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NYO-1163

PROBLEMS OF REFINING URANIFEROUS RESIDUES

Technology—Feed Materials Progress Report for July 1953

By

H. Fleck

J. E. Summers

August 15, 1953

Vitro Manufacturing Company

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PROBLEMS OF REFINING URANIFEROUS RESIDUES

Vitro Manufacturing Company

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Hugh Kinser
Authorizing Official
Date: 11/24/09

H. Fleck and J. E. Summers

August 15, 1953

AEC RESEARCH AND DEVELOPMENT REPORT

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PROBLEMS OF REFINING URANIFEROUS RESIDUES
TECHNOLOGY - FEED MATERIALS

A. SUMMARY

GENERAL

1. Scope of Program

As of July 1, 1953, the Research Program consisted of the following projects:

Digestion and Refining of C-Slags and Other Residues
(Project No. 1)

Investigate the applicability and refinement of various procedures for the treatment and digestion of C-Slags and other uraniferous residues for recovery of uranium values, placing emphasis on methods adaptable to the Canonsburg Plant with minimum change of equipment.

Refining of BFC-6
(Project No. 4)

Investigate the recovery of uranium from BFC-6 placing emphasis on procedures which will not chemically alter the copper and tin media.

Refining and Processing of Sodium Carbonate Residues
(Project No. 5)

Investigate methods of recovery of uranium from various residues, resulting from sodium carbonate digestion of

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miscellaneous uranium bearing materials, to reduce the U_3O_8 content of the residues below 0.05%.

2. Previous Reports

NYO-1145	March	1952 - Progress Report No. 11
NYO-1146	April	1952 - " " No. 12
NYO-1147	May	1952 - " " No. 13
NYO-1148	June	1952 - " " No. 14
NYO-1149	July	1952 - " " No. 15
NYO-1150	August	1952 - " " No. 16
NYO-1151	September	1952 - " " No. 17
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NYO-1157	March	1953 - " " No. 23
NYO-1158	April	1953 - " " No. 24
NYO-1159	May	1953 - " " No. 25
NYO-1162	June	1953 - " " No. 26

PROJECT STATUS

3. Research Project - Digestion and Refining of C-Slags and Other Residues

Experiments were conducted on samples from recent shipments of FMPC Sump Cake. Mineral acid digestion was tested on both calcined and uncalcined material. The purpose of experimentation was to obtain an acid discard residue containing less than 0.05% U_3O_8 . Hydrochloric acid and sulfuric acid were ineffectual on uncalcined material. Calcination of the raw material prior to acid digestion lead to satisfactory results.

Additional experiments were conducted in order to determine if a satisfactory means of extraction could be found which would eliminate the calcination phase. The raw material was digested in alkaline reagents including both sodium carbonate and sodium hydroxide prior to acid digestion. Acid insoluble residues obtained after prior digestion in an alkaline reagent contained less than 0.05% U_3O_8 . Additional experiments conducted using sodium carbonate

exclusively as a digest media indicate that uranium may be quantitatively removed from the uncalcined material leading to a discard residue containing less than 0.05% U_3O_8 .

4. Research Project - Digestion and Refining of BFC-6

A series of experiments was conducted to determine the effectiveness of various oxidizing agents with regard to the extraction of uranium from BFC-6, leading to insoluble residues containing less than 0.05% U_3O_8 .

Experiments were conducted on uncalcined BFC-6 to determine if sodium hydroxide digestion at low pressures would be effective in removing metallic tin from the raw material. Accumulated data indicates that this procedure would be only partially effective.

5. Research Project - Digestion and Refining of Sodium Carbonate Residues

This project, during the present report period, was confined to pilot level experiments. However, finalized data is not yet available and will not be reported during this period.

B. DIGESTION AND REFINING OF C-OXIDE AND OTHER RESIDUES

Research Project No: 1

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

6. Digestion and Refining of FMPC Sump Cake

Experiments were initiated on newly arrived shipments of FMPC sump cake to determine if the material would

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be amenable to hydrochloric acid or sulfuric acid digest techniques. Fifty gram samples of the raw material were slurried in 300 ml of water and digested in the presence of hydrochloric and sulfuric acids respectively at approximately pH 0.0 for three hours at 90° C. Sodium chlorate was used to oxidize. An examination of the acid insoluble residue resulting from this treatment indicated a uranium level of approximately 0.70% U_3O_8 . It is fairly obvious from previous experimental data that this material differs somewhat from the original experimental raw material.

Samples of the raw material were calcined at 1200° F for one-half hour and then subjected to mineral acid digestion. Fifty gram samples of the calcined material were slurried in 300 ml of water and digested in hydrochloric acid at pH 0.0 for three hours at 90° C. Acid insolubles were separated from the digest slurry by filtration and analyzed for uranium content. The insoluble residue contained less than the maximum specification of .05% U_3O_8 for a discard residue. It is apparent from preliminary data that calcination of the raw material will be necessary to process the material unless other procedures can be developed. Average data may be found in Table I.

Experimentation was initiated to determine if a process could be found which would eliminate calcination of sump cake prior to acid digestion and give satisfactory extraction of uranium. It was felt that digestion in an alkaline media prior to hydrochloric acid digestion might be effective in resolving uranium complexes which did not permit complete acid dissolution.

Fifty gram samples of uncalcined material were slurried in 300 ml of water and digested in the presence of 10% (w/v) sodium hydroxide for six hours at 90° C. The filtrate was removed from the residue by a process of decantation and filtration. The resultant residue was slurried in 300 ml of water and digested in hydrochloric acid at pH 0.0 for three hours at 90° C in the presence of sodium chlorate to oxidize. Acid insolubles were removed by filtration and washed with water until color free. An examination of the uranium content indicated a uranium level well below the maximum specification of 0.05% U_3O_8 for a discard residue.

In addition, experiments were conducted using sodium carbonate as a digest media prior to hydrochloric acid digestion. Fifty gram samples of uncalcined sump cake were slurried in 300 ml of water and were digested in the presence of 10% (w/v) sodium carbonate for six hours at 90° C. The pregnant filtrate was separated from the digest slurry by filtration. The insoluble residue was washed with water until color free. The resultant residue contained on the average .25 - .30% U_3O_8 . When this residue was slurried in 300 ml of water and digested in hydrochloric acid at pH 0.0 for three hours at 90° C using sodium chlorate as an oxidizing agent, the uranium content was reduced to less than 0.05% U_3O_8 . Preliminary data indicates that either procedure will be satisfactory in extracting uranium from FMPC sump cake without preliminary calcination of the raw material. Average data may be found in Table II.

It appeared to be propitious to continue the experimentation on sodium carbonate digestion of uncalcined sump cake in order to determine if additional digestion of this type would lead to a satisfactory residue. Fifty gram samples of the raw material were slurried in 300 ml of water and digested in 10% (w/v) sodium carbonate for six hours at 90° C. The insoluble residue was separated from the pregnant filtrate by filtration and washed with hot 5% sodium carbonate solution until color free. Analysis of the residue indicated a uranium level averaging between .25 - .30% U_3O_8 . This residue was reslurried in 300 ml of water and digested in the presence of 5% (w/v) sodium carbonate for three hours at 90° C. The carbonate residue, after separation by filtration and adequate washing, was again analyzed for uranium content. Analysis indicated the uranium level within the discard specification of 0.05% U_3O_8 . It may be that this would be the most satisfactory procedure for the extraction of uranium from this material. Average data may be found in Table III.

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C. DIGESTION AND REFINING OF BFC-6

Research Project No.: 4

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

7. Extraction of Uranium from BFC-6 Using Mineral Acid Digestion

Experiments to date on the extraction of uranium from BFC-6 using mineral acid digest techniques had been unsuccessful in reducing uranium content of acid insoluble residues to less than 0.05% U_3O_8 . An investigation of the oxidization phase of acid digestion was indicated. A series of experiments was completed in which the effectiveness of various oxidizing agents was studied. One hundred gram samples of the raw material were calcined at 1,000° F for one hour and slurried in 300 ml of water. Digestion was carried out in the presence of sulfuric acid at approximately pH 0.0 for six hours at 95° C. The digest was oxidized during digestion using various oxidizing agents such as calcium hypochlorite, sodium hypochlorite, magnesium dioxide, hydrogen peroxide and varied amounts of sodium chlorate. Acid insolubles were separated from the digest slurry by filtration and washed with water until color free. Analysis of the insoluble residue for uranium content did not indicate any appreciable beneficiation from the variation in oxidizing agents. No further experimentation of this nature is indicated. Acid insolubles averaged between .15 - .8% U_3O_8 . Average data may be found in Table IV.

Prior to this report period experiments had been conducted on uncalcined BFC-6 to determine if metallic

tin could be removed from the raw material by digestion in sodium hydroxide. This treatment had been only very moderately successful. Experiments were initiated to determine if digestion of the raw material in sodium hydroxide would be materially improved by digestion at pressures up to 65 psig. Two-hundred-fifty gram samples of uncalcined BFC-6 were slurried in 1,000 ml of water and digested in the presence of 1,000 grams of sodium hydroxide at 45 - 65 psig for six hours. The supernatant liquor was removed by a process of decantation and filtration. While the procedure was successful in dissolving some of the metallic media, very little success may be reported. The uranium level was elevated from approximately 19% to 25% U_3O_8 . No further experimentation is considered necessary due to the poor preliminary results. Representative data may be found in Table V.

D. REFINING AND PROCESSING OF SODIUM CARBONATE RESIDUES

Research Project No.: 5

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

8. The Extraction of Uranium from Pitchblende Carbonate Residues by a Process of Sodium Carbonate and Hydrochloric Acid Digestion

Experimental work during the present report period on Pitchblende carbonate residues was confined to pilot level operations. Data from this experimentation has not reached a finalized stage and will not be reported during the present report period.

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EXHIBITS

TABLE I
MINERAL ACID DIGESTION OF FMPC SUMP CAKE

Assay: 10 - 12% U_3O_8

EXPERIMENT NUMBER

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Calcination Temperature (°F)	1200	1200	-	-
Time of Calcination (hr.)	0.5	0.5	-	-
Weight of Sample (gm.)	50	50	50	50
Weight of H_2O (gm.)	300	300	300	300
Acid Used	HCl	H_2SO_4	HCl	H_2SO_4
Weight of Acid (gm.)	110	66.5	110	66.5
Digest Time (hr.)	3	3	3	3
Digest pH	0.0	0.0	0.0	0.0
Digest Temperature (°C)	95	95	95	95
Acid Insoluble Residue (gm.)	4.2	41.2	4.5	40.8
Acid Insoluble Residue (% U_3O_8)	.05	.07	.72	.54

TABLE II
REFINING OF SUMP CAKE BY ALKALINE AND ACID DIGESTION

Assay: 10 - 12% U_3O_8

EXPERIMENT NUMBER

	<u>1</u>	<u>2</u>
Weight of Sample (gm.)	50	50
Weight of H_2O (gm.)	300	300
Alkali Used	NaOH	Na_2CO_3
Weight of Alkali (gm.)	30	30
Digest Time (hr.)	6	6
Digest Temperature (°C)	95	95
H_2O Used to Slurry Residue (gm.)	300	300
Weight of HCl (gm.)	90	60
Digest Time (hr.)	3	3
Digest Temperature (°C)	90	90
Digest pH	0.0	0.0
Acid Insoluble Residue (gm.)	3.8	4.1
Acid Insoluble Residue (% U_3O_8)	Traces	Traces

//

TABLE III

SODIUM CARBONATE DIGESTION OF FMPC SUMP CAKE

Assay: 10 - 12% U_3O_8

	EXPERIMENT NUMBER	
	1	2
Weight of Sample (gm.)	50	50
Weight of H_2O (gm.)	300	300
Weight of Na_2CO_3 (gm.)	30	30
Digest Time (hr.)	6	6
Digest Temperature ($^{\circ}C$)	95	95
Residue (% U_3O_8)	.25	.28
Weight of H_2O to Slurry Residue (gm.)	300	300
Weight of Na_2CO_3 (gm.)	15	15
Digest Time (hr.)	3	3
Digest Temperature ($^{\circ}C$)	90	90
Residue (gm.)	42.5	43.0
Residue (% U_3O_8)	.035	.055

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TABLE IV

EFFECT OF OXIDIZING AGENT ON THE H_2SO_4 DIGESTION OF BFC-6Assay: 19.6% U_3O_8

EXPERIMENT NUMBER

	1	2	3	4	5	6	7	8
Calcination Temperature ($^{\circ}\text{F}$)	1000	1000	1000	1000	1000	1000	1000	1000
Calcination Time (hr.)	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Weight of Sample (gm.)	100	100	100	100	100	100	100	100
Weight of H_2O (gm.)	300	300	300	300	300	300	300	300
Weight of H_2CO_4 (gm.)	62.5	86.5	64.4	60.7	71.7	79.0	64.4	75.4
Oxidizing Agent	$\text{Ca}(\text{OCl})_2$	MnO_2	NaClO_3	NaClO_3	H_2O_2	NaClO	NaNO_3	-
Weight of Oxidizing Agent (gm.)	21.1	19.0	10.0	15.0	30.0	15.0	14.0	-
Digest Time (hr.)	6	6	6	6	6	6	6	6
Digest Temperature ($^{\circ}\text{C}$)	95	95	95	95	95	95	95	95
Digest pH	0.0	0.2	0.0	0.1	0.0	0.0	0.0	0.0
Acid Insoluble Residue (gm.)	85.2	64.3	60.6	59.0	62.9	64.2	63.4	67.0
Acid Insoluble Residue (% U_3O_8)	.168	.41	.24	.49	.40	.80	.50	4.7
Recovery (%)	99.2	98.7	99.2	98.6	98.7	97.4	98.4	98.4

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Table V

SODIUM HYDROXIDE DIGESTION OF BFC-6 AT LOW PRESSURES

Assay: 19.6% U_3O_8

	EXPERIMENT NUMBER	
	<u>1</u>	<u>2</u>
Weight of Sample (gm.)	250	250
Weight of H_2O (gm.)	1000	1000
Weight of NaOH (gm.)	1000	1000
Digest Time (hr.)	6	6
Digest Pressure (psig)	45	65
Residue (% U_3O_8)	25.20	25.54

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