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UNITED STATES ATOMIC ENERGY COMMISSION

PROCESSING OF AFRICAN ORES

By
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May 7, 1943

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Tonawanda, New York

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I. INTRODUCTION

A study has been made of methods of processing both high grade and low grade uranium ores from pitchblende deposits for recovery of uranium and radium. Although these ores have been processed for many years primarily for their radium contents, no detailed discussion of the processing is available in the literature. Accordingly, a laboratory study of the extraction of uranium and recovery of radium was made on two types of ore from the Belgian Congo. It was desired that a process should be developed which was adaptable to the existing equipment employed in the processing of carnotite ores.

II. DISCUSSION

It was necessary that any process adapted yield high recoveries of uranium in a purified state, and also provide for high recoveries of radium in the gangue. In addition, the process had to be adaptable to available equipment installed for the treatment of domestic uranium ore concentrates.

The processing of high grade pitchblende was first investigated, and the effect of various factors on the extraction and purification of uranium was studied. The adaptability of these procedures to low grade ore was then considered. Finally, since the process had been designed wholly from the point of view of uranium recovery, the behaviour of radium in the various steps was investigated.

Most of these experiments were first carried out in the laboratory, and the results then tested in the large scale laboratory.

III. EXPERIMENTAL

A. TREATMENT OF HIGH GRADE ORE

The high grade pitchblende was a 1300 pound sample of ore from Ketanga. The composition, as given by three laboratories, was that expressed in Table A.

1. EXTRACTION OF URANIUM

Since the literature indicates that a high concentration of sulfuric acid and an oxidizing agent are necessary for the extraction of uranium, tests were made in which the concentration and amount of sulfuric acid and the type and amount of oxidizing agents varied. In these tests a mixture of sulfuric acid, oxidizing agent, and ore was digested at 95°C. for several hours, and then filtered and washed. Table I shows the uranium extraction as a function of the amount and concentration of sulfuric acid, using 14 g. nitric acid per 100 g. of ore. All weights, in this table and subsequent tables, are given in terms of 100 g. of ore.

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TABLE A

Composition of Pitchblende Ore

	<u>Analysis after Drying at 110°C.</u>		
	<u>Linde</u>	<u>Metallurgical Laboratory</u>	<u>Bureau of Standards</u>
U ₃ O ₈	68.84%	66.95%	69.66%
PbO	6.07	—	6.27
SiO ₂	10.83	9.76	10.83
RE ₂ O ₃	0.47	0.035 (R.E.)	—
V ₂ O ₅	0.003	—	—
Fe ₂ O ₃	0.26	0.253 (Fe)	0.35
Al ₂ O ₃	—	—	1.55
MoO ₃	—	0.543 (Mo)	0.75
B	—	0.0029	0.0020
CaO	2.86	—	2.13
BaO	.04	—	—
Co	—	0.298	—
Cu	—	0.0070	—
Ni	—	0.856	0.86
Ra	—	—	1.78 x 10 ⁻⁵
Moisture at 110°C.	11.67%		

Table IVariation of Concentration and Amount of Sulfuric Acid

<u>Concentration</u> <u>H₂SO₄</u>	<u>Weight H₂SO₄ (96%)</u>	<u>% Uranium Extracted</u>			
		<u>25g.</u>	<u>50 g.</u>	<u>75 g.</u>	<u>100 g.</u>
50%		92.8	97.9	98.6	97.9
40%		94.6	94.5	97.5	98.0
30%		95.0	95.8	96.6	100.0
15%		91.3	91.0	80.5	89.0

These data indicate that concentrations of sulfuric acid above 15% are necessary for good uranium extraction, but the poorer results with 15% acid are not due to incomplete attack of the ore. The filtration and washing times for 15% acid were about 60 hours, whereas, the higher concentrations of acid gave filtration times of about one hour. The lower results with 15% acid are thus probably due to incomplete washing, and this is borne out by work which is summarized later in the report.

The necessity for an oxidizing agent, and the comparative value of various oxidizing agents is shown in Table II.

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Table II

Effect of Oxidizing Agents on Uranium Extraction

<u>Oxidizing Agent</u>	<u>Weight Oxidizing Agent</u>	<u>Uranium Extraction</u>
None	—	44.6%
Nitric Acid	8 g.	97.5
" "	11	96.6
" "	14	97.9
" "	21	98.9
Sodium Chlorate	3.3	75.6
" "	5	97.5
" "	6.6	98.1
" "	8	96.6
" "	12	94.5
Hydrogen Peroxide (30%)	25	39.6
Manganese Dioxide	10	94.4
(African Ore)		
" "	15	96.5
" "	20	96.0
" "	25	95.5
Manganese Dioxide	10	85.4
(Montana Ore)		
" "	15	95.5
" "	20	97.1
" "	25	96.0

It was concluded that nitric acid, sodium chlorate, and manganese dioxide could be used to oxidize pitchblende. The choice depends on the relative costs and the resistance of materials of construction to attack by the oxidizing agents. If wood tanks are employed, manganese dioxide is the preferred oxidizing agent.

Although the above results show that it would be possible to extract uranium from pitchblende with concentrated sulfuric acid, it was considered advantageous to use a process that could be applied to either carnotite or pitchblende with only slight modification on changing from one material to the other.

If pitchblende is treated with concentrated sulfuric acid, it is necessary to purify the solution of uranyl sulfate by the addition of sodium carbonate. This precipitates the basic impurities and leaves the uranium in solution as a double carbonate. The treatment of carnotite is the same in principle, but there is no intermediate filtration of acid-insoluble impurities and dilute sulfuric acid is used instead of concentrated acid.

It has been noted with carnotite concentrates that filtration of the acid slurry is markedly slower than filtration of the slurry obtained by adding soda ash to the acid slurry. When pitchblende was treated with concentrated sulfuric acid and the acid slurry converted to a soda ash slurry without intermediate filtration, the same difference in rates of filtration was observed.

The effect of alkaline digestion on rate of filtration was particularly pronounced when dilute sulfuric acid was used to treat pitchblende; 15% sulfuric acid gave an acid slurry which required 60 hours of filtration, while a filtration time of 35 minutes was sufficient when an alkaline digestion followed the use of 10% sulfuric acid. In addition, the extraction of uranium increased from 90% to 98%. This was partially due to the decreased hold-up of uranium in the cake.

That high uranium extractions can be obtained with dilute sulfuric acid when the acid slurry is subsequently treated with soda ash is shown in Tables III and IIIA. In these tables the uranium extractions are shown after treatment with various amounts of sulfuric acid. Ore was digested at 95°C. with sulfuric acid and oxidizing agent, and the acid slurry was poured into a cold solution of soda ash. The volume of the final slurry was kept at about 1500 cc. per 100 g. ore by varying the amount of water used to dissolve the soda ash. Substantially the same results would be obtained if solid soda ash were added to acid slurry diluted with cold water. After the soda ash addition the suspensions were agitated and digested at 95°C. for several hours.

Table III

Extraction of Uranium as Sodium Uranyl Carbonate
After Digestion with 10% Sulfuric Acid and 15 g. Pyrolusite*

<u>Weight of</u> <u>H₂SO₄</u>	<u>Time of</u> <u>Digestion</u> <u>with Acid</u>	<u>Weight of</u> <u>Na₂CO₃</u>	<u>Time of</u> <u>Digestion</u> <u>with Na₂CO₃</u>	<u>Per cent of</u> <u>Total Uranium</u> <u>Extracted</u>
60 g.	2 hrs.	200 g.	1 hr.	98%
50	1	200	1	94
50	2	200	1	95.5
50	4	200	1	96
50	6	200	1	95.5
50	2	230	1	98.2
50	2	165	1	97
50	2	135	1	95.5
50	2	200	2	95.9
50	2	200	3	97.2
50	2	200	5	97.8
40	2	123	1	96.5
40	2	135	1	96.8
40	2	200	1	97.0
35	2	135	1	95.2
32	4	135	2	97.9
32	4	114	3	95.9
32	2	135	2	94.7
32	2	114	3	93.5
30	2	200	1	87.8

* Nitric acid or sodium chlorate could have been used equally well.

Table IIIA

Concentration H_2SO_4	Weight 100% H_2SO_4	Time of Digestion	Weight Na_2CO_3	Time of Digestion	Oxidizing Agent	% Total Uranium Extracted
50%	50 g.	2 hrs.	200 g.	3	HNO_3 - 8g.	97.5%
50	50	2	200	3	MnO_2 - 15	99.5
10	50	2	200	3	HNO_3 - 8	100
10	50	2	200	3	MnO_2 - 15	99
10	33	4	135	3	HNO_3 - 8	91.1
10	33	4	114	3	HNO_3 - 8	94.0
10	33	4	135	3	MnO_2 - 15	93.7
10	33	4	114	3	MnO_2 - 15	91.6
10	33	4	135	3	HNO_3 - 12	97.1
10	33	4	114	3	HNO_3 - 2	95.6
10	35	2	135	3	MnO_2 - 8	82.7
10	35	2	135	3	MnO_2 - 9	90.3
10	35	2	135	3	MnO_2 - 10	91.3
10	35	2	135	3	MnO_2 - 12	96.3
10	32	7	135	3	MnO_2 - 10	92.5
10	31	7	135	3	MnO_2 - 10	91.8
10	35	2	110	3	MnO_2 - 12	93.1
10	35	2	100	3	MnO_2 - 12	90.5
10	35	2	90	3	MnO_2 - 12	88.5
10	35	2	85	3	MnO_2 - 12	86.8
10	32	2	90	3	MnO_2 - 10	84.1

The conditions for extraction which were selected from the data in Tables III and IIIA are: acid digestion - 2 hrs., alkaline digestion - 3 hrs., H_2SO_4 (100%) - minimum of 35 g. per 100 g. ore, Na_2CO_3 - minimum of 150 g. per 100 g. ore, MnO_2 - minimum of 12 g. per 100 g. ore, Volume of alkaline slurry - 1500 cc. per 100 g. ore. These conditions were recommended to the Large Scale Laboratory. The results which were obtained there are given in Table IIIB.

Table IIIB

Extraction of Uranium from 25-lb. Samples of Ore

Digestion	Per Cent Total Uranium Extracted		
	Liquor	Residue	Material Balance
1	77.2%	99.5%	72.6%
2	90.3	99.5	91.2
3	98.8	99.0	99.9

The high extractions which are shown by the analysis of the residues in Digestions 1 and 2 indicate that the extraction obtained in Digestion 3 is correct. The good material balance in Digestion 3 also shows that 98% or more of the uranium can be extracted.

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Since very good uranium extractions were obtained in the Large Scale Laboratory, the proposed precessing appears to be satisfactory for use in the Plant.

Certain data which are required in estimating equipment capacities were obtained during the tests of the process:

1. The filtration rate of the slurry was 3.45 gal. of filtrate per sq.ft. per hr., with a final cake 1" thick.
2. The washing rate was 1.5 gal. per sq.ft. per hr. for a 1" cake.
3. One pound of ore was equivalent to 1.8 gal. of filtrate.
4. The wet cake density was 70 lb. per cu.ft.
5. The apparent density of the dry cake was 97 lb. per cu.ft.
6. The moisture content of the wet cake was 65 - 70%.

From these data it was estimated that the Moore filter in the processing Plant could handle twice the planned production.

2. PURIFICATION OF URANIUM LIQUORS

Analysis of the alkaline extraction liquor showed that substantially all of the molybdenum present in the ore was extracted and that an appreciable amount of lead was dissolved by the sodium carbonate solution. Since only a limited amount of molybdenum is soluble in concentrated uranyl nitrate solutions, and since this limited amount is extracted by water in the ether purification step, it would probably not be necessary to introduce a molybdenum removal step. However, the separation was studied so that a process would be available if needed.

The possibility of precipitating molybdenum as lead molybdate was first explored. Lead sulfate was added to solutions of sodium uranyl carbonate which contained 1.05% Mo (based on U_3O_8) after the pH was adjusted with sulfuric acid. The slurry was digested at 90°C. for three hours and filtered.

The treatments are summarized in Table IV.

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Table IV.

pH		% Mo in Solution (based on U_3O_8)		% Total Mo in Solution
Initial	Final	PbSO ₄ (10 x theory)	PbSO ₄ (50 x theory)	
9	9.4	0.55%		53%
9	9.3		0.21%	20.4
8	9.2	0.51		48.8
8	9.2		0.40	38.5
7.5	9.2	0.53		50.5
7.5	9.0		0.26	25.2
7	9.25	0.51		48.8
7	8.7		0.39	34.2
3	3.3	0.03		2.63
3	4.4		0.22	21.4
2	2.0	0.02		2.0
2	2.3		0.02	1.6

The molybdenum is removed to the greatest extent from weakly acid solutions, but the lead content increases to 1% (based on U_3O_8) at the same time. This procedure would not be as satisfactory as the sulfide precipitation, which is described below.

Since the lead which dissolves in the sodium carbonate solution must be precipitated as lead sulfide, it would require very little modification of the lead removal step to remove molybdenum as the sulfide. When hydrogen sulfide is added to an alkaline solution containing both lead and molybdenum, insoluble lead sulfide and soluble sodium thiomolybdate are formed. On acidification sodium thiomolybdate decomposes to molybdenum sulfide.

In the tests sodium sulfide was added to the alkaline solution obtained by extracting pitchblende. Since lead sulfide is insoluble in both acid and alkali, while molybdenum sulfide is insoluble in acid only, two procedures were used: in one, the lead sulfide was removed before acidification; in the other, the molybdenum and lead were removed together. The results are presented in Table V.

Table V.

Precipitation of Lead and Molybdenum Sulfides

		2 x Theory		5 x Theory		10 x Theory		20 x Theory	
		A*	B**	A*	B**	A*	B**	A*	B**
Filtered before and after acidification	Mo	79%	0.15%	87%	0.09%	99%	0.06%	99.9%	0.009%
	Pb	18	0.13	41	0.09	88	0.02	95	0.009
Filtered after acidification only	Mo	77	0.16	86	0.10	99	0.01	99.9	0.008
	Pb	27	0.12	53	0.08	87	0.02	93	0.01

* Per cent of total metal removed.

** Per cent in solution, based on U_3O_8 .

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These data show that no appreciable difference in purity is obtained by the use of the two procedures; however, a single filtration has the advantage of economy of operation, and a double filtration yields a molybdenum concentrate. There was no loss of uranium in the sulfide precipitates.

If only lead were removed, it is apparent that larger amounts of sodium sulfide would be required than for a solution free of molybdenum, because some sulfide reacts to form thiomolybdate.

The leach liquor contained 3.38 g. U_3O_8 , 0.035 g. MoO_3 , and 0.069 g. PbO per 100 cc. The theoretical amount of sodium sulfide corresponds to 0.023 lb. per lb. U_3O_8 in this case.

In order to insure complete conversion of molybdenum to thiomolybdate, the test for completeness of precipitation of lead can be used, since comparison of the lead can be used, since comparison of the lead and molybdenum removals in Table V shows that sulfide reacts preferentially with molybdenum. Complete precipitation of lead in the alkaline solution, therefore, means that molybdenum is completely converted to thiomolybdate.

If the uranium solution were not purified, the final product would contain some molybdenum. This would not amount to all of the molybdenum that is present in the digestion liquors, since some is lost in the processing to black oxide. Tests indicate that 20-30% of the molybdenum passes through the acid-leach step with the uranium. This means that molybdenum would tend to build up in the product precipitation step.

Black oxide containing molybdenum can be processed in Step II, because MoO_3 has a limited solubility in concentrated uranyl nitrate solution. Black oxide was dissolved in nitric acid and the solution was evaporated to dryness, g. 1.85 and filtered. The initial and final molybdenum contents are given in Table VI.

Table VI.

Solubility of MoO_3 in Uranyl Nitrate

Per cent Mo Based on U_3O_8	
<u>Initial</u>	<u>Final</u>
0.15%	0.081%
0.15	0.048
0.47	0.041

These results show that the molybdenum content of uranyl nitrate will not exceed 0.1%. It has been shown before that this amount is removed in the ether step.

B. TREATMENT OF LOW GRADE ORE

A test of the general applicability of the process, developed for the extraction of uranium from carnotite and high grade pitchblende, was carried

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out with a sample of low grade ore from the Belgian Congo. The ore was a material containing 24.8% U_3O_8 . It was amenable to the treatment with dilute sulfuric acid without change in the processing scheme. Representative extractions and reagent requirements are given in the following table:

Table VII.

Extraction of Uranium from Low Grade African Ore
with 10% Sulfuric Acid

Run	Weight H_2SO_4	Weight Na_2CO_3	Weight MnO_2	Per Cent Total Uranium Extracted
1	15 g.	60 g.	4.5 g.	99.5
2	18	65	4.5	99.6
3	22.5	70	4.5	98.9
4	30	80	4.5	99.3

Since 15 g. of sulfuric acid were sufficient, the following conditions were selected as the minimum for good extraction: H_2SO_4 - 15 g. per 100 g. ore, Na_2CO_3 - 60 g. per 100 g. ore, MnO_2 - 4.5 g. per 100 g. ore, volume of alkaline slurry - 500 cc. per 100 g. ore, acid digestion - 2 hrs., alkaline digestion - 3 hrs.

When these conditions were used with low grade ore in the Lerre Scale Laboratory, a 98% extraction of uranium was obtained.

C. RECOVERY OF RADIUM FROM PITCHBLLENDE

When pitchblende is processed for its uranium content, it is necessary to recover the radium values of the ore at the same time. The low concentration of radium in the ore means that care must be taken to insure complete recovery, and this can be accomplished by using processing steps which do not dissolve radium. Since the extraction of uranium had been studied primarily from the standpoint of uranium recovery, it was necessary to carry pitchblende through the proposed processing and find the distribution of radium.

The results indicate that 99.9% or more of the radium is retained in the filter cake which results when uranium is extracted in acid or alkaline solution. It appears that it is not necessary to add additional barium to insure complete recovery of radium, although the addition would introduce a safety factor, if radium analyses are not to be part of a routine analytical procedure.

In this work filter cakes and uranium liquors were analyzed for radium after uranium had been dissolved by treatment of pitchblende with sulfuric acid and an oxidizing agent, and after the above acid slurry had been added to sodium carbonate solution. Various oxidizing agents and various concentrations and amounts of sulfuric acid were tested.

The samples which were obtained were analyzed for radium by the Radioactivity Section of the National Bureau of Standards.

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The conditions of treatment and radium recoveries are presented in Tables VIII and IX. Two series of determinations were made: in one, pitchblende was digested with sulfuric acid and an oxidizing agent, and the insoluble residue, containing radium sulfate, was filtered off; in the other, a similar acid digestion was made, but the uranium was obtained in purer form by adding the acid slurry to a solution of sodium carbonate. In the second treatment the residue contained more foreign matter than the first, and the volume of filtrate was greater because the solubility of sodium uranyl carbonate is lower than that of uranyl sulfate.

The radium analyses of the filtrates were quite consistent, but the analyses of the solid were very erratic. This is probably due to heterogeneity of the solids and to errors in weighing the cakes.

Comparison of the radium contents of acid and alkaline solutions seems to indicate an increased solubility in the alkaline solutions. While there may be a slightly greater solubility, the greater amount in solution is due to the fact that the volume of solution was 5 to 7 times greater in the case of alkaline liquors.

The proposed processing appears very favorable from the standpoint of radium recovery, since recoveries do not seem to be affected by pH, concentration of acid or by change of oxidizing agent. It is understood that radium producers are not unduly disturbed unless the recoveries at this point go below 95%.

Table VIII.

Extraction of 100 g. Samples of Pitchblende
with 50% Sulfuric Acid

Conditions				Radium Recovery	
Amount of 96% H ₂ SO ₄	Oxidizing Agent	Barium Chloride		% of Total Ra in Solution	% of Total Ra in Residue
100 g.	HNO ₃ - 14 g.	0		0.006%	99.7%
100	"	"	1 g.	0.0001	112
75	"	"	1	0.004	97
100	"	11	1	0.003	101
100	"	21	1	0.003	115
75	"	8	1	0.010	113
100	"	14	2	0.003	94.7
100	"	"	0.5	0.004	104
100	"	"	0.25	0.004	100.8
100	NaClO ₃ - 6.6	1		0.006	99.5
100	"	8	1	0.002	99.4
100	"	12	1	0.004	113

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Table IX

Extraction of Uranium as $\text{UO}_2(\text{CO}_3)_3$
from 100 g. Samples of Pitchblende

<u>Conditions</u>			<u>Radium Recovery</u>			
<u>Concentration</u> <u>H_2SO_4</u>	<u>Amount of</u> <u>96% H_2SO_4</u>	<u>Oxidizing</u> <u>Agent</u>	<u>Barium</u> <u>Chloride</u>	<u>Sodium</u> <u>Carbonate</u>	<u>% of Total Radium</u>	
					<u>In Solution</u>	<u>In Residue</u>
10%	50 g.	HNO_3 - 8 g.	1 g.	200 g.	0.11%	101.8%
10	50	MnO_2 - 15	1	200	0.105	110
50	50	HNO_3 - 8	1	200	0.088	91.8
50	50	MnO_2 - 15	1	200	0.064	90.4
10	33	MnO_2 - 15	1	135	0.130	109
10	33	MnO_2 - 15	1	114	0.102	83.6
10	33	HNO_3 - 12	1	135	0.062	117.5
10	33	HNO_3 - 12	1	114	0.146	102.8

D. CONVERSION OF BARIUM SULFATE TO BARIUM CARBONATE

When this work was started, it was understood that radium-bearing residues would be processed by some other company. A literature survey of radium extraction was made, and it was concluded that the major chemical step, which would require investigation if the decision to prepare radium concentrates in the uranium processing Plant were made, was the conversion of barium-radium sulfate to barium-radium carbonate.

Barium sulfate was heated with various amounts of sodium carbonate and water at a number of temperatures. The results, which are summarized in Table X, show that 98% conversion can be obtained by heating 100 parts of BaSO_4 with 200 parts Na_2CO_3 and 200 parts H_2O at 135°C . for 1 hour.

Table X.

Conversion of BaSO_4 to BaCO_3

<u>Variable</u>	<u>BaSO_4</u>	<u>Na_2CO_3</u>	<u>H_2O</u>	<u>Temperature</u>	<u>Time</u>	<u>Per Cent Conversion</u>
Soda Ash						
Ratio	100 g.	100 g.	200 g.	150°C .	1 hr.	51.2
	100	150	200	150	1	90
	100	200	200	150	1	98.2
	100	250	200	150	1	94.6*
Water Ratio	100	200	200	150	1	98.2
	100	200	250	150	1	96.3

* Solidified because of insufficient water.

Table X (Cont'd.)

<u>Variable</u>	<u>BrSO₄</u>	<u>Na₂CO₃</u>	<u>H₂O</u>	<u>Temperature</u>	<u>Time</u>	<u>Per Cent Conversion</u>
Temperature	100	200	200	100	1	66.4
	100	200	200	135	1	98.5
	100	200	200	140	1	98.7
	100	200	200	150	1	98.2
	100	200	200	165	1	98.8
<u>Time</u>	100	200	200	150	1/2	96.5
	100	200	200	150	1	98.2
	100	200	200	150	2	98.3
	100	200	200	150	3	98.7

IV. SUMMARY

A research program was carried out to develop a process for treating ore from pitchblende deposits which would allow high recoveries of uranium, and recovery of radium in the residue, and which would be adaptable to existing process equipment. Laboratory and Large Scale Laboratory work lead to the development of such a process, and the determination of the optimum conditions and reagent requirements for successful operation. High uranium extractions were allowed, leaving 99.9% of the radium in the residue from the uranium extraction, and a method for the removal of molybdenum and lead from the liquors was developed.

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