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NYO-1159

PROBLEMS OF REFINING URANIFEROUS RESIDUES

Vitro Manufacturing Company

H. Fleck and J. E. Summers

June 15, 1953

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PROBLEMS OF REFINING URANIFEROUS RESIDUES

TECHNOLOGY - FEED MATERIALS

A. SUMMARY

GENERAL

1. Scope of Program

As of May 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 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
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
NYO-1157	March	1953 - " " No. 23
NYO-1158	April	1953 - " " No. 24

PROJECT STATUS

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

Experiments were continued on FMPC Sump Cake using mineral acid digestion to determine if uranium could be extracted from this material resulting in insoluble discard residues containing less than .05% U_3O_8 . Experiments were conducted which included calcination of raw material prior to acid digestion. Preliminary results indicated that this treatment will lower insoluble uranium levels to less than the maximum specification of .05% U_3O_8 .

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4. Research Project - Refining of BFC-6

The investigation of BFC-6 with regard to ways and means of reducing the insoluble uranium level to less than .05% U_3O_8 using mineral acid digest techniques was continued. An investigation concerning the influence of calcination time on optimum recovery of uranium values was initiated. Preliminary data indicates that time of calcination is not a factor on optimum recovery.

A series of experiments was conducted to determine if the use of excess concentrations of sulfuric acid during digestion would lead to satisfactory extraction of insoluble uranium. Preliminary results indicate that the use of acid concentrations up to five molar has no appreciable beneficial effect with regard to lowering uranium content of insoluble residues to the discard level.

Experiments were initiated using hydrochloric acid and nitric acid to determine if these reagents would be superior to sulfuric acid as a digestant. Preliminary data indicates that these acids will be no more effective than sulfuric acid in lowering uranium content of discard residues to within specifications.

Preliminary experiments were initiated during the present report period to determine the effect of particle size of the raw material on maximum recovery. Experiments were conducted on the raw material over a range from 100 - 325 mesh.

Preliminary experimentation was initiated to determine what would be the most satisfactory means of extracting uranium from a clarified sulfuric acid digest filtrate resulting from the digestion of BFC-6. Ammonium precipitation of solubilized uranium followed by sodium hydroxide digestion of precipitated ammonium salts was investigated as a possible procedure.

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

Extensive experiments were conducted on the crude uranyl ammonium phosphate residue resulting from

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the purification of Vitro Sodium Carbonate Residues to determine optimum means of further purifying this material.

Purification of the crude UAP residue by sodium hydroxide digestion was investigated. Preliminary experiments indicate that this method will not be altogether satisfactory in obtaining an acceptable end product.

Digestion of the crude residue in sulfuric acid, removal of acid insolubles by filtration, removal of solubilized contaminants by neutralization of excess acid by sodium carbonate, followed by precipitation of uranium from the carbonate filtrate by the addition of sodium hydroxide was investigated as a possible method for refining this residue. A satisfactory end-product may be obtained in this manner.

Experiments were completed on the refining of the crude UAP residue which indicated that sodium carbonate digestion of the UAP precipitate, removal of insoluble contaminants by filtration followed by precipitation of uranium from the carbonate filtrate with sodium hydroxide would probably be the most satisfactory means of purifying this residue.

Repulp washing of the insoluble carbonate residue resulting as a by-product of this process, will lower the uranium level to less than maximum specifications for a discard residue.

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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 FMPC Sump Cake, Using
Mineral Acid Digestion and Ammonium Precipitation

Previous experimentation on FMPC Sump Cake had indicated that a mineral acid digestion of this material would not reduce insoluble uranium content to less than the maximum specification for discard residue of .05% U_3O_8 . Experiments were conducted in which samples of this material were calcined at 1200 - 1400° F for one hour prior to hydrochloric or sulfuric acid digestion. Results from experiments of this nature indicated that calcination of the raw material would be effective in reducing insoluble uranium content of the acid residue to less than .05% U_3O_8 . Fifty gram samples of FMPC Sump Cake were calcined at 1200 - 1400° F for one hour and slurried in 200 ml of water. Hydrochloric acid and sulfuric acid were used as digest media. Sodium chlorate was utilized as an oxidizing agent. Digest of the calcined material was carried out for three hours at 90° C. Acid insolubles were separated from the digest slurry by filtration and analyzed for uranium content. Uranium content of the discard residue averaged less than .045%. On this basis the procedure

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outlined may be considered satisfactory. No further experimentation on this material is considered necessary at this time.

Average data may be found in Table I.

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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 by the Use of Mineral Acid Digestion

Experimentation was continued on BFC-6 during the present report period. Optimum conditions necessary to extract uranium from this material using mineral acid digest procedures leading to acid insoluble residues containing less than .05% U_3O_8 were sought. As indicated by previous experimentation ordinary procedures would not be satisfactory in achieving satisfactory recovery of uranium from this material.

Experiments were conducted to determine if inadequate calcination of the raw material was a factor leading to unsatisfactory extraction. One hundred gram samples of the water-leached, raw material were calcined at 1000° F. for periods of time from 1 - 4 hours. Calcined samples were slurried in 300 ml of water and digested in the presence of sulfuric acid at pH 0.0 with sodium chlorate to oxidize for 6 hours at 95 - 100° C. Acid insolubles were separated from the digest slurry by filtration and washed with water until colorless. The acid insoluble residues were examined for uranium content and were found to contain uranium values considerably in excess of the maximum specification of .05% U_3O_8

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for a discard residue. Apparently longer periods of calcination time will not be effectual in further reducing uranium content to an acceptable level. No further experiments along this line are planned.

Representative data may be found in Table II.

A series of experiments was conducted to determine if the use of excess quantities of sulfuric acid during digestion would effectively solubilize maximum amounts of uranium. Acid concentrations ranging from 1 to 5 molar in excess acid after equilibrium, were used as a digest media. One hundred gram samples of the water leached material were calcined at 1000° F. for one hour and slurried in 300 ml of water. Sulfuric acid was added in molar concentrations ranging from 1 - 5 after equilibrium was attained. Sodium chlorate was used as an oxidizing agent during digestion. Acid insolubles resulting from this treatment were separated from the digest slurry by filtration and washed with water until colorless. An examination of the acid insoluble residue indicated no further reduction of insoluble uranium. The uranium content of acid insoluble residues remained well in excess of .05%.

Representative data may be found in Table III.

The possibility of using either hydrochloric acid or nitric acid as a digest reagent with this material was investigated. One hundred gram samples of the raw material were calcined at 1000° F. for one hour prior to digestion. Samples were slurried in 300 ml of water and digested in hydrochloric acid and nitric acid respectively in the presence of sodium chlorate as an oxidizing agent for periods of time approximating six hours at 95 - 100° C. Acid insolubles were removed by filtration and analyzed for uranium content. No further increase in efficiency of extraction of uranium from this material was noted using this procedure. Since the use of these acids is not completely desirable with regard to this material, no further experimentation of this nature is indicated.

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Representative data may be found in Table IV.

Experiments were conducted to assay the influence of particle size of the raw material on optimum extraction of uranium using sulfuric acid digest procedures. Samples of BFC-6 were graded from 100 to less than 325 mesh and calcined at 1000° F. for one hour. Samples were slurried in 300 ml of water and digested in sulfuric acid at pH 0.0, in the presence of sodium chlorate as an oxidizing agent, for periods of time approximating 6 hours at 95 - 100° C. Acid insolubles from these experiments were separated from the digest slurry by filtration and washed with water until colorless. Analysis for uranium content indicated no reduction in the insoluble uranium level due to a finely divided particle.

Representative data may be found in Table V.

Preliminary experiments designed to determine a satisfactory means of obtaining an end product from a clarified sulfuric acid digest filtrate resulting from digestion of BFC-6 were initiated. Pregnant liquor resulting from sulfuric acid digestion of calcined BFC-6 was neutralized to pH 9.5 with ammonium hydroxide and digested for one hour at 90° C. to remove solubilized copper. The ammonium salts were separated by filtration and washed with water until colorless. Ammonia salts resulting from this treatment were slurried in water and digested in a 10% sodium hydroxide solution to remove precipitated tin as soluble sodium stannate. The residue was separated from the digest filtrate by filtration and analyzed for uranium content. The end product obtained in this manner approximated 61.00% U_3O_8 .

Additional experiments were conducted in which this procedure was reversed. Pregnant acid filtrates resulting from BFC-6 digestion in sulfuric acid were neutralized to pH 11.0 using sodium hydroxide. The insoluble residue was removed by filtration and washed

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with water until colorless. This residue was slurried in water and dissolved in sulfuric acid at pH 1.0. Ammonium was added to pH 9.5 to remove copper as soluble copper hydroxide. Ammonia salts were collected by filtration and washed until color-free. Analysis of the residue indicated an end product containing approximately 60.5% U_3O_8 . Additional experiments are planned to confirm results.

Preliminary data may be found in Table VI.

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D. REFINING AND PROCESSING OF SODIUM CARBONATE
RESIDUES

Research Project No: 5

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

8. The Purification of the Crude Uranyl Ammonium Phosphate
Precipitate Obtained by the Refining of Vitro Sodium Car-
bonate Residues

Samples of a crude uranyl ammonium phosphate residue obtained by a pilot-level refining of Vitro sodium carbonate residues were used for experimentation. Uranium content of the residue was determined at 10.6% U_3O_8 . Several procedures and methods were attempted to establish the most feasible and economic means of obtaining an end product of high uranium and low phosphate content by further purification of this material.

The benefit of sodium hydroxide digestion were evaluated with regard to further purification of the crude uranyl ammonium phosphate residue. Fifty gram samples of the crude residue were slurried in 200 ml of water and digested in the presence of fifty grams of commercial sodium hydroxide for one hour at 90° C. In this manner precipitated aluminum and residual P_2O_5 were removed. The resultant residue was separated from the digest slurry by decantation and filtration. Average analysis of the end product obtained in this manner averaged 22.5% U_3O_8 . This method probably could not be generally considered an acceptable

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means of purification. Its effectiveness would depend upon the proportionate amounts of alkaline earths, iron and aluminum present in the feed material. These values are known to fluctuate considerably. Since the treatment will apparently yield a product of relatively low and uncertain uranium content, further experimentation or investigation of this procedure is not planned.

Representative data may be found in Table VII.

A procedure involving sulfuric acid digestion of the UAP residue, oxidation with sodium chlorate, removal of acid insolubles by filtration, removal of contaminants by neutralization of excess acid with sodium carbonate followed by precipitation of uranium from the clarified carbonate filtrate by the addition of sodium hydroxide was considered. Fifty gram samples of the crude residue were slurried in 200 ml of water and digested in sulfuric acid at pH 0.0 for three hours at 90° C. in the presence of sodium chlorate as an oxidizing agent. Acid insolubles were removed by filtration and the clarified acid filtrate neutralized with sodium carbonate. The carbonate slurry was digested for two hours at 60° C and filtered to remove alkaline earth and R_2O_3 carbonates. The clarified carbonate filtrate was treated with 2% (W/V) sodium hydroxide and the resultant precipitate collected by filtration. The end product obtained in this manner averaged 75% U_3O_8 and contained less than 1.0 P_2O_5 . The procedure as outlined comprises the normal Vitro procedure of purification for residues of this type. There is little doubt that it would be effective in producing an acceptable end product.

Representative data may be found in Table VIII.

Direct sodium carbonate digestion of the crude UAP residue was considered. Fifty gram samples of the crude residue were slurried in 300 ml of a 10% (W/V) sodium carbonate solution. Digestion was carried out at 90 - 95° C for a period of three hours. The

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insoluble residue was removed by filtration and washed until color-free using a hot 5% (W/V) sodium carbonate solution. The carbonate residue when analyzed was found to contain approximately .15% U_3O_8 . Since this value would exceed maximum specifications for a discard residue, the carbonate residue was re-slurried in 100 m. of a 5% (W/V) sodium carbonate solution and digested for two hours at 90 - 95° C. The insoluble residue was again collected by filtration and washed until color-free using a hot 5% (W/V) sodium carbonate solution. Investigation of the uranium content of the carbonate residue indicated an average of less than .02% U_3O_8 . This value is well below the maximum specification of .05% U_3O_8 for a discard residue. The clarified filtrates from both treatments were combined with the wash water and uranium precipitated by the addition of 2% (W/V) sodium hydroxide. The resultant precipitate was analyzed and found to contain an average of 75 - 80% U_3O_8 . P_2O_5 content was less than .5%. In general this appears to be the most economical and the most satisfactory procedure for upgrading the crude UAP precipitate. Additional experiments are planned to confirm the preliminary data.

Average data may be found in Table IX.

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EXHIBITS

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TABLE I

EXTRACTION OF URANIUM FROM FMPC SUMP CAKE

Material: "FMPC" Sump Cake

Assay: 14.7% U_3O_8

	<u>EXPERIMENT NUMBER</u>	
	<u>1</u>	<u>2</u>
Calcination Temperature ($^{\circ}F$)	1300	1300
Calcination Time (hr.)	1.0	1.0
Weight of Sample (gm.)	50	50
Weight of H_2O (gm.)	200	200
Acid Used	HCl	H_2SO_4
Weight of Acid (gm.)	115	65
Digest Time (hr.)	3.0	3.0
Digest pH	0.0	0.0
Digest Temperature ($^{\circ}C$)	90	90
Acid Insoluble (gm)	16.5	36.5
Acid Insoluble (% U_3O_8)	.045	.035
Recovery (%)	98.9	98.3

TABLE II

EFFECT OF CALCINATION TIME OF RAW MATERIAL ON THE
EXTRACTION OF URANIUM USING H_2SO_4

Material: BFC-6
Assay: 19.6% U_3O_8

	<u>EXPERIMENT NUMBER</u>			
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Weight of Sample (gm.)	100	100	100	100
Calcination Temperature ($^{\circ}\text{F}$)	1000	1000	1000	1000
Calcination Time (hr.)	1.0	2.0	3.0	4.0
Weight of H_2O (gm.)	300	300	300	300
Weight of H_2SO_4 (gm.)	82.80	82.80	73.60	69.92
Weight of NaClO_3 (gm.)	6.8	5.3	6.3	6.1
Digest Time (hr.)	3.0	3.0	3.0	3.0
Digest Temperature ($^{\circ}\text{C}$)	90	90	90	90
Digest pH	0.1	0.1	0.1	0.1
Acid Insolubles (gm.)	64.4	65.0	63.3	63.2
Acid Insolubles (% U_3O_8)	.42	.51	.45	.43
Total Recovery (%)	98.6	98.3	98.5	98.7

TABLE III

EFFECT OF EXCESS ACID ON THE EXTRACTION OF URANIUM FROM BFC-6

Material: BFC-6
Assay: 19.6% U_3O_8

	<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
Calcination Temperature (°F)	1000	1000	1000	1000	1000
Weight of H_2O (gm.)	300	300	300	300	300
H_2SO_4 Concentration (molar)	1.0	2.0	3.0	4.0	5.0
Digest Time (hr.)	6.0	6.0	6.0	6.0	6.0
Digest Temperature (°C)	95-100	95-100	95-100	95-100	95-100
Acid Insolubles (gm.)	63.2	59.3	57.5	56.7	54.5
Acid Insolubles (% U_3O_8)	.43	.38	.19	.40	.18
Recovery (%)	98.7	98.9	99.4	98.9	99.5

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TABLE IV
EXTRACTION OF URANIUM FROM BFC-6 USING HCl OR HNO₃

Material: BFC-6
Assay: 19.6% U₃O₈

	<u>EXPERIMENT NUMBER</u>	
	<u>1</u>	<u>2</u>
Weight of sample (gm.)	100	100
Calcination Temperature (°F)	1000	1000
Weight of H ₂ O	300	300
Acid Used	HCl	HNO ₃
Weight of Acid (gm.)	107	88.1
Digest Time (hr.)	6.0	6.0
Digest Temperature (°C)	95	95
Digest pH	0.1	0.0
Acid Insolubles (gm.)	54.2	68.2
Acid Insolubles (% U ₃ O ₈)	.26	.43
Recovery (%)	99.3	98.5

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TABLE V
EFFECT OF PARTICLE SIZE ON THE EXTRACTION OF URANIUM USING H₂SO₄

Material: BFC-6
Assay: 19.6% U₃O₈

	<u>EXPERIMENT NUMBER</u>			
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Weight of Sample (gm.)	100	100	80	70
Particle Size (mesh)	+100	-100	-200	-325
Calcination Temperature (°F)	2000	1000	1000	1000
Weight of H ₂ O (gm.)	300	300	300	300
Weight of H ₂ SO ₄ (gm.)	75.5	55.20	68.08	60.72
Weight of NaClO ₃	9.0	5.8	6.5	6.5
Digest pH	0.1	0.1	0.0	0.0
Digest Time (hr.)	6.0	6.0	6.0	6.0
Digest Temperature (°C)	95	95	95	95
Acid Insolubles (gm.)	71.5	60.9	49.7	36.3
Acid Insolubles (% U ₃ O ₈)	.12	.29	.52	.29
Recovery (%)	99.5	99.1	98.4	99.2

TABLE VI

EXTRACTION OF URANIUM FROM AN H_2SO_4 DIGEST
FILTRATE OF BFC-6

Material: BFC-6
Assay: 3.2% U_3O_8

	<u>EXPERIMENT NUMBER</u>	
	<u>1</u>	<u>2</u>
Volume of Liquor (ml)	300	300
pH of Precipitation	9.5	11.0
Precipitant	NH_4OH	$NaOH$
Weight of Precipitant (gm.)	97	33.3
Digest Time (hr.)	1.0	1.0
Digest Temperature ($^{\circ}C$)	90	90
Residue (gm.)	24.2	22.3
Residue (% U_3O_8)	51.52	39.36
Weight of H_2O to Slurry (gm.)	250	300
Weight of H_2SO_4 (gm.)	-	47.8
Digest pH	-	1.0
Digest Reagent	$NaOH$	NH_4OH
Weight of Reagent	30.0	120
pH of Digestion	-	9.5
Digest Time (hr.)	1.0	1.0
Digest Temperature ($^{\circ}C$)	90	90
Residue (gm.)	19.5	17.2
Residue (% U_3O_8)	60.76	61.04

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TABLE VII
PURIFICATION OF CRUDE UAP BY SODIUM HYDROXIDE DIGESTION

Material: UAP Precipitate
Assay: 10.6% U_3O_8

	<u>EXPERIMENT NUMBER</u>	
	<u>1</u>	<u>2</u>
Weight of Sample	50	50
Weight of H_2O (gm.)	200	200
Weight of $NaOH$ (gm.)	50	50
Digest Time (hr.)	1.0	1.0
Digest Temperature	90	90
Residue (gm.)	23.5	23.8
Residue (% U_3O_8)	22.42	22.67

TABLE VIII

PURIFICATION BY H_2SO_4 DIGESTION, Na_2CO_3 AND
NaOH PRECIPITATION

Material: Crude UAP
Assay: 10.6% U_3O_8

	<u>EXPERIMENT NUMBER</u>	
	<u>1</u>	<u>2</u>
Weight of Sample (gm.)	50	50
Weight of H_2O (gm.)	200	200
Weight of H_2SO_4	33.0	33.0
Digest pH	0.25	0.20
Digest Time (hr.)	3.0	3.0
Acid Insolubles (% U_3O_8)	.035	.05
Acid Insolubles (gm.)	3.0	3.2
Weight of Na_2CO_3 (gm.)	35	35
Digest Time (hr.)	2.0	2.0
Digest Temperature ($^{\circ}\text{C}$)	60	60
Residue (gm.)	44.0	45.0
Residue (% U_3O_8)	.24	.21
Residue after retreatment (% U_3O_8)	.046	.035
NaOH added (gm.)	8.0	8.0
Precipitate (% U_3O_8)	77.56	76.24
Precipitate (gm.)	6.2	6.5

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TABLE IX

PURIFICATION BY Na_2CO_3 DIGESTION AND
NaoH PRECIPITATION

Material: Crude UAP
Assay: 10.6% U_3O_8

	<u>EXPERIMENT NUMBER</u>	
	<u>1</u>	<u>2</u>
Weight of Sample (gm.)	50	50
Weight of H_2O (gm.)	300	300
Weight of Na_2CO_3 (gm.)	30	30
Digest Time (hr.)	3.0	3.0
Digest Temperature ($^{\circ}\text{C}$)	90	90
Residue (gm.)	40.0	40.0
Residue (% U_3O_8)	.17	.14
Weight of H_2O to Re-slurry residue (gm.)	100	100
Weight of Na_2CO_3 (gm.)	10	10
Digest Time (hr.)	2.0	2.0
Digest Temperature ($^{\circ}\text{C}$)	90	90
Residue (% U_3O_8)	.017	.023
NaoH Added to Filtrate (gm.)	8.0	8.0
Residue (gm.)	6.5	6.6
Residue (% U_3O_8)	76.46	77.86

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