. George

Approved for A. M. Gaudin, Director

By A. W. Owens
A. W. Owens, Editor

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MASSACHUSETTS INSTITUTE OF TECHNOLOGY
DIVISION OF INDUSTRIAL COOPERATION
CAMBRIDGE 39, MASS.

MIT MINERAL ENGINEERING LABORATORY
BUILDING 421, WATERTOWN ARSENAL
WATERTOWN 72, MASSACHUSETTS

IV

DIC Project 6583 -- Gen 211

March 8, 1950

Mr. Evan F. Wilson, Assistant Manager
Raw Materials Operations
U. S. Atomic Energy Commission
P. O. Box 30, Ansonia Station
New York 23, New York

This document consists of 16 pages.
No. 10 of 27 copies. Series A.

Dear Mr. Wilson:

Attached is Report MITG-231 entitled "Recovery of Uranium from
Climax Brannerite Concentrates, C-7" by D'Arcy R. George.

The sample of brannerite concentrate which you submitted to us for
examination and testing represents a possible by-product of a by-product
obtained in the treatment of the tailing of the Climax Molybdenum Com-
pany's mill.

Although the uranium in this uranium-titanium mineral is not soluble
in acid leaching, baking of the mineral with sulfuric acid makes the
uranium water-soluble. This treatment is similar to that used commer-
cially on ilmenite and monazite.

Even though the quantity of uranium is small, the results are of
interest because they indicate a method of treating materials containing
refractory uranium minerals.

Very truly yours,

John Dasher
John Dasher
Executive Officer

JD:rgb

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TOPICAL REPORT MITG-231

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TOPICAL REPORT MITG-231

RECOVERY OF URANIUM FROM CLIMAX BRANNERITE CONCENTRATE, C-7

By

D'Arcy R. George

I. INTRODUCTION

The Climax Molybdenum Corporation at Climax, Colorado, recovers a heavy mineral concentrate, after flotation of molybdenum, by passing the sand tailings over Humphrey spirals. This concentrate is then cleaned of quartz, feldspar and light minerals by tabling, and the concentrate from this operation is floated to remove sulfides, principally pyrite. The pyrite flotation tailing is then further concentrated with magnetic separators and tables to recover wolframite, cassiterite and monazite.

In Topical Report MITG-204* the results of mineralogical studies and magnetic separation and leaching tests on Climax heavy mineral concentrate C-4, representing the end product after recovery of wolframite, was presented. This report showed that the chief source of the radioactivity was monazite but that in addition a heavy, black, highly radioactive mineral was present to the extent of approximately 0.01 to 0.05 per cent. The mineral in question was never isolated in amounts sufficient to permit definite identification, but based upon its distribution in gravity and magnetic concentration products and partial solubility in dilute acid a tentative identification of uranothorianite was given.

The MIT Mineral Engineering Laboratory was requested (Appendix A) to conduct tests to determine the ease of extracting uranium from a heavy mineral concentrate representing the residue obtained from the gravity and magnetic concentration of monazite at Climax. It was stated that the concentrate had a radioactivity equivalent to 2.80 per cent U_3O_8 and that the chief source of the radioactivity was the mineral, brannerite. The potential production of brannerite (Appendix B) appears to be approximately 300 to 350 pounds per day. No data are available on the brannerite content of the heavy mineral concentrates prior to separation of the wolframite, cassiterite and monazite, but it is probable that recovery of brannerite in a concentrate comparable in U_3O_8 content to the sample worked on at MIT is low.

* George, D'Arcy R., "Mineralogy, Magnetic Fractionation and Leaching of Heavy-Mineral Concentrates from Climax Molybdenum Corporation", Topical Report MITG-204, October 29, 1948.

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II. SUMMARY AND CONCLUSIONS

A sample of brannerite (essentially titanium-uranium oxide) concentrate (C-7) from Climax, Colorado, containing 2.41 per cent U_3O_8 was submitted with the request that tests be conducted to determine the ease with which the uranium could be extracted. By fuming the sample with sulfuric acid in the ratio of one part of acid per part of ore and then leaching the sulfated residue with water, extractions of 95 to 99 per cent of the uranium were obtained. Exploratory precipitation tests using MgO or Na_2CO_3 and MgO yielded precipitates containing from 16 to 48 per cent U_3O_8 . The consumption of reagents was very high and further research is required to develop an economical precipitation procedure.

On the basis of the limited amount of test work the following conclusions have been drawn:

1. The amount of acid used was probably in excess of that required and on a larger scale it is believed that acid requirements of 1000 to 1500 pounds per ton will be adequate.
2. Use of less acid and strong fuming of the sulfated ore, before water leaching, will reduce the amount of alkali required for neutralization of the pregnant solutions.
3. The grade of precipitate obtainable can probably be improved by a selective precipitation or retreatment process. This Laboratory has sufficient data on such processes so that a prolonged investigation would not be required.
4. The recovery of wolfram from the concentrates has not been investigated but since at current prices the concentrates contain some \$300 to \$400 of WO_3 per ton, the economic recovery of uranium would depend upon recovery of a substantial part of the wolfram (tungsten).

III. DESCRIPTION OF SAMPLE

The sample of heavy-mineral concentrates from Climax has been designated C-7. The material received weighed approximately 180 grams and consisted largely of minus 100-mesh ilmenite, wolframite, cassiterite, monazite, tourmaline, pyrite, rutile, and an estimated 5 per cent of brannerite. The U_3O_8 content as determined by various procedures was found to be as follows:

Table 1. U_3O_8 Content of Sample C-7

Radioassay	2.45% (equiv. pitchblende std)
Fluorimetric	2.31%
Microvolumetric	2.41%

The value of 2.41 per cent U_3O_8 had been accepted for the purposes of checking and calculating material balances during subsequent test work presented in this report. A qualitative spectrographic analysis showed that the sample also contained more than 10 per cent wolfram and 0.1 to 1.0 per cent tin besides large amounts of titanium, iron, silicon and aluminum.

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IV. DESCRIPTION OF BRANNERITE

Brannerite is a very rare oxide of titanium, uranium and iron containing small amounts of calcium, the rare earths, lead and thorium. ^{1/2} The mineral has been described officially only from Kelley Gulch, Custer County, Idaho, where it was found as small detrital crystals in a placer.

The amount of C-7 available was not adequate to permit the separation of a sufficient quantity of pure brannerite for chemical analysis. A spectrographic analysis of a few milligrams of hand-picked grains showed that the essential constituents are titanium, uranium and iron. The following analyses are taken from the literature and from the author's files:

Table 2. Analyses of Brannerite From Idaho and Spain

	1. <u>Fuenteovejuna, Spain</u>	2. <u>Kelley Gulch, Idaho *</u>
SiO ₂	0.16	0.6
FeO	2.25	2.9
Al ₂ O ₃	nil	--
TiO ₂	32.45	39.0
ZrO ₂	nil	0.2
CaO	2.74	2.9
ThO ₂	1.20	4.1
U ₃ O ₈	51.76	42.4
Rare Earths	1.10	3.9
PbO	2.34	0.2
H ₂ O at 110°C	<u>2.35</u>	<u>2.0</u>
Total	96.35	98.2
Sp. Gr.		4.5 to 5.43

The brannerite in C-7 varies in color from jet black to dull green and brownish green. The lighter colored varieties are the result of hydration and oxidation which condition probably increases its acid solubility and accounts for the extraction of a minor portion of the uranium during cold, dilute-acid leaching.

*J.D. Dana, et al, "System of Mineralogy," John Wiley & Sons, New York, 7th Ed. p. 774, 1944.

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V. URANIUM EXTRACTION

Because of the small amount of material available for test work and in view of the poor uranium extraction obtained by a cold, dilute acid- MnO_2 leach of the earlier sample from Climax it was thought that a high extraction of uranium could probably best be obtained by digesting the ore with concentrated sulfuric acid in the manner used commercially to open ilmenite and monazite concentrates. A few cold or dilute acid leaching tests were made for purposes of comparison.

Approximately 125 grams of the sample was ground with an automatic mortar and pestle to a fineness of 100 per cent minus 200 mesh and this material was used during the subsequent test work. The decomposition of the ore was made in small porcelain evaporating dishes on charges weighing from 5 to 25 grams using acid containing 1.0 gram H_2SO_4 /ml. The mixtures of ore and acid were heated on an electric hot plate for one to three hours at a temperature sufficient to cause continuous slow evolution of SO_3 . This treatment resulted in the formation of a hard, dry, but occasionally slightly pasty, sulfated mass which was diluted with water and boiled for 15 minutes to 2.5 hours. The residues were filtered without the aid of suction, washed, dried, ignited, weighed and analyzed for uranium. The combined strong filtrates and washings were made up to a standard volume and likewise analyzed for uranium. Details of the test results and procedures are shown in Table 3.

Examination of the data in Table 3 shows that by digesting the concentrates for two or three hours with one ton of sulfuric acid per ton of ore followed by lixiviation of the sulfated mass with water, an extraction of 95 to 99 per cent of the uranium was obtained. The tests show further that hot leaching with the same amount of sulfuric or nitric acids in the presence of water in the ratio of 2.5 to 3 parts of water per part of concentrate does not yield uranium extractions in excess of about 50 per cent and that prolonged leaching at room temperature with 500 lb of H_2SO_4 and 100 lb of MnO_2 per ton of concentrate is totally inadequate.

Only one acid digestion test (A-7) was made with less than one ton of acid per ton of concentrate but because of differences in procedure and analytical errors the results are of doubtful value. The results obtained gave an indicated extraction of 89 per cent after baking the ore in a muffle furnace at 275-300°C for 15 minutes using 0.5 ton of acid per ton of ore. In view of this it is believed that extractions in excess of 95 per cent can probably be obtained on a larger scale using considerably less than one ton of acid per ton of ore. This would be particularly desirable not only because of the cost of the acid but because of the necessity for destroying excess acid during subsequent neutralization of the pregnant solution. The excess acid, however, could also be eliminated by fuming the sulfated mass strongly before taking up with water.

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Table 3. Details of Acid Treatment of C-7 Ore

Test No.	Sample Wt, g	Reagents Lb/Ton	Residue		Preg. Soln		Calc.	Ext	Remarks
			Wt, g	% U ₃ O ₈	ml	U ₃ O ₈ g/l	% U ₃ O ₈	% U ₃ O ₈	
<u>Acid Leach Tests:</u>									
C7-A-2	10	2000 HNO ₃	9.595	0.124	250	0.447	2.31	48.5	Boiled 0.5 hour with 25 ml H ₂ O
A-3	10	500 H ₂ SO ₄ 100 MnO ₂	10.169	2.04	250	0.121	2.37	12.7	Cold leach 64 hours with 100 ml H ₂ O.
A-4	10	2000 H ₂ SO ₄	10.475	1.21	250	0.611	2.79	54.7	Boiled 4 hours at constant volume with 30 ml H ₂ O.
<u>Acid Bake Tests:</u>									
C7-A-1	5	2000 H ₂ SO ₄	--	--	250	0.935	--	97.1*	Heated slowly to SO ₃ fumes, 1 hour, taken up with water, boiled 15 minutes and filtered.
A-5	5	2000 H ₂ SO ₄	4.617	0.136	250	0.516	2.70	95.6	Diluted with 10 ml H ₂ O fumed twice over period of 2.5 hours, taken up with water, boiled and filtered.
A-7	5	1. 800 H ₂ SO ₄ 2. 200 H ₂ SO ₄	4.601	0.285?	250	0.229?	1.42	89.2**	Added 2.0 g H ₂ SO ₄ and baked in muffle at 275-300°C until fuming ceased - 15 mins. Boiled residue with 0.5 g H ₂ SO ₄ and 25 ml H ₂ O for 3 hours.
A-8	25	1. 1360 H ₂ SO ₄ 2. 640 H ₂ SO ₄	21.375	0.023	250	2.44	2.46	99.2	Mixed ore with 17g H ₂ SO ₄ and fumed slowly for 3 hours. Sample diluted twice with water during process. Sample cooled overnight, added 8 g H ₂ SO ₄ , fumed 0.5 hour, diluted and boiled for 2.5 hours with constant stirring.

*Based on solution and head analysis.

**Based on residue and head analysis.

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VI. PRECIPITATION OF URANIUM

Two precipitation tests were made using the pregnant solution from test A-8 as the head solution. In the first test (A-9) 50 ml of the liquor which contained originally 2.44 g of U_3O_8 per liter and had a pH of 0.2 was diluted with three volumes of water, boiled for 15 minutes to precipitate the titanium and filtered. The filtrate was neutralized with MgO slurry over a period of 30 minutes with constant stirring to pH 7.2 and the precipitate filtered off, washed, dried, weighed and analyzed. The results are shown in Table 4.

In the second test (A-10) the titanium was precipitated from a 50-ml sample in similar fashion. The filtrate was then neutralized to pH 8.5 with Na_2CO_3 , stirred for 30 minutes and the precipitate filtered off and washed. The filtrate was acidified to pH 3.0 with sulfuric acid and boiled for 15 minutes to eliminate CO_2 and then neutralized to pH 7.0 with MgO and stirred for 30 minutes. This precipitate was likewise filtered and washed. Analyses, distribution of products and consumption of reagents are shown in Table 4.

Table 4. Precipitation of Pregnant Solution from Test C-7-A-8

Test A-9

Product	Wt. or Vol.	U_3O_8	Per Cent Distribution
Precipitate	0.515 g	16.6%	90.9
Barren Solution	270 ml	0.032 g/l	9.1
Calc. Head	50 ml	1.88 g/l	100.0

Reagents

MgO-----0.55 g/g ore* = 1100 lb/ton

Test A-10

Product	Wt. or Vol.	U_3O_8	Per Cent Distribution
Na_2CO_3 Ppt	0.823 g	2.19%	19.4
MgO Ppt	0.156 g	47.76%	80.2
Barren Solution	225 ml	0.0017 g/l	0.4
Calc. Head	50 ml	1.86 g/l	100.0

Reagents

Na_2CO_3 -----1.4 g/g ore = 2800 lb/ton

H_2SO_4 -----0.2 g/g ore = 480 lb/ton

MgO -----0.02 g/g ore = 40 lb/ton

*In test A-8, 250 ml of pregnant solution was obtained from 25 grams of ore. Thus 50 ml of pregnant solution are equivalent to 5.0 grams of ore.

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Table 4 shows that bulk precipitation of the pregnant solution after removal of titanium by hydrolysis yields a comparatively low-grade precipitate but that precipitates containing about 50 per cent U_3O_8 can be obtained by removing the bulk of the impurities, probably chiefly iron, alumina and silica, with Na_2CO_3 prior to the precipitation of uranium. In each case, however, the consumption of reagents was excessive. Further research is therefore indicated to reduce the excess acidity of the pregnant solution and it is possible that a different type of solution chemistry will be better adapted to the recovery of a high-grade uranium precipitate at an economic reagent cost. This is particularly true in view of the fact that on a larger scale the pregnant solutions would have a concentration of uranium and other elements approximately 10 times that found in the pregnant solution from Test A-8.

In both tests the calculated heads are in good agreement with each other but are a poor check on the head solution which was originally assayed at 2.44 g of U_3O_8 per liter. In each test, the titanium precipitate obtained by hydrolysis was rejected without being assayed since the pure white color and small weight suggested that no appreciable amount of uranium was being lost in this product. However, in view of the low figures for the calculated heads, the remainder of the pregnant solution from Test A-8 was used to determine the loss of uranium.

Only 12 ml of the solution was available. This material was diluted with 3 volumes of water and boiled to precipitate the titanium as in Tests A-9 and 10. The precipitate was washed, ignited and assayed. The filtrate was made up to 100 ml and likewise assayed. Results of these determinations are shown in Table 5.

Table 5. Distribution of Uranium in $Ti(OH)_2$ Precipitate and Filtrate

Product	Wt. or Vol.	U_3O_8	Per Cent Distribution
<u>Test A-11</u>			
$Ti(OH)_2$ Ppt.	0.0427 g	0.217%	0.4
Filtrate	100 ml	0.249 g/l	99.6
Calc. Head	12 ml	2.12 g/l	100.0
Assay Head	12 ml	2.44 g/l	

The data in Table 5 show that the loss of uranium as a result of the hydrolysis of the titanium is negligible. The loss of uranium in Tests A-9, -10 and -11 may be in part the result of experimental and analytical errors but is probably chiefly due to the precipitation of uranium from the pregnant solutions while in storage. All of the pregnant solutions produced from C-7 formed precipitates on standing overnight and the original pregnant solution analyses were made on aliquots of solution and precipitate. It is to be expected that in actual plant practice this condition would not occur or, if it did, the precipitate could be periodically collected and treated.

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VII. RECOVERY OF OTHER ELEMENTS

Attention has been given only to the recovery of uranium but since the concentrates contain more than 10 per cent wolfram, as determined by spectrographic analysis, it seems likely that the economic recovery of uranium would be in large part dependent upon recovering a substantial part of the wolfram.

The manner in which this might be accomplished has not been investigated nor is there sufficient material at hand to carry on such an investigation. It seems probable, however, that leaching of the sulfated residues, with NH_4OH , after removal of uranium by the water leach, would recover part of the wolfram. Fusion of the concentrates with Na_2CO_3 or other fluxes or leaching of the concentrates in an autoclave with concentrated Na_2CO_3 solution may also be useful. The recovery of other elements, specifically titanium and tin, does not appear to be economic.

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APPENDIX A

COPY

United States
Atomic Energy Commission
Raw Materials Operations
New York Office

September 7, 1949

Mr. John Dasher, Executive Officer
Massachusetts Institute of Technology
Watertown Mineral Engineering Laboratory
Building 421, Watertown Arsenal
Watertown 72, Massachusetts

Subject: Sample for Study, New York Laboratory No. 258F

Dear Mr. Dasher:

Under separate cover we are sending you a portion of a black mineral concentrate originating at Climax, Colorado, with the Climax Molybdenum Company. The sample has been produced largely by gravity operations together with some magnetic treatment; it is the black residue from a monazite separation.

This concentrate assays 2.8% U_3O_8 equivalent against pitchblende standards, and the chief source of radioactivity has been identified by our Mineralogical Laboratory to be the rare uranium titanium oxide, brannerite. In addition to the brannerite, the sample contains a variety of minerals, among them monazite, black opaques including tin and tungsten minerals, quartz, pyrite and rutile (?). These minerals cannot be separated readily either by simple gravity means or magnetically. Spectrographic analysis on hand picked grains of brannerite shows, in addition to Ti, U, Y, Fe and Th, trace amounts of Ca and Pb.

Climax is anxious to learn if this material has any value to us and, therefore, we request that you make a few leaching tests on this sample in an effort to determine the ease with which uranium can be extracted. Although the results on somewhat similar material from Climax as described in your report MITG-204 were not promising, we believe that this additional limited work should be undertaken.

When you have completed your tests, it is requested that a report of the results together with such recommendations and suggestions as you may consider appropriate be submitted to this office.

Very truly yours,

/s/

Evan F. Wilson
Assistant Manager
Raw Materials Operations

CC: J. K. Gustafson
M. Mathez

COPY

APPENDIX B

10

United States
Atomic Energy Commission
Raw Materials Operations
New York Office

January 30, 1950

Mr. D'Arcy George
Massachusetts Institute of Technology
Mineral Engineering Laboratory
Building 421, Watertown Arsenal
Watertown 72, Massachusetts

Subject: Climax Molybdenum Company - Brannerite Concentrate

Dear Mr. George:

Following our recent discussions I have attempted to secure a clearer picture as to the quantities of Brannerite Concentrate produced by the Climax Molybdenum Company.

The information which I have been able to secure is not as specific as I would like, but it definitely adds up to the conclusion that only negligible quantities of brannerite concentrate are being produced by Climax. A letter from the Climax Molybdenum Company to the Atomic Energy Commission dated September, 1949 advises that the production of black residue (brannerite concentrate) would amount to only about 100 pounds per day on the basis of mine production rates then prevailing. It is my understanding at about that time Climax was operating at a rate of about 7,000 tons of ore per day. Therefore, at its maximum production rates there would be produced only some 300 to 350 pounds of brannerite concentrate per day. As this material apparently assays approximately 2% U_3O_8 , the indicated maximum amount of U_3O_8 which might be produced in the brannerite concentrate is of the order of 6 to 7 pounds per day.

I also have at hand a statement as to the amount of brannerite concentrate produced during the fourth quarter of 1949. This statement indicates that 3,250 pounds of concentrate were produced or, assuming 90 production days during the quarter, approximately 35 pounds per day. I do not know whether this production of brannerite concentrate represents the full mill production during that period or only a fraction of the total mill production. In any event it obviously represents an even smaller U_3O_8 potential than the figures quoted in the paragraph immediately above.

I trust that the above will be of assistance to you.

Very truly yours,

/s/

Arthur J. Beyer
Raw Materials Operations