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UNITED STATES ATOMIC ENERGY COMMISSION

NYO-1157

PROBLEMS OF REFINING URANIFEROUS RESIDUES

Technology Feed Materials, Progress Report –
March 1953

By

H. Fleck

J. E. Summers

April 15, 1953

Vitro Manufacturing Company

Technical Information Service, Oak Ridge, Tennessee

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UNITED STATES ATOMIC ENERGY COMMISSION

PROBLEMS OF REFINING URANIFEROUS RESIDUES

Progress Report [for] March 1953

By
H. Fleck
J. E. Summers

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PROBLEMS OF REFINING URANIFEROUS RESIDUES
TECHNOLOGY - FEED MATERIALS

A. SUMMARY

GENERAL

1. Scope of Program

As of March 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 of 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.

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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 miscellaneous uranium bearing materials, to reduce the U308 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
NYO-1152	October	1952 - " " No. 18
NYO-1153	November	1952 - " " No. 19
NYO-1154	December	1952 - " " No. 20
NYO-1155	January	1953 - " " No. 21
NYO-1156	February	1953 - " " No. 22

PROJECT STATUS

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

Experimentation was initiated on AJ-4 to determine if uranium could be extracted from this material using either mineral acid or alkali carbonate techniques. In addition, experiments were conducted on the clarified filtrates to determine if the precipitation of solubilized uranium with ammonia would result in an acceptable end product containing more than 50% U and less than .5% F.

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Preliminary experiments were completed to study the effect of conversion temperature on the fluorine content of black oxides prepared from continuously precipitated ammonium di-uranate. In addition, experiments were conducted to assay the influence of temperatures of calcination on the fluorine content of black oxides prepared from ammonium di-uranates precipitated using batch techniques.

Preliminary experiments were completed on the continuous precipitation of ammonium di-uranate from a hydrochloric acid digestion filtrate prepared from C-Liner. Data was obtained on the influence of elevated calcination temperatures on fluorine content of black oxides prepared from this material. Additional experiments were completed to determine the effect of recycling continuously precipitated ammonium di-uranate prepared from C-Liner through the precipitate cycle. Preliminary results indicate substantial reduction in fluorine content of the end product and elevation of the uranium content.

4. Research Project - Refining of BFC-6

Experiments were conducted on BFC-6 using sulfuric acid digest procedures. Experiments were initiated concerning optimum digest conditions for the extraction of uranium from the raw material leaving discard residues containing less than .05% U308. pH of digestion, time of digestion and calcination temperature of the raw material prior to digestion were factors under investigation.

5. Digestion and Refining of Sodium Carbonate Residues

This project was held in abeyance during the present report period in order to concentrate on obtaining data of a more pressing nature.

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

Research Project No. 1

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

6. The Extraction of Uranium From AJ-4 Using Mineral Acid and Alkali Carbonate Digestion Techniques and Ammonium Precipitation

Experimentation on AJ-4 was initiated during the present report period. The purpose of experimentation was to extract residual uranium from this material using either mineral acid or alkali carbonate digest techniques, leaving discard residues containing less than .05% U308.

200 gram samples of the raw material were pulverized to less than 100 mesh, slurried in 400 grams of water, and digested in hydrochloric acid and sulfuric acid respectively at pH values approximating .25. Oxidation of the digest solution was effected using sodium chlorate or sodium nitrate, depending upon the digest media. Digestion was carried out over a three hour period at temperatures approximating 90° C. After digestion, acid insolubles were

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of the original volume and contained approximately .23% U308. Since the desirable uranium content for a discard residue is considerably lower than this value, the treatment is not considered practical with regard to AJ-4. Representative data may be found in Table I.

7 Study of the Effect of Conversion Temperature on the Fluorine Content of Black Oxides Prepared from Ammonium Di-Uranate

Some data had been obtained which indicated that temperature of calcination might have a decided effect upon the fluorine content of black oxides prepared from continuously precipitated ammonium di-uranate. A more extensive investigation of this possibility was deemed expedient at this time. Samples were taken from black oxides prepared on the production level from continuously precipitated ammonium di-uranate having fluorine contents in excess of 1.5%. These black oxides were recalcined over a temperature range extending from 1500° - 1800° F. Data obtained in this manner indicates that temperature of calcination has a very decided influence on the final fluorine content of the black oxide. It has been tentatively established that at 1700° F fluorine content of the residue will be substantially lowered and within statistical limits will meet maximum specifications of 0.5% fluorine content for an end product.

In addition, experiments of this nature were conducted on "batch" precipitated ammonium di-uranate. Both production and laboratory samples were used for experimentation. Data obtained to date indicates that if temperatures of calcination are elevated in excess of 1700° F for periods of time approximating one-half hour, that the fluorine level of the resultant black oxide will be less than .5% F. Preliminary data may be found in Table II.

8. The Purification of C-Liner Using Hydrochloric Acid
Digestion and Continuous Ammonium Precipitation Techniques

Preliminary experiments were initiated to determine if C-Liner could be adapted to the hydrochloric acid digestion and continuous ammonium precipitation process as presently established for C-Oxide. Previous experimentation had indicated that ammonium di-uranate precipitated from pregnant filtrates of C-Liner, when calcined to a black oxide at 1500° F, would contain excessive amounts of fluorine up to 5%. Maximum fluorine content of a product of this nature must be less than .5% F. to meet end production specifications. Experimentation was considered necessary to determine whether or not elevated temperatures of conversion would have a beneficial effect upon the fluorine content of black oxides obtained in this manner.

500 gram samples of uncalcined C-Liner were pulverized to less than 100 mesh, slurried in 500 ml of water and digested in commercial hydrochloric (20° B) at pH 0.0 for one and one-half hours in the presence of .5% (w/v) sodium chlorate at 90° C. Acid insolubles were separated by filtration and washed until color free with water. The clarified filtrate was combined with the wash and ammonium di-uranate was continuously precipitated at approximately pH 6.0.

The ammonium precipitate was separated from the slurry by filtration, divided into equal portions which were calcined at 1500° F and 1700° F respectively. Black oxides formed at 1500° F were found to contain fluorine in the order of magnitude of one to one and one-half percent F. Black oxides formed from continuously precipitated ammonium di-uranate at temperatures approximating 1700° F contained fluorine in quantities of less than .1% F.

Apparently the benefits of elevated conversion temperatures will extend to continuously precipitated ammonium di-uranate prepared from hydrochloric acid digestion of C-Liner. Preliminary data may be found in Table III.

Experiments were conducted on the continuous precipitation of ammonium di-uranate from C-Liner to determine what effect recycling the precipitate through the process would have on the fluorine content of the end product.

500 gram samples of uncalcined, less than 100 mesh C-Liner, were slurried in 500 grams of water and digested in 20° B commercial hydrochloric acid at pH 0.0 in the presence of .5% (w/v) sodium chlorate for one and one-half hours at 90° C. Acid insolubles were removed from the digest slurry by filtration and the pregnant filtrate was used for experimentation. Ammonium di-uranate was continuously precipitated over a range of pH 6 - 7 using 3:1 ammonium hydroxide. The precipitate was separated by filtration and washed until calcium and magnesium free.

Ammonium di-urante from this procedure was re-slurried in water and dissolved by the addition of hydrochloric acid to approximately pH 1.0. The pregnant solution was recycled through the continuous precipitation procedure using dilute 3:1 ammonia as a precipitant. pH of precipitation approximated 6.0. The precipitate was separated by filtration and dried at 100° C. Analysis of ammonium di-uranates obtained in this manner indicated substantial reduction in fluorine content. Portions of ammonium di-uranates obtained by the recycling process were converted to black oxide at approximately 1500° F. Fluorine reduction was considerable with fluoride contents of black oxides averaging less than .1% F. There was a corresponding increment in uranium content from approximately 40 - 45% U308 to 70 - 72% U308. Preliminary data may be found in Table IV.

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It is apparent from preliminary data that a satisfactory product as regards fluorine content could be obtained from C-Liner by this procedure.

C. REFINING OF BFC-6

Research Project No. 4

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

9. Mineral Acid Digestion of BFC-6

The project concerning the extraction of uranium from BFC-6 was reactivated. Experiments were initiated using mineral acid digest techniques with intent to obtain discard residues containing less than .05% U308. Sulfuric acid was selected as a solvent since metallic copper is insoluble in dilute sulfuric and tin is very slowly soluble under the same conditions. Some loss of copper and tin is to be considered inevitable but since the purification of this material and subsequent recovery of uranium is considered important at this time the loss need not be considered. Preliminary experiments concerning optimum digest factors have been completed.

A series of experiments was completed concerning optimum pH of digestion. 100 gram samples of the raw material were water leached to remove the soluble barium

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chloride contaminant, and calcined at 1000° F for one hour. The samples were slurried in 400 ml of water and digested in sulfuric acid for three hours over a pH range extending from 0.0 - 1.0. Sodium chlorate was used as an oxidizing agent. Preliminary results indicate that pH 0.0 will be necessary to effect maximum recovery. Representative data is found in Table V.

Additional experiments were conducted to determine the length of digest time necessary to obtain discard residues containing less than .05% U308. 100 gram samples of the raw water leached material were calcined at 1000° F for one hour and digested in sulfuric acid at pH 0.0 in the presence of sodium chlorate to oxidize at temperatures approximating 90° C over a time range extending from one to six hours. Preliminary results indicate that more protracted periods of digestion will be necessary to obtain extraction leading to discard residues containing less than maximum specification. Recovery at the six hour period was maximum to date with insoluble residues averaging .10% U308. Loss of the copper - tin media averaged 20% of the original volume. Representative data may be found in Table VI.

There was some indication that temperature of calcination might have an effect upon the extractability of contained uranium values. Experiments were conducted using 100 gram samples of the water leached material calcined over a temperature range extending from 800° F - 1200° F. Samples were slurried in 400 ml of water and digested in sulfuric acid at pH 0.0 in the presence of sodium chlorate to oxidize, for a period of three hours at 90° C. Preliminary results indicate that extraction is improved by calcination of the raw material at lower temperature. Optimum temperatures ranged between 800 - 900° F. Calcination temperatures exceeding 1000° have a detrimental effect upon the solubilization of contained uranium. Data is found in Table VII.

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Additional experiments were conducted on time of digestion using 800° F as a calcination value. Preliminary data from these experiments indicate that six hours will not be sufficient to extract uranium in satisfactory proportions. Discard residues averaged .1% U308. Additional experiments are to be conducted to determine the necessary time of digest to obtain discard residues containing less than .05% U308. Preliminary data may be found in Table VIII.

D. REFINING AND PROCESSING OF SODIUM CARBONATE RESIDUES

Research Project No: 5

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

10. Development of Processes For the Extraction of Uranium From Sodium Carbonate Residues Resulting as a By-Product of Pitchblende Purification.

Development work on the Pitchblende Carbonate Residues was held in abeyance during the period under report in order to concentrate on obtaining data of more pressing importance at this time.

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E X H I B I T S

TABLE I

EXTRACTION OF URANIUM FROM AJ-4 USING MINERAL ACID
DIGESTION AND SODIUM CARBONATE DIGESTION

Material: AJ-4

Assay: 0.91% U_3O_8 EXPERIMENT NUMBER

	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample (gm)	200	200	200
Weight of H_2O (gm.)	400	400	400
Solvent Used	HCl	H_2SO_4	Na_2CO_3
Weight of Solvent (gm)	60	63	30
Digest Time (hr.)	3	3	3
Digest Temperature ($^{\circ}C$)	90	90	90
Digest pH	0.25	0.20	10.0
Oxidant	$NaClO_3$	$NaNO_3$	---
Weight of Oxidant (gm)	0.5	2.0	---
Slurry Volume (ml)	675	580	550
ml Solids/100 ml Slurry	19.0	21.0	20.5
Insoluble Residue (gm)	166	168	181.0
Insoluble Residue (% U308)	.10	.045	.23
Filtrate Volume (ml)	800	750	700
pH of Precipitation	7.0	7.0	11.5
Precipitant Used	NH_4OH	NH_4OH	NaOH
Weight of Precipitant (gm)	50.0	50.0	14.0
ml Solids/100 ml Slurry	11.0	8.5	0.75
Residue (gm)	7.0	4.0	1.0
Residue (% U308)	25.0	60.96	73.0
Residue (% F)	.38	.24	---

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TABLE II

DETERMINATION OF EFFECT OF CALCINATION TEMPERATURE ON
FLUORINE CONTENT OF BLACK OXIDES

Material: Ammonium Di-Uranate

Source: Production Processes

Method of Precipitation	Cont.	Cont.	Cont.	Cont.	Cont.	"batch"	"batch"
Time of Calcination (hr.)	0.5	0.5	0.5	0.5	0.5	0.5	0.5
1500° F (% F)	.47	1.62	1.12	1.71	1.60	1.47	1.73
1600° F (% F)	---	1.54	1.00	.99	.83	1.33	1.12
1700° F (% F)	---	.53	.40	.45	.02	1.14	.024
1800° F (% F)	.02	.21	---	---	---	.27	---

TABLE III

DETERMINATION OF EFFECT OF CONVERSION TEMPERATURE
ON FLUORINE CONTENT OF
BLACK OXIDES

Material: Ammonium Di-Uranate Prepared From C-Liner

	<u>EXPERIMENT NUMBER</u>				
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
pH of Precipitation	6.1	6.0	6.2	5.8	7.0
1500° F (%F)	1.02	1.12	2.16	1.41	4.86
1700° F (%F)	.62	.11	.48	.13	2.77

TABLE IV
THE RE-CYCLING OF CONTINUOUSLY AND "BATCH"
PRECIPITATED AMMONIUM DI-URANATE

Material: C-Liner

Assay: 1.50% U_3O_8

	<u>EXPERIMENT NUMBER</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample (gm)	500	500	500
Weight of H_2O (gm)	500	500	500
Weight of HCl (gm)	1320	1320	1320
Weight of $NaClO_3$ (gm)	1.0	1.0	1.0
Digest pH	0.0	0.0	0.0
Digest Temperature ($^{\circ}C$)	90	90	90
Digest Time (hr.)	1 1/2	1 1/2	1 1/2
Insoluble Residue (gm)	102	196	192
Insoluble Residue (% U_3O_8)	.035	.055	.015
Filtrate (% U_3O_8)	.40	.38	.44
Filtrate (% F)	.11	.14	.10
pH of Precipitation	6.0	7.0	7.0
Type of Precipitation	continuous	continuous	batch
Ammonium Uranate (% U_3O_8)	44.15	43.80	44.20
Ammonium Uranate (% F)	3.53	4.27	3.86
Calcination Temperature ($^{\circ}F$)	1500	1500	1500
Black Oxide (% F)	1.11	1.24	1.43
Weight of H_2O To Reslurry (gm)	1000	1000	1000
Weight of HCl (gm)	38.5	38.5	38.5
pH of Digestion	1.0	1.15	.98
Time of Digestion (hr.)	1/2	1/2	1/2
Feed Liquor (% U_3O_8)	1.03	1.10	1.07
Feed Liquor (% F)	.17	.21	.24
pH of Precipitation	6.0	7.0	7.0
Type of Precipitation	continuous	continuous	batch
Ammonium Uranate (% U_3O_8)	63.0	60.96	61.20
Ammonium Uranate (% F)	.78	.64	.83
Calcination Temperature ($^{\circ}F$)	1500	1500	1500
Black Oxide (% U_3O_8)	70.16	72.24	71.50
Black Oxide (% F.)	.02	traces	.10

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TABLE V
DETERMINATION OF OPTIMUM pH FOR SULFURIC ACID DIGESTION

Material: BFC-6

Assay: 14.66% U_3O_8

Preparation: Calcined at 1000° F for One Hour

	<u>EXPERIMENT NUMBER</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample (gm)	100	100	100
Calcination Time (hr.)	1.0	1.0	1.0
Weight of H_2O (gm)	400	400	400
Weight of H_2SO_4 (gm)	92.0	62.56	29.5
Weight of $NaClO_3$ (gm)	5.0	6.7	1.0
pH of Digestion	0.0	0.5	1.0
Time of Digestion	2	2	2
Temperature of Digestion	90	90	90
Insoluble Residue (gm)	59.6	67.3	70.8
Insoluble Residue (% U308)	.28	.35	.525
Recovery (%)	98.9	98.4	97.5

TABLE VI
DETERMINATION OF OPTIMUM TIME OF SULFURIC ACID DIGESTION

Material: BFC-6

Assay: 14.66% U_3O_8

Preparation: Calcined at 1000° F For One Hour

	<u>EXPERIMENT NUMBER</u>				
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
Weight of Sample (gm)	100	100	100	100	100
Weight of H_2O (gm)	400	400	400	400	400
Weight of H_2SO_4 (gm)	41.7	92.0	73.6	86.48	101.2
pH of Digestion	0.1	0.0	0.1	0.1	0.1
Time of Digestion (hr)	1.0	2.0	3.0	4.0	5.0
Temperature of Digestion (°C)	90	90	90	90	90
Insoluble Residue (gm)	64.9	67.3	63.5	66.1	65.2
Insoluble Residue (% U308)	.45	.35	.365	.175	.105
Recovery (%)	98.1	98.5	98.4	99.3	99.5

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TABLE VII

DETERMINATION OF OPTIMUM CALCINATION TEMPERATURE OF MATERIAL
FOR SULFURIC ACID DIGESTION

Material: BFC-6

Assay: 14.66% U_3O_8

Digest Conditions: Three Hours at 90° C at pH 0.0 - 0.1

EXPERIMENT NUMBER

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
Weight of Sample (gm)	100	100	100	100	100
Weight of H_2O (gm)	400	400	400	400	400
Weight of H_2SO_4 (gm)	80.9	84.6	84.6	80.9	84.6
Weight of $NaClO_3$ (gm)	13.2	12.3	13.6	8.5	4.2
Calcination Temperature (°F)	800	900	1000	1100	1200
Calcination Time (hr.)	1.0	1.0	1.0	1.0	1.0
Insoluble Residue (gm)	65.0	65.7	65.0	62.5	64.7
Insoluble Residue (% U_3O_8)	.126	65.7	65.0	62.5	64.7
Insoluble Residue (% U_3O_8)	.126	.168	.28	.385	.385
Recovery (%)	99.4	99.3	98.8	98.5	98.6

TABLE VIII

DETERMINATION OF OPTIMUM TIME OF DIGESTION AS CORRELATED TO
CALCINATION TEMPERATURE

Material: BFC-6

Assay: 14-66% U_3O_8

Preparation: Calcined at 800° F For One Hour

Digest Condition: 90° C at pH 0.0 - 0.1

	<u>EXPERIMENT NUMBER</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample (gm)	100	100	100
Weight of H_2O (gm)	400	400	400
Weight of H_2SO_4 (gm)	75.4	71.9	74.3
Time of Digestion (hr.)	4	5	6
Insoluble Residue (gm)	66.2	66.1	66.5
Insoluble Residue (% U_3O_8)	*.21	*.26	*.175
Recovery (%)	99.0	98.9	99.2

* No explanation for apparent deviation of these results.
Additional confirming experiments are planned.

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