UNCLASSIFIED

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

¹
THE VITRO MANUFACTURING COMPANY
PITTSBURGH, PENNSYLVANIA

NYO-1150

PROBLEMS OF REFINING URANIFEROUS
RESIDUES

Progress Report No. 16

Contract: AT-(30-1)-1241

Period: August 1952

Date: September 17, 1952

Submitted by:

H. Fleck
H. Fleck, Works Manager

Approved by:

J. Summers
J. Summers, Project Leader
A. Johnson
A. Johnson, AEC Contract
Administrator

Photostat Price \$ 4.80

Microfilm Price \$ 2.70

Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

UNCLASSIFIED

UNCLASSIFIED

141-1

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

NYO-1150

PROBLEMS OF REFINING URANIFEROUS RESIDUES
TECHNOLOGY - FEED MATERIALS

A. SUMMARY

GENERAL

1. Scope of Program

As of August 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 coppertin 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 U308 content of the residues below 0.05%.

2. Previous Report

NYO-1149, July Progress Report - Dated August 15, 1952.

PROJECT STATUS

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

During the month covering the present report period, experimental work on C-type residues remained static. No progress may be reported. However, the prospective program of experimentation for the next period includes further investigation of refining techniques as applied to these residues.

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

The accumulation of final data was continued during the report period. This project is scheduled for discontinuation after final evaluation of all data with regard to the efficiency of the process specifically developed for the refining of this material. However, sufficient data has not been collected to date and no progress may be reported for this period.

5. Research Project - Refining and Processing Sodium Carbonate Residues

Research during the period under report was concentrated on the accumulation of critical data concerning the extraction of uranium as uranyl ammonium phosphate from those sodium carbonate residues resulting from Vitro's current production process. Experiments were conducted covering all phases of the prospective process from calcination of the raw material to the refining of the crude uranyl ammonium phosphate residue that was obtained. In general, optimum conditions for digestion and refining have been tentatively established.

As previously established and reported, calcination of current sodium carbonate residues will be necessary to effect optimum extraction of the uranium contained. A survey of calcination temperatures from 1000°F to 1800°F was completed. Time of calcination was evaluated from one-half hour to two hours.

On the assumption that salt concentration of the pregnant acid digest filtrate was a critical factor in successful precipitation of uranyl ammonium phosphate, extensive experiments were completed which were designed to correlate maximum extraction of uranium values with minimum dissolution of contaminating salts. pH of digestion was varied from pH 0.0 to pH 2.0 and an accurate picture of digestion factors was obtained. Minimum time of digest was also established although found not to be critical when used in conjunction with the proper acid concentration.

An investigation of the minimum requirements of mono-basic ammonium phosphate for quantitative precipitation of uranium, using this procedure was initiated. It is apparent from the results obtained that quantities of the precipitant considerably in excess of the theoretical requirements will be necessary for satisfactory recovery. In addition, it was possible to correlate successful conditions of precipitation with acquired digestive factors.

The influence of pH of precipitation of uranyl ammonium phosphate as regards both satisfactory recovery and the purity of the resultant product was studied through an additional series of experiments. Preliminary experiments have established that pH of precipitation is critical as regards quantitative recovery of contained uranium values and has considerable effect upon the quantity of ammonium phosphate required for satisfactory precipitation.

From conclusions drawn from the preceeding experiments, it was considered necessary to re-evaluate the minimum requirements of ammonium phosphate necessary for optimum recovery, and an additional series of experiments was conducted. Preparation of the pregnant liquor,

prior to precipitation, by the addition of excess ammonium ion was found to be beneficial in reducing phosphate requirements for precipitation. The addition of ammonium ion was effected by adding excess amounts of hydrochloric acid, followed by neutralization with ammonium hydroxide to a point of optimum precipitation.

In view of economic factors it was considered necessary to determine the effectiveness of other forms of phosphate in precipitating uranium using these procedures. Di-basic ammonium phosphate was found to be effective as a precipitating agent. Other phosphate compounds are under consideration but data is not yet available.

A group of exploratory experiments was carried out to determine whether or not the pregnant hydrochloric acid digest filtrate would lend itself to a precipitation of $UO_4 \cdot 2H_2O$ at low pH values in the presence of hydrogen peroxide. It was readily apparent from preliminary experiments that such a procedure would be entirely unfeasible due to high iron salt concentration bringing about rapid decomposition of the peroxide.

The crude uranyl ammonium phosphate residue obtained under optimum conditions was found to contain approximately 7% U308. Further purification of the residue may be easily effected by normal procedures and refining processes presently established at the Canonsburg installation. Since no indication has been given to date as to purity specifications of the finished product, it is difficult to determine what will be a satisfactory purification process. Data on the two methods considered, yielding diverse uranium content, are presented.

Experiments of a scoping nature were conducted to obtain process data using optimum conditions as presently established. Samples were carried through the uranyl ammonium phosphate recovery process to obtain precipitate volumes, weights, slurry densities, etc. for flow sheet calculations.

Pitchblende Carbonate Residues were found to be unadaptable to the UAP extraction process. An attempt was made to

elevate the acid insoluble level, thus lowering the salt concentration of pregnant liquor, by calcining the raw material at elevated temperatures. While this procedure was successful in elevating the acid insoluble level from 3% to 13%, salt concentrations of the pregnant liquor remained prohibitively high and satisfactory precipitation of uranium as UAF was not possible.

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

Research Project: No. 1

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

6. Continuous Precipitation of Ammonium Diuranate

Experimental work on this phase of refining has been discontinued until such time as automatic pH control equipment, now on order, is made available. There is little doubt that this is a feasible process and that ammonium diuranate, when spontaneously precipitated from the HCl digest filtrate of C-Oxide under carefully controlled conditions and calcined to black oxide at 800°C will contain less than .5% F., and more than 50% U. On this basis the procedures or techniques are to be applied to production refining of C-Oxide as rapidly as possible.

C. DIGESTION AND REFINING OF BFC-6

Research Project: No. 4

Research Supervisor: J.E. Summers

PILOT OPERATIONS

7. Determination of the Efficiency of the Autoclave Refining Process

Work in the final stages of this project has been confined to a systematic gathering and evaluation of finalized data to determine the overall efficiency of the sodium carbonate autoclave leach with respect to the recovery of uranium values from BFC-6. Due to various mechanical and other difficulties, it has not been possible to obtain all of the required data during this report period. It is probable that all data will be collected during the next period. A final statement will be made and the project discontinued. It is assumed that on the basis of the process as established that BFC-6 will be carried into a production phase. All pilot work during the past period has been confined to material handling and sampling.

D. REFINING AND PROCESSING OF SODIUM CARBONATE RESIDUES

Research Project: No. 5

Research Supervisor: J.F. Summers

LABORATORY OPERATIONS

8. The Extraction of Uranium as Uranyl Ammonium Phosphate From Sodium Carbonate Process Residues

Extensive experiments were conducted during the present report period on all phases of a prospective process involving the extraction of uranium as uranyl ammonium phosphate from the Vitro sodium carbonate residues resulting from the current production process. These residues assay an average .26% U308. An effort was made to establish optimum conditions for digestion and extraction in order to push the project into the pilot stage as soon as possible. It must be assumed that optimum conditions as established are tentative since confirmation of all results have not as yet been obtained.

As established by previous experimentation, it was found to be necessary to calcine Vitro's sodium carbonate residues prior to acid digestion in order to effect satisfactory dissolution of contained uranium. In order to establish optimum calcination time and temperature, 100 gram samples were calcined at elevated temperatures ranging from 1000°F to 1800°F for two hours. Subsequently, the samples were digested in hydrochloric acid at pH 0.0 for two hours, the residue separated by filtration and washed until colorless. Comparative analyses of the residue indicated 1400°F to be the most efficient temperature of calcination. Data is presented in Table I.

To determine time of calcination, 100 gram samples were calcined at 1400°F for periods varying from one-half hour to two hours, slurried in 200 ml of H₂O, and subsequently digested in HCl at pH 0.0. After analysis of the residues, results were evaluated and one hour calcination time was tentatively indicated to be optimum. Data may be found in Table II.

From several experimental failures, it was indicated that success or failure as regards the precipitation of uranyl ammonium phosphate from the pregnant acid filtrate of Vitro's sodium carbonate residues would depend largely upon the salt concentration of the pregnant liquor. On this assumption, it was presumed that pH of digestion would have considerable influence on the concentration of solublized salts. A series of experiments was conducted, designed to correlate maximum extraction of uranium values with minimum dissolution of contaminating salts. 100 gram samples of calcined material were slurried in water at a 2:1 ratio and digested in hydrochloric acid at pH values varying from 0.0 to 2.0, and an accurate picture of digestive factors was obtained. It was found that pH values of approximately 0.9 would be satisfactory in extracting optimum quantities of uranium while at the same time solublizing only about 15% of the residue bulk. It may be seen that solublization of the raw material varied from 35% to 8%, depending upon the concentration of acid. Representative data may be found in Table III.

The pregnant clarified filtrates resulting from experiments of this nature were used for further experimentation. UAP was precipitated from them under unvaried conditions using approximately 800% of the theoretical requirements of $\text{NH}_4\text{H}_2\text{PO}_4$ at pH 1.5. The results easily confirm that salt concentration of the filtrate is critical with regard to satisfactory precipitation and purity of the resultant residue. Precipitation from a pregnant liquor of high salt concentration was incomplete and the partial precipitate was of a colloidal non-filterable type. Using optimum digestive conditions, satisfactory precipitation was readily obtained, and the residue easily separated by filtration. Representative data may be found in Table IIIA.

Minimum time of digest was established by slurrying 100 gram samples in 200 ml of H_2O and digesting in hydrochloric acid at approximately pH 1.0 for periods of time varying from one-half to two hours. Thirty minutes is indicated to be a satisfactory period of digestion. Apparently, time of digest is not critical when used in conjunction with the proper acid concentration, so far as salt concentration of the filtrate is concerned. Data is presented in Table IV.

An investigation was conducted to ascertain the minimum requirements of mono-basic ammonium phosphate necessary for quantitative precipitation of uranium. Precipitant weights were varied from approximately 200% of the theoretical requirements to approximately 1000% of the theoretical requirements. The clarified filtrates from hydrochloric acid digests under optimum conditions were combined with the residue wash and used for experimentation. Mono-basic ammonium phosphate was added in proportionate amounts and the pH adjusted to 1.5 for precipitation. It may be seen from the data obtained from initial experiments that precipitation could not be considered satisfactory. Recovery was completely inadequate and increased with the addition of larger amounts of the precipitant. No ready explanation for this sudden failure was apparent, but it was assumed that it was due to a lack of sufficient ammonium ion in solution. Combining the residue wash with the pregnant liquor elevated the pH of the liquor to pH 1.0 - 1.1 and only small quantities of NH_4OH were needed to adjust the pH of precipitation. In general, the solution must be at least 0.5 molar in NH_3 ion to effect quantitative recovery. Data resulting from these experiments may be found in Table V.

Elevation of the pH of precipitation resulted in successful extraction of uranium. This bears out the hypothesis that insufficient ammonium ion results in adequate recovery. Assuming that the pregnant liquor is lacking at the pH of precipitation in sufficient ammonium ion for quantitative precipitation, elevation of pH or further neutralization of excess acid by dilute ammonium hydroxide would have a beneficial effect. Samples were digested in hydrochloric acid under optimum conditions, the clarified filtrates were combined with the residue wash and subjected to phosphate extraction, using 800% of the theoretical requirements. pH of precipitation was raised from 1.5 to 2.5 and recovery was satisfactory. Data may be found in Table VI.

Experiments were completed, designed to determine the effectiveness of adding 0.5 molar NH_3 ion prior to precipitation of UAP. To clarified HCl digest filtrates obtained under optimum digest conditions, 0.5 molar HCl was added in excess. After the addition of approximately 800% of the theoretical requirements of $\text{NH}_4\text{H}_2\text{PO}_4$, the solution was neutralized to pH 1.5 with dilute ammonia. Precipitation

was quantitative and the purity of the resultant residue exceeded that obtained at elevated pH values. Data may be found in Table VII.

From conclusions drawn by the preceeding experiments, it was believed that pH of precipitation or the presence of excess ammonium ion would have considerable effect upon minimum requirements of phosphate for precipitation. Clarified hydrochloric acid filtrates were prepared from several samples and excess hydrochloric acid added to 0.5 molar. Again the requirements of mono-basic phosphate were varied to determine if satisfactory precipitation could be obtained using smaller quantities. By neutralizing excess hydrochloric acid with dilute ammonia to pH 1.5 it was found that the phosphate requirements did not exceed approximately 400% of the theoretical requirements. Obvious gaps exist in the data, and additional series of experiments are planned to further evaluate all factors of precipitation in the light of the data obtained to date. Representative data is presented in Table VIII.

Economically speaking, it was considered necessary to determine the effectiveness of other forms of phosphate in precipitating uranium using these procedures. By substituting dibasic ammonium phosphate in optimum quantities and using optimum procedures for precipitation throughout, this compound was found to be as equally effective as mono-basic ammonium phosphate as a precipitating agent. Other phosphate compounds such as phosphoric acid are under consideration but data is not yet available. Preliminary data is found in Table IX.

Scoping experiments were conducted to determine whether or not uranium could be precipitated as $UO_4 \cdot 2H_2O$ in the presence of hydrogen peroxide from the pregnant hydrochloric acid digest filtrates of current carbonate residues. It was hoped that Fe_2O_3 content of the pregnant liquor would not be high enough to retard precipitation. However, it was readily apparent from initial experiments that such a procedure would be entirely unsatisfactory. Rapid decomposition of the peroxide was effected by the high salt concentration of the filtrate. Addition of quantities of hydrogen peroxide far in excess of the theoretical requirements did not benefitiate extraction.

The crude uranyl ammonium phosphate residue obtained using optimum conditions was found to contain approximately 7% U308. Purification of the residue was effected by the use of the established Vitro refining process consisting of dissolution in sulphuric acid, removal of the R2O3 group by neutralization of excess acid with sodium carbonate, and precipitation of sodium diuranate from the carbonate liquor by the addition of sodium hydroxide. This product assays approximately 80% U308. However, it is possible to obtain a refining product by a simpler treatment although uranium specifications will not be nearly so high. By digestion of the crude phosphate residue in 1:1 sodium hydroxide solution at 90°C and removal of the insoluble residue by decantation and filtration, it is possible to obtain a finished product containing approximately 20 to 25% U308. Primary experiments using this technique had indicated a much higher U308 content of the finished product, but later data has indicated that the crude residue will contain approximately 25% Fe2O3 which is carried over to the finished product. Additional experiments are in progress to firm-up this data. There is no knowledge at the moment as to what constitutes a satisfactory product. It is assumed that one or the other of these processes may be used depending upon the end product desired. Data obtained to date is presented in Table X.

Since it is considered desirous to carry this treatment into the pilot phase as soon as possible, experiments were conducted which were designed to obtain process data under optimum conditions. Samples were carried throughout the entire procedure up to the point of phosphate precipitation. Precipitate volumes, weights, slurry densities, etc. were obtained for design flow sheet calculations. Data is found in Table XI.

An attempt was made to precipitate uranyl ammonium phosphate from a hydrochloric acid digest filtrate of the Vitro pitchblende carbonate residues assaying 1.6% U308. A prior effort to accomplish this had indicated that the salt concentration of the pregnant filtrate was excessively high and that the precipitation of UAP would not be feasible. However, an attempt was made to elevate the acid insoluble level thus lowering the salt concentration of the pregnant liquor by calcining the raw material

at 1600°F. While this procedure was partially successful in elevating the acid insoluble level from 3 to 13%, the salt concentration of the pregnant liquor remained prohibitively high and the attempt to precipitate UAP was completely ineffectual. It is assumed that the phosphate is complexed by the high Fe content.

EXHIBITS

17

TABLE I

DETERMINATION OF CALCINATION TIME

MATERIAL: VITRO SODIUM CARBONATE RESIDUE

ASSAY: .26% U308

MESH: 85% -100

	<u>Experiment No.</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 ₂ O (gm.)	200	200	200	200	200
Weight of HCl (gm.)	100	100	100	100	100
Calcination Temperature (°F)	1000	1200	1400	1600	1800
Digest Time (hr.)	2	2	2	2	2
Digest pH	0.0	0.0	0.0	0.0	0.0
Residue - gm.	74.0	67.0	65.5	65.0	66.5
Residue - U308	.14	.055	.017	.017	.063
Recovery - %	60	86.2	95.8	95.9	84.7

18

TABLE II

DETERMINATION OF TIME OF CALCINATION

MATERIAL: VITRO SODIUM CARBONATE RESIDUE

ASSAY: .26% U308

MESH: 85% -100

	<u>Experiment No.</u>			
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Time of calcination (hr.)	0.5	1.0	1.5	2.0
Weight of sample (gm.)	100	100	100	100
Weight of H ₂ O (gm.)	200	200	200	200
Weight of HCl (gm.)	100	100	100	100
Calcination Temperature (°F)	1400	1400	1400	1400
Digest Time (hours)	2.0	2.0	2.0	2.0
Digest Temperature (°C)	90	90	90	90
Digest pH	0.0	0.0	0.0	0.0
Residue - gm.	66.5	66.0	65.0	66.5
Residue - % U308	.055	.035	.035	.035
Recovery - %	86.2	91.1	91.6	90.8

19

TABLE III

DETERMINATION OF pH OF DIGESTION

MATERIAL: VITRO SODIUM CARBONATE RESIDUE

ASSAY: .26% U308

MESH: 85% -100

	<u>Experiment No.</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 ₂ O	200	200	200	200	200
Weight of HCl (gm.)	25	50	75	100	125
pH of digest	1.95	0.9	0.35	0.0	-0.0
Time of digest (hr.)	2	2	2	2	2
Temperature of Digest	90	90	90	90	90
Specific gravity of Filtrate	1.044	1.080	1.098	1.134	1.146
Residue (gm.)	92.0	85.0	75.0	66.5	65.0
Residue - % U308	.28	.050	.055	.054	.035

20
TABLE III-A

PRECIPITATION OF UAP ACCORDING TO DIGEST CONDITIONS

MATERIAL: VITRO SODIUM CARBONATE RESIDUE

ASSAY: .26% U308

MESH: 85% -100

	<u>Experiment No.</u>			
	<u>1A</u>	<u>2A</u>	<u>3A</u>	<u>4A</u>
Weight of sample (gm.)	100	100	100	100
Volume of Filtrate (ml.)	260	275	305	350
Specific gravity of filtrate	1.044	1.080	1.098	1.134
NH ₄ H ₂ PO ₄ added (gm.)	2.0	2.0	2.0	2.0
pH of precipitation	1.5	1.5	1.5	1.5
Residue - gm.	1.0	4.0	6.5	colloidal non-filterable
Residue - % U308	2.128	5.15	.56	--
Discard Effluent - % U308	.002	Traces	.01	--
Recovery - %	98.0	100	85.2	--

21

TABLE IV

DETERMINATION OF DIGEST TIME

MATERIAL: VITRO SODIUM CARBONATE RESIDUES

ASSAY: .26% U308

	<u>Experiment No.</u>			
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Weight of sample (gm.)	100	100	100	100
Weight of H ₂ O (gm.)	200	200	200	200
Weight of HCl (gm.)	50	50	50	50
Digest pH	0.85	0.90	0.85	0.90
Digest Time (hr.)	0.5	1.0	1.5	2.0
Residue (gm.)	84.5	83.5	85.5	85.2
Residue - % U308	.035	.055	.035	.035
Recovery - %	88.9	84.3	88.2	88.6

22

TABLE V

DETERMINATION OF $\text{NH}_4\text{H}_2\text{PO}_4$ REQUIREMENTS

MATERIAL: VITRO SODIUM CARBONATE RESIDUE

ASSAY: .26% U308

DIGEST CONDITION: 1/2 HR. AT 90°C

DIGEST pH: 0.8 - 0.9

	<u>Experiment No.</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
Volume of Filtrate wash (ml)	320	320	320	320	320
Specific gravity of filtrate	1.078	1.078	1.076	1.078	1.079
pH prior to precipitation	1.05	1.10	1.00	1.05	1.05
pH of precipitation	1.5	1.5	1.5	1.5	1.5
$\text{NH}_4\text{H}_2\text{PO}_4$ added (gm.)	0.5	1.0	1.5	2.0	2.5
Residue (gm.)	2.0	2.0	3.0	3.0	3.4
Residue - % U308	.56	1.4	5.04	5.65	6.2
Discard effluent - % U308	.065	.060	.032	.025	.018
Recovery	23.0	27.0	61.6	69.3	79.5

23

TABLE VI

DETERMINATION OF EFFECT OF pH OF PRECIPITATION

MATERIAL: VITRO SODIUM CARBONATE RESIDUE

ASSAY: .26% U308

DIGEST CONDITIONS: 1/2 HR. AT 90°C

DIGEST pH: 0.8 - 0.9

	<u>Experiment No.</u>			
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Weight of sample (gm.)	100	100	100	100
Volume of Filtrate & Wash (ml)	330	330	330	330
Specific gravity of Filtrate	1.075	1.075	1.072	1.078
pH of precipitation	2.0	2.0	2.5	2.5
NH ₄ H ₂ PO ₄ added (gm)	2.0	2.0	2.0	2.0
Residue - gm	4.1	4.3	4.5	4.4
Residue - % U308	6.65	6.50	6.3	6.38
Discard Effluent & % U308	Traces	.0001	Traces	Traces
Recovery - %	100	99.7	100	100

24

TABLE VII

DETERMINATION OF THE EFFECT OF ADDITION OF EXCESS NH_3 ION

MATERIAL: VITRO CARBONATE RESIDUE

ASSAY: .26% U308

DIGEST CONDITIONS: 1/2 HR. AT 90°C

DIGEST pH: 0.8 - 0.9

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of sample	100	100	100
Volume of Filtrate & Wash (ml)	330	330	300
Specific gravity of filtrate	1.076	1.076	1.074
Excess HCl added (molar)	0.5	0.5	0.5
pH of precipitation	1.5	1.5	1.5
$\text{NH}_4\text{H}_2\text{PO}_4$ added (gm)	2.0	2.0	2.0
Residue - gm	3.7	3.9	3.75
Residue - % U308	7.56	7.30	7.50
Discard Effluent - % U308	Traces	.0001	Traces
Recovery - %	100	99.9	100

25

TABLE VIII

DETERMINATION OF $\text{NH}_4\text{H}_2\text{PO}_4$ REQUIREMENTS AFTER ADDITION OF EXCESS NH_3 ION

MATERIAL: VITRO SODIUM CARBONATE RESIDUE

ASSAY: .26% U308

DIGEST CONDITIONS: 1/2 HR. AT 96°C

DIGEST pH: 0.8 - 0.9

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of sample (gm.)	100	100	100
Volume of filtrate & wash (ml)	320	320	320
Specific gravity of Filtrate	1.072	1.072	1.074
Excess HCl added (molar)	0.5	0.5	0.5
pH of precipitation	1.5	1.5	1.5
$\text{NH}_4\text{H}_2\text{PO}_4$ added (gm.)	1.0	1.5	2.0
Residue - gm.	3.5	3.8	3.95'
Residue - % U308	8.05	7.4	6.8
Discard effluent - % U308	.0006	.0003	Traces
Recovery - %	99.5	99.75	100

26

TABLE IX

DETERMINATION OF REQUIREMENTS OF $(\text{NH}_4)_2\text{H PO}_4$

MATERIAL: VITRO SODIUM CARBONATE RESIDUE

ASSAY: .26% U308

DIGEST CONDITIONS: 1/2 HR. AT 90°C

DIGEST pH: 0.8 - 0.9

	<u>Experiment No.</u>	
	<u>1</u>	<u>2</u>
Weight of sample	100	100
Excess HCl added (molar)	0.5	0.5
pH of precipitation	1.5	1.5
$(\text{NH}_4)_2\text{H PO}_4$ added (gm.)	1.5	1.5
Residue - % U308	7.2	7.45
Residue - gm.	3.9	3.80
Discard Effluent	.0002	Traces

TABLE X
REFINING DATA USING VITRO PURIFICATION PROCESS
MATERIAL: CRUDE URANYL AMMONIUM PHOSPHATE
ASSAY: 7.10% U308

	<u>Experiment No.</u>	
	<u>1</u>	<u>2</u>
Weight of phosphate residue (gm)	30	30
Weight of H ₂ O (gm)	100	100
Weight of H ₂ SO ₄ (gm)	18.5	18.5
Digest pH	6.6	6.0
Digest Time (hr)	2	2
Digest Temperature (°C)	90	90
Na ₂ CO ₃ added to filtrate (gm)	20	20
Digest Time (hr)	1	1
Digest Temperature (°C)	60	60
NaOH added to filtrate (gm)	3.0	3.0
Digest Time (hr)	1/2	1/2
Digest Temperature (°C)	90	90
Residue - gm	2.50	2.35
Residue - % U308	78.3	80.2
Discard Effluent - % U308	.002	.003

TABLE X (CON'T.)

REFINING DATA USING NaOH DIGESTION

MATERIAL: CRUDE URANYL AMMONIUM PHOSPHATE

ASSAY: 6.85% U308

	<u>Experiment No.</u>	
	<u>1</u>	<u>2</u>
Weight of sample (gm)	20	20
Weight of H ₂ O (gm)	100	100
Weight of NaOH (gm)	50	50
Digest Time (hr)	2	2
Digest Temperature (°C)	90	90
Residue weight (gm)	6.50	5.85
Discard Effluent (% U308)	Traces	Traces
Residue - % U308	21.5	24.2

29

TABLE XI

PROCESS DATA OF THE UAP EXTRACTION
MATERIAL: VITRO SODIUM CARBONATE RESIDUE

DIGEST CONDITION: 1/2 HR. AT 90°C

PRECIPITANT: $\text{NH}_4\text{H}_2\text{PO}_4$

ASSAY: .26% U308

DIGEST pH: 0.8 - 0.9

	<u>Wet Weight (gm)</u>	<u>ml solids per 100 ml slurry</u>	<u>Slurry Volume (ml)</u>	<u>ml solids Per gram Raw Mat'l</u>	<u>approx. % U308 Content</u>
HCl Acid Insolubles	A. 104.5	A. 35.0	A. 300	A. 1.05	A. 035
	B. 103.0	B. 36.0	B. 300	B. 1.08	B. 035
Uranyl Ammonium Phosphate	A. 8.5	A. 18.0	A. 350	A. .63	A. 6.80
	B. 8.0	B. 18.5	B. 350	B. .64	B. 7.00