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NYO-1152
PROBLEMS OF REFINING URANIFEROUS RESIDUES
TECHNOLOGY FEED MATERIALS
Progress Report No. 18

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NYO-1152
Page: 2

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PROBLEMS OF REFINING URANIFEROUS RESIDUES

TECHNOLOGY FEED MATERIALS

A. SUMMARY

GENERAL

1. Scope of Program

As of October 1, 1952, 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 in 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 miscellaneous uranium bearing materials, to reduce the U3O8 content of the residues below 0.05%.

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2. Previous Reports

NYO-1139	September 1951	- Progress Report No. 5
NYO-1140	October 1951	- " No. 6
NYO-1141	November 1951	- " No. 7
NYO-1142	December 1951	- " No. 8
NYO-1143	January 1952	- " No. 9
NYO-1144	February 1952	- " No. 10
NYO-1145	March 1952	- " 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

PROJECT STATUS

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

Development work was initiated on certain high grade feed materials, notably C-2,, Harshaw Press Cake, C-35, DC-22, Reactor A B and Graphite Ash, to determine the feasibility of processing these and other high grade feed materials to a black oxide rather than sodium di-uranate, presently the end product now obtained from the Vitro purification process.

The process under consideration is similar to the one which has been developed for C-oxide. The procedure consists of hydrochloric acid digestion, oxidation during digestion by the addition of sodium chlorate, the removal of acid insolubles by filtration, the precipitation of ammonium di-uranate from the clarified filtrate by the addition of either dilute or concentrated ammonia, using either batch or continuous precipitation techniques as the circumstance demands. After removal of the uraniferous precipitate by filtration, black oxide is obtained by calcination at 800° C. It is hoped that in this manner a product may be obtained from high grade material which will be satisfactory as regards uranium and fluorine content.

Benefits accruing from the possible adaptation of high grade feed materials to a treatment of this nature would be the establishment of a universal process for the purification of most by-product residues, possible reduction of chemical costs and possible release of production facilities which could be used to process additional materials leading to a substantially lower unit overhead cost.

Primary experiments of this nature were conducted on Harsnaw Press Cake and C-27. The combined weight of these two residues constitute the largest proportion of the total weight of high grade feed materials now being processed. Ammonium di-uranate was obtained from a clarified hydrochloric acid digest filtrate of the residues by precipitation at various pH levels, using both dilute and concentrated ammonia and batch techniques of precipitation. Consideration of the end product before and after calcination at high temperature readily illustrates that in order to obtain an end product within fluorine specifications, it will be necessary to fire the ammonium salt to black oxide. In addition, it was immediately determined that filtration of the acid insoluble residue, resulting from a hydrochloric acid digest of Harshaw Press Cake, would be extremely difficult. However, satisfactory results were obtained by using various commercial filter aids.

Preliminary experiments were also initiated in which the same procedure of HCl digestion, followed by ammonia salt precipitation, was applied to other high grade feed materials such as DC-22, Reactor Ash, Graphite Ash and C-35. Insofar as C-35 is concerned, the process appears to be entirely satisfactory. The filtration of acid insolubles resulting from digestion of graphite ash is somewhat unsatisfactory but can be expedited by using filtering aids. Acid insolubles resulting from digestion of DC-22 contain uranium values in excess of purity specifications indicating that longer periods of digestion will be required. The end product obtained from processing the materials was of satisfactory purity with the exception of that obtained from reactor ash. It is apparent that reactor ash contains fluorine in such excessive amounts that quantitative precipitation by ammonia is not possible. However, since reactor ash constitutes a proportionately small amount of the total volume of high grade material, it may be possible the problem can be resolved by blending with other material prior to digestion.

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

Final results of the Pilot Operation concerning the purification of BFC-6 using the sodium carbonate-autoclave leach have been collected and evaluated. Complete statistical data concerning the efficiency of recovery and economic factors is presented. No additional experiments of a pilot nature are contemplated unless appropriate, improved equipment may be obtained. A summation report covering laboratory and pilot operations is being prepared. The project has been discontinued.

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

A new composite sample of the Vitro stockpile of low grade sodium carbonate residues averaging .26% U3O8 was prepared in order to further evaluate the feasibility of adapting uranyl ammonium phosphate recovery techniques to the entire stockpile. Portions of the new sample were processed under optimum conditions previously established, and uranium was recovered as uranyl ammonium phosphate from the resultant pregnant liquor. Statistical data obtained in this manner coincides with data already accumulated from previous experimentation, thus indicating that there is every probability that the majority of the Vitro Stockpile will adapt to UAP recovery processes.

Additional experiments were conducted concerning the extraction of uranium from ammonia salts, precipitated from the acid digest filtrate of Vitro sodium carbonate residues, by digestion of the ammoniacal precipitate in the presence of sodium carbonate. The data obtained confirmed that uranium may be successfully extracted in this manner leaving residues for discard containing less than .05% U3O8. Comparative experiments were also run concerning direct carbonation of the filtrate resulting from acid digestion of the Vitro residues which resulted in unsuccessful extraction. In order to determine why carbonate digestion of the ammonia salt leads to successful extraction and direct carbonation of the acid filtrate does not a study of slurry density of the precipitate and precipitate particle characteristics was conducted during which the material was centrifuged and microscopically examined. It may be seen by this study that a combination of one or more factors could easily account for failure or success.

Samples were obtained from a Vitro stockpile of sodium carbonate residues which are a by-product of pitchblende purification. Assay of the sample was .7% U3O8. Preliminary experiments were initiated designed to investigate various digestive procedures for the successful extraction of the contained uranium values by using hydrochloric acid, sulfuric acid and sodium carbonate as the digest media. Both uncalcined and calcined material were processed. Chemical characteristics were generally similar to process data obtained using other pitchblende residues.

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

Research Project: No. 1

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

6. Refining of High Grade Feed Materials

Considerable thought has been given to ways and means to increase production capacity and produce a substantially lower unit overhead cost. One means under consideration would be to adapt high grade feed materials to the process now in use for the refining of C-type raw materials. This process consists of hydrochloric acid digestion, oxidation by sodium chlorate, removal of the acid insolubles by filtration, and precipitation of ammonium di-uranate from the clarified acid filtrate by neutralization of excess acid with ammonium hydroxide. The precipitate formed, when collected by filtration and calcined at 800° C, is converted to a black oxide of high purity and low fluoride content. Accordingly, experiments were initiated to determine the feasibility of adapting all high grade feed materials to a procedure of this nature. It should be noted that the present purification process consisting of sulphuric acid digestion, removal of impurities by neutralization with sodium carbonate, followed by the precipitation of sodium di-uranate by the addition of sodium hydroxide, is a more diverse and positive system of purification as regards fluorides and miscellaneous contaminants. However, since the majority of the high grade feed materials are somewhat standardized, the possibility of converting some or part of these materials to a simpler procedure may be attractive from an economic standpoint. The possibility of obtaining low cost AEC procured hydrochloric acid would make the procedure attractive as far as a chemical cost is concerned. Preliminary experiments have

been concluded on six types of high grade feed material constituting the bulk of this type of material normally processed by the Corporation. Preliminary results are encouraging although it is apparent that additional experimentation will be necessary. Preliminary information as regards this procedure, when adapted to high grade feed material, may be summarized as follows:

C-27

The material used for experimentation contained 65.24% U3O8, the remainder being composed mostly of alkaline earth fluorides. After calcining the material at 1200 to 1400° F and pulverizing to approximately 85% -100 mesh, 500 gram samples were slurried in 2000 grams of water and digested in 15° Baume hydrochloric acid at pH 0.4 to 0.6 in the presence of .5% (w/v) sodium chlorate for three hours at 90° C. The insoluble residue resulting from the digest was removed by filtration and found to average about 21% of the original weight. In no instance did undissolved uranium values exceed .05% U3O8. Clarified filtrates from a digest of this nature were neutralized with both dilute and concentrated ammonium hydroxide at pH values ranging from 6.0 to 7.4. Precipitated ammonium salts were removed by filtration, washed until calcium and magnesium free, and calcined to a black oxide at 800° C. The product obtained from this procedure exceeded 95% U3O8 and in every instance contained less than .1% fluorine. The ammonia salt prior to calcination contained more than 2.5% F. There is little doubt that this material could be easily adapted to a process of this nature but additional experimentation is considered necessary to confirm the data obtained. Complete representative data may be found in Table I.

Harshaw Press Cake

This material plus C-27, at the moment, comprises the majority of the high grade feed materials now being processed. Uranium content of the material usually fluctuates between 20-65% U3O8. Experimental samples contained 24.42% U3O8. Samples were dried, calcined and pulverized before digestion. 500 gram portions were slurried in 2000 grams of water and digested in 15° Baume hydrochloric acid at low pH values for

three hours at 90° C in the presence of .5% (v/v) sodium chlorate. Attempted removal of the acid insolubles by filtration immediately indicated that filtration without the use of filter aids would be almost impossible. The acid insolubles formed were of an exceedingly slimy nature. Experiments were conducted in which standard Supercel and other Celite filter aids were used and it is now indicated that filtration of the insoluble residue will be possible in the presence of these aids. Clarified filtrates obtained through the use of filter aids were neutralized using dilute and concentrated ammonium over a range extending from pH 6.0 to pH 7.0. The resultant salts were removed by filtration and fired to a black oxide at 800° C. Uranium content of the product exceeded 90% U3O8 and fluoride content was less than .1%. Again, the ammonia salt, prior to calcination, contained excessive quantities of fluorine. In addition, the residue contained approximately 1.25% P2O5 which may or may not be a problem. At this time the purity specifications for the Vitro product do not specify limitations on phosphate content. Additional experiments are indicated to confirm the data obtained. Preliminary data may be found in Table II.

C-35

Samples of this material containing approximately 69% U3O8 were treated in a similar manner. Normal range of uranium content is from 30-70% U3O8. After calcining and pulverizing to -100 mesh, 500 gram portions were slurried in water at a ratio of 4:1 and digested in 15° Baume hydrochloric acid in the presence of .5% (w/v) sodium chlorate at temperatures in excess of 90° C for three hours. Acid insolubles were easily removed by filtration and were found to average approximately 11% of the original bulk. Uranium content of the insoluble residue was less than .05% U3O8. Ammonium salts which were precipitated from the pregnant liquor at pH values in excess of 6.0, using dilute and concentrated ammonium, contained uranium in excess of 82% U3O8, and averaged 2.0% fluorine. However, calcination of the ammonia salts to black oxide at 800° C elevated uranium levels to an average of 90% U3O8 and reduced fluorine content to less than 0.1%. Data may be found in Table III.

DC-22

Normal uranium content of this material is widely variable ranging from 15 to 50% U3O8. The sample used exceeded 48%. This material was processed by digesting 500 gram samples of pulverized and calcined material in hydrochloric acid at appropriate pH values in the presence of sodium chlorate, the acid insolubles were removed by filtration and ammonium salts were precipitated from the clarified liquor by neutralizing excess acid with ammonium hydroxide. The product obtained from this procedure contained uranium and fluoride values well within purity specifications. Average analysis, after calcination to black oxide at 800° C, was 93% U3O8 and .05% F. However, the acid insoluble obtained from the acid digest, which represents approximately 24% of the original bulk, contained uranium values in excess of .5% U3O8. Apparently, longer periods of digest are indicated. Additional experiments shall be conducted to determine whether or not the uranium content of the acid insoluble may be reduced to a level less than .05% which would be within purity specifications for discard residues. Average data is presented in Table IV.

Reactor Ash

The usual variation of uranium content for this material is from 35-40% U3O8 but the sample used for experimentation exceeded 42%. To date it has not been possible to purify this material using hydrochloric acid digestive procedures followed by precipitation of ammonium di-uranate with ammonium hydroxide. The hydrochloric acid digest was satisfactory with acid insolubles readily removed by filtration, comprising approximately 47% of the original bulk and containing less than .05% U3O8. Unfortunately, the fluoride content of this material is so high that even in the presence of excess amounts of ammonium hydroxide the pregnant hydrochloric acid digest filtrate does not lend itself to a quantitative precipitation of uranium. Further experimentation is indicated and it may be possible that this material can be satisfactorily handled by a process of blending it in small amounts with larger volumes of other high grade feed materials. Preliminary data may be found in Table V.

Graphite Ash

The material processed is the residual ash resulting from reduction of the original material by prolonged burning. Samples used for experimentation exceeded 76.0% U3O8. No difficulty is foreseen in adapting this material to the hydrochloric acid digestive procedure although it is likely that it will be necessary to use filtering aids to expedite removal of the acid insolubles. 500 gram samples of the pulverized, calcined material were slurried in water at a ratio of 4:1 and digested in hydrochloric acid at low pH values in the presence of 0.5% (w/v) sodium chlorate for three hours at 90° C. Ammonium di-uranate was precipitated from the pregnant clarified liquor at pH values approaching neutrality by using concentrated ammonium hydroxide. The precipitate thus formed was removed by filtration and calcined to a black oxide at 800° C. The residue, when analyzed, was found to contain uranium values in excess of 96% U3O8 and less than .05% F. Data obtained to date may be found in Table VI.

C. DIGESTION AND REFINING OF BFC-6

Research Project: No. 4

Research Supervisor: J. E. Summers

PILOT AND LABORATORY OPERATIONS

7. Determination of the Efficiency of the Autoclave Refining Process

Final data has been accumulated on the pilot operation and evaluation of the efficiency of the process as now established has been completed. It is now apparent that total efficiency of the process has been impaired by the failure of the calcination cycle for raw material, due to the necessity of using obsolete equipment. It should be pointed out that if a cycle of desired efficiency is to be established as regards the extraction of contained uranium values from BFC-6, using the autoclave-sodium carbonate leach, that modern and appropriate equipment will have to be procured before standards of efficiency can be established on a production level comparable to the laboratory data. Final data is presented in Table VII, and a summation report is being prepared covering experimental work on the project including both pilot and laboratory operations.

D. REFINING AND PROCESSING OF SODIUM CARBONATE RESIDUES

Research Project: No. 5

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

8. Extraction of Uranium as Uranyl Ammonium Phosphate From Vitro's Sodium Carbonate Process Residues

A new composite sample of the entire stockpile of Vitro's sodium carbonate residues was prepared in order to more fully determine whether or not it seems possible to adapt the entire stockpile to a refining technique such as the extraction of uranyl ammonium phosphate. When portions of this new sample were processed under optimum conditions, already ascertained from previous experimentation, extraction of uranium was quantitative. In general, the technique used consisted of slurrying 100 gram samples in 200 grams of water, digestion in hydrochloric acid at approximately pH 0.8 to 0.9 without the use of oxidizing agents, followed by removal of the insolubles by filtration and precipitation of uranyl ammonium phosphate from the clarified acid digest filtrate in the presence of optimum quantities of monobasic ammonium phosphate. Data obtained from experiments conducted on the new sample was identical with the data already accumulated from experimentation on other samples. It appears that such a process of uranium extraction may be successfully applied to the entire stockpile. Representative data may be found in Table VIII.

9. Extraction of Uranium as a Soluble Carbonate From Ammonia Salts Precipitated From the Digest Liquor of Sodium Carbonate Processed Residues

Additional experiments were performed which indicate that uranium may be successfully extracted as a soluble carbonate by digesting an ammonia salt, precipitated from a pregnant acid filtrate resulting from hydrochloric acid digestion of Vitro sodium carbonate residues, in a 5% sodium carbonate solution. 100 gram samples were slurried in 200 ml of water and digested in hydrochloric acid at approximately pH 0.9. The acid insolubles were then removed by filtration and ammonia salts were precipitated from the clarified filtrate at pH 6.0 by neutralizing excess acid with ammonium hydroxide. The resultant precipitate was removed by filtration, washed and reslurried in H_2O . 5% (w/v) sodium carbonate was added and digestion was carried out at 90° C for periods of time up to two hours. The insoluble residue was removed by filtration, washed, dried and analyzed and was found to contain less than .05% U_3O_8 . At the same time that the preceding experiments were conducted parallel experiments were run involving direct carbonation of a pregnant clarified acid filtrate resulting from treatment of the Vitro carbonate residues. Carbonate residues were removed by filtration, washed and dried and in every instance were found to contain more than .1% U_3O_8 . Comparative data was obtained on precipitate-slurry density and a microscopic study of particle characteristics was completed. It is apparent from the data obtained that the insoluble carbonate residue resulting from direct carbonation has considerably more bulk than the ammonia salts. In addition, precipitate characteristics are somewhat different. The carbonate residue was found to be a medium sized, uneven particle interspersed with large agglutinated islands while the ammonia salt precipitate, after carbonate digestion, was found to be comprised of small, evenly divided and evenly dispersed particles. Complete experimental data may be found in Table IX.

10. Digestion of Pitchblende Carbonate Residues

The Vitro pitchblende carbonate residues have been roughly divided into two categories for experimental purposes. Prior experimentation has been concerned with the first group of pitchblende residues which assayed 1.6% U_3O_8 . The results of

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this experimentation have been detailed in a previous report. Preliminary experimentation was initiated on the second group which contains approximately 0.7% U3O8. Optimum digestive procedures were sought. Primary digests were carried out in the presence of hydrochloric acid, sulphuric acid and sodium carbonate using both calcined and uncalcined material. Experimental data indicates that there is little variation in chemical characteristics between the two groups of residues. Sulphuric acid was completely ineffectual in extracting uranium from the residue on a satisfactory basis. Acid insolubles remained in excess of .2% U3O8. Sodium carbonate was not an effective digest agent, leaving residues containing, on the average, 0.3% U3O8. Calcination of the raw material had no apparent beneficial effect upon extraction, however, hydrochloric acid was successful in extracting contained values, leaving insoluble residues assaying less than .05% U3O8. Additional confirming experiments are planned. Comparative data may be found in Table X.

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E X H I B I T S

TABLE I
PURIFICATION OF HIGH GRADE FEED MATERIAL

MATERIAL: C-27

ASSAY: 65-24% U_3O_8

MESH: 85% -100

	<u>Experiment No.</u>			
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Weight of Sample (gm)	500	500	500	500
Weight of H_2O (gm)	2000	2000	2000	2000
Weight of HCl (gm)	500	500	500	500
Calcination Temperature for Raw Material	1300	1300	1300	1300
Digest pH	0.65	0.60	0.43	0.40
$NaClO_3$ Used to Oxidize (gm)	1.5	1.5	1.5	1.5
Digest Time (hr.)	3	3	3	3
Digest Temperature ($^{\circ}C$)	90	90	90	90
Acid Insolubles (gm)	107	108	107	1065
Acid Insolubles (% U_3O_8)	.032	.050	.012	.032
Dilution of Precipitant (NH_4OH)	3:1	3:-	conc.	conc.
pH of Ammonia Salt Precipitation	6.0	7.0	6.2	7.4
Discard Effluent - % U_3O_8	.0007	.0001	.0005	traces
Ammonia Salts (% U_3O_8)	85.68	86.42	84.42	83.86
Ammonia Salts (%F)	2.65	2.43	2.60	2.84
Calcination Temperature for Ammonia Salts ($^{\circ}C$)	800	800	800	800
Black Oxide (% U_3O_8)	97.16	96.84	95.76	94.92
Black Oxide (%F)	.048	.056	.11	.20

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TABLE II
PURIFICATION OF HIGH GRADE FEED MATERIAL

MATERIAL: HARSHAW PRESS CAKE

ASSAY: 24.42% U_3O_8

MESH: 85% -100

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample (gm)	500	500	500
Weight of H_2O (gm)	2000	2000	2000
Weight of HCl (gm)	390	390	390
Calcination Temperature for Raw Material ($^{\circ}C$)	1300	1300	1300
$NaClO_3$ Used to Oxidize (gm)	1.25	1.25	1.25
Digest pH	0.55	0.65	0.05
Digest Time (hr)	3	3	3
Digest Temperature ($^{\circ}C$)	90	90	90
Acid Insolubles (% U_3O_8)	.21	.20	.055
Dilution of Ammonia Used to Precipitate	3:1	conc.	conc.
pH of Precipitation	6.0	6.2	7.0
Discard Effluent (% U_3O_8)	.0003	.0001	.0001
Ammonia Salts (% U_3O_8)	86.80	85.86	85.60
Ammonia Salts (% F)	1.13	1.24	1.18
Calcination Temperature for Ammonia Salts ($^{\circ}C$)	800	800	800
Black Oxide (% U_3O_8)	95.76	95.20	94.89
Black Oxide (% F)	.012	.025	.020

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TABLE III
PURIFICATION OF HIGH GRADE FEED MATERIAL

MATERIAL: C-35

ASSAY: 68.79% U_3O_8

MEAN: 85% -100

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample (gm)	500	500	500
Weight of H_2O (gm)	2000	2000	2000
Weight of HCl (gm)	555	555	550
Calcination Temperature for Raw Material ($^{\circ}C$)	1300	1300	1300
$NaClO_3$ Used to Oxidize (gm)	1.75	1.75	1.75
Digest pH	0.35	0.55	0.20
Digest Time (hr)	3	3	3
Digest Temperature ($^{\circ}C$)	90	90	90
Acid Insolubles (% U_3O_8)	.050	.10	.035
Acid Insolubles (gm)	60	62	58.5
Dilution of Precipitant (NH_4OH)	3:1	conc.	conc.
pH of Precipitation	6.0	6.0	6.0
Discard Effluent (% U_3O_8)	.0005	.0007	.0002
Ammonia Salts (% U_3O_8)	82.60	83.42	81.68
Ammonia Salts (% F)	1.53	1.50	1.68
Calcination Temperature for Ammonia Salts ($^{\circ}C$)	800	800	800
Black Oxide (% U_3O_8)	90.16	90.20	90.02
Black Oxide (% F)	.009	.050	.034

TABLE IV
PURIFICATION OF HIGH GRADE FEED MATERIAL

MATERIAL: DC-22

ASSAY: 48.37% U_3O_8

MESH: 85% -100

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample (gm)	500	500	500
Weight of H_2O (gm)	2000	2000	2000
Weight of HCl (gm)	832	832	832
Calcination Temperature for Raw Material ($^{\circ}C$)	1300	1300	1300
$NaClO_3$ Used to Oxidize (gm)	2.0	2.0	2.0
Digest pH	0.50	0.65	0.12
Digest Time (hr)	3	3	3
Digest Temperature ($^{\circ}C$)	90	90	90
Acid Insolubles (gm)	120	118	123
Acid Insolubles (% U_3O_8)	0.805	0.51	0.42
Dilution of Precipitant (NH_4OH)	3:1	conc.	conc.
pH of Precipitation	6.0	6.10	7.0
Discard Effluent (% U_3O_8)	.0007	.0002	traces
Ammonia Salts (% U_3O_8)	80.92	79.80	80.21
Ammonia Salts (% F)	1.80	2.25	2.40
Calcination Temperature for Ammonia Salts ($^{\circ}C$)	800	800	800
Black Oxide (% U_3O_8)	93.52	92.80	91.98
Black Oxide (% F)	.014	.026	.042

TABLE V
PURIFICATION OF HIGH GRADE FEED MATERIAL

MATERIAL: REACTOR ASH

ASSAY: 42.21% U₃O₈

MESH: 85% -100

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample (gm)	500	500	500
Weight of H ₂ O (gm)	2000	2000	2000
Weight of HCl (gm)	400	400	400
Calcination Temperature for Raw Material (°C)	1300	1300	1300
NaClO ₃ Used to Oxidize (gm)	1.25	1.25	1.25
Digest pH	0.55	0.60	0.32
Digest Time (hr)	3	3	3
Digest Temperature (°C)	90	90	90
Acid Insolubles (gm)	235	231	241
Acid Insolubles (% U ₃ O ₈)	.050	.070	.032
Dilution of Precipitant (NH ₄ OH)	3:1	conc.	conc.
pH of precipitation	6.0	7.0	8.2
Discard Effluent (% U ₃ O ₈)	0.87	0.69	0.49
Ammonia Salts (% U ₃ O ₈)	--	20.05	21.56
Ammonia Salts (% F)	--	4.23	4.68
Calcination Temperature for Ammonia Salts (°C)	--	800	800
Black Oxide (% U ₃ O ₈)	--	26.82	27.16
Black Oxide (% F)	--	.076	.14

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TABLE VI
PURIFICATION OF HIGH GRADE FEED MATERIAL

MATERIAL: GRAPHITE ASH

ASSAY: 15.10% U_3O_8

MESH: 85% -100

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample	500	500	500
Weight of H_2O (gm)	2000	2000	2000
Weight of HCl (gm)	440	440	440
Calcination Temperature for Raw Material ($^{\circ}C$)	1300	1300	1300
$NaClO_3$ Used to Oxidize (gm)	1.50	1.50	1.50
Digest pH	0.65	0.60	0.10
Digest Time (hr)	3	3	3
Digest Temperature ($^{\circ}C$)	90	90	90
Acid Insolubles (gm)	50	101	100
Acid Insolubles (% U_3O_8)	.10	.09	.050
Dilution of Precipitant (NH_4OH)	3:1	conc.	conc.
pH of Precipitation	6.0	6.0	7.0
Discard Effluent (% U_3O_8)	.0007	.0007	traces
Ammonia Salts (% U_3O_8)	83.44	82.89	83.10
Ammonia Salts (% F)	.10	.08	.11
Calcination Temperature for Ammonia Salts ($^{\circ}C$)	800	800	800
Black Oxide (% U_3O_8)	96.32	95.89	96.00
Black Oxide (% F)	.0036	.0028	.004

TABLE VII
DIGESTION AND REFINING OF BFC-6
ASSAY: 6.9% U_3O_8
PROCESS: SODIUM CARBONATE-AUTOCLAVE LEACH

	LOT NO: 20
Gross Weight (lb.)	29,852.8
Raw Material (% U_3O_8)	6.94
Raw Material (lb. U_3O_8)	2,071.8
Weight of Water Leached Material (lb.)	14,559
Water Leached Material (% U_3O_8)	14.30
* Weight of Sodium Carbonate Used (lb.)	30,900
Chemical Cost/lb. U_3O_8 (¢)	26.10
Weight of Insoluble Residue (lb.)	10,646.9
Insoluble Residue (% U_3O_8)	1.85
Insoluble Residue (lb. U_3O_8)	196.95
** Total Recovery (%)	90.05

- * Due to the experimental nature of the work, Na_2CO_3 was consumed in larger quantities than would be normally expected.
- ** It should be noted that this figure includes several experimental failures that resulted in only partial extraction of the contained uranium values.

TABLE VIII

EXTRACTION OF URANIUM AS URANYL AMMONIUM PHOSPHATE

MATERIAL: VITRO SODIUM CARBONATE RESIDUES

ASSAY: .27% U_3O_8

MESH: 85% -100

	<u>Experiment No.</u>	
	<u>1</u>	<u>2</u>
Weight of Sample (gm)	400	400
Weight of H_2O (gm)	800	800
Weight of HCl (gm)	200	200
Calcination Temperature for Raw Material ($^{\circ}C$)	1350	1350
Digest pH	0.90	0.85
Digest Time (hr)	1	1
Digest Temperature ($^{\circ}C$)	90	90
Acid Insoluble (gm)	330	334
Acid Insoluble (% U_3O_8)	.025	.032
Volume of Filtrate plus Wash (ml)	1400	1400
Specific Gravity of Filtrate	1.064	1.070
$NH_4H_2PO_4$ added (gm)	2.0	2.0
pH of Precipitation	2.5	2.5
UAP Residue (gm)	12.0	12.10
UAP Residue (% U_3O_8)	9.45	9.40
Discard Effluent (% U_3O_8)	traces	.0001

TABLE IV
EXTRACTION OF URANIUM AS A SOLUBLE CARBONATE
BY DIGESTION OF AMMONIA SALTS

MATERIAL: VITRO SODIUM CARBONATE RESIDUES

ASSAY: .27% U_3O_8

MESH: 85% -100

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample (gm)	100	100	100
pH of Precipitation of Ammonia Salts	6.0	6.0	6.0
Discard Effluent (% U_3O_8)	.0007	.0005	.0005
H ₂ O to Slurry (gm)	200	200	200
Na ₂ CO ₃ Added (Gm)	20	20	20
Digest Time (hr.)	2	2	2
Digest Temperature (°C)	90	90	90
ml-solids/100 ml of Slurry	22.5	22	22
Residue (% U_3O_8)	.050	.032	.055

TABLE IX-A

EXTRACTION OF URANIUM AS A SOLUBLE CARBONATE
BY DIGESTION OF AMMONIA SALTS

MATERIAL: VITRO SODIUM CARBONATE RESIDUES

ASSAY: .27% U_3O_8

MESH: 85% -100

	<u>Experiment No.</u>	
	<u>1</u>	<u>2</u>
Weight of Sample (gm)	100	100
Na_2CO_3 Added to Neutralize (gm)	25	25
Digest Time (hr)	2	2
Digest Temperature ($^{\circ}C$)	90	90
ml-solids/100 ml-slurry	32.0	32.0
Residue (% U_3O_8)	.12	.10

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TABLE X

MINERAL ACID AND ALKALI DIGESTION OF PITCHBLIND
SODIUM CARBONATE RESIDUES

ASSAY: .69% U₃O₈

MESH: 85% -100

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Sample (gm)	100	100	100
Weight of H ₂ O (gm)	200	200	200
Solvent Used	HCl	H ₂ SO ₄	Na ₂ CO ₃
gm. of Solvent	222	110	25
Digest pH	0.50	0.10	--
Digest Temperature (°C)	90	90	90
Digest Time (hr)	3	3	3
Insolubles (gm)	1	54	89
Insolubles (% U ₃ O ₈)	.032	.14	.30

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