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THE VITRO MANUFACTURING COMPANY

PITTSBURGH, PENNSYLVANIA

AEC RESEARCH AND DEVELOPMENT REPORT

NYO-1151

PROBLEMS OF REFINING URANIFEROUS RESIDUES

TECHNOLOGY - FEED MATERIALS

Progress Report No. 17

Contract: AT-(30-1)-1241

Period: September, 1952

Date: October 15, 1952

Submitted by:

H. Fleck
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NYO-1151
Page 3

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NYO-1151

PROBLEMS OF REFINING URANIFEROUS RESIDUES
TECHNOLOGY - FEED MATERIALS

A. SUMMARY

GENERAL

1. Scope of Program

As of September 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 HFC-6 (Project No. 4)

Investigate the recovery of uranium from HFC-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%.

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NYO-1151

Page: 4

2. Previous Report

NYO-1150, August Progress Report - Dated September 17, 1952.

PROJECT STATUS

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

Since all phases of experimental work on C-type residues have entered the pilot phase and the Canonsburg Operation was inoperative due to labor difficulties during the month of September no progress may be reported for this period. Experimental work on the pilot level regarding C-oxide will be initiated again this month.

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

The shutdown of the operating facility during the month of September has made it impossible to complete this project as planned. However, assuming no further difficulty, the program of experimentation will be carried to a conclusion during the following report period.

5. Research Project - Refining and Processing Vitro Sodium Carbonate Residues

Research during the period under report was concentrated on obtaining confirmation of critical data concerning the precipitation of uranyl ammonium phosphate from the acid digest filtrates of Vitro sodium carbonate residues assaying .26% U3O8. In particular, experiments were conducted which were designed to integrate and correlate all factors influencing quantitative precipitation. Experiments were conducted designed to fill statistical blanks in the data already accumulated in order to obtain a clearer picture of optimum factors influencing satisfactory recovery.

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A series of experiments was conducted to correlate the influence of pH of precipitation of uranyl ammonium phosphate with minimum requirements of monobasic ammonium phosphate necessary for quantitative and satisfactory precipitation. Data gathered from completed experiments indicates that the pH of precipitation has considerable influence on the quantities of precipitant necessary for successful extraction.

In addition, experiments were completed and data accumulated from which it is possible to assay the influence of excess ammonium ion as correlated with optimum values of monobasic ammonium phosphate necessary for quantitative recovery of UAP. Results of these experiments may be used to establish the most effective procedure for precipitation as regards both economics and efficiency of extraction.

A further investigation of the possibility of using phosphate compounds other than basic ammonium phosphate was completed. It is readily apparent from the data obtained that the only readily available compounds which will be effective as precipitating agents are mono and di-basic ammonia phosphates. However, from an economic standpoint the procedure does not appear to be unattractive.

Refining procedures as regards the resultant UAP precipitate obtained, using techniques of extraction as outlined, were reviewed and confirming data obtained. More extensive experiments regarding the purification of the phosphate precipitate are not contemplated at this time since specifications concerning a satisfactory end product are not yet indicated.

Preliminary experiments were conducted concerning the extraction of uranium from ammonia salts precipitated from the acid digest liquor of Vitro sodium carbonate residues by digestion of the ammoniacal precipitate in the presence of various forms of carbonate compounds. The experiments were designed to determine whether or not there was any appreciable difference in this type of treatment as compared to direct carbonation of the acid digest filtrate. Therefore, experiments were also conducted in parallel by direct carbonation of a hydrochloric

acid digest filtrate of Vitro carbonate residues. Comparative data was obtained and is presented.

In addition, the calcination phase of the prospective process were again reviewed to obtain confirmation of critical data.

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

Research Project: No. 1

Research Supervisor: J. E. Summers

PILOT OPERATIONS

6. Continuous Precipitation of Ammonium Diuranate

The Canonsburg facility was completely inoperative during the month of September, and no experiments of a pilot nature were possible. However, since operations have now been resumed, present planning calls for continuation of pilot experiments regarding the continuous precipitation of ammonia salts from the acid digest filtrates of C-Oxide by spontaneously combining the acid stream with ammonia, under carefully controlled conditions in the presence of vigorous agitation. Additional data concerning this extraction procedure will be reported as rapidly as it is accumulated.

C. DIGESTION AND REFINING OF HFC-6

Research Project: No. 4

Research Supervisor: J. E. Summers

PILOT OPERATIONS

7. Determination of the Efficiency of the Autoclave Refining Process

It has not been possible to complete this project as planned due to the work stoppage during the period under report. Assuming no further operative difficulties, final statistical data designed to evaluate overall efficiency of the sodium carbonate autoclave leach of HFC-6, will be presented during the next period. A summation report regarding this phase of Research and Development activities will be prepared as soon as the project is finally discontinued.

D. REFINING AND PROCESSING OF SODIUM CARBONATE RESIDUES

Research Project: No. 5

Research Supervisor: J. E. Summers

LABORATORY OPERATIONS

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

It was apparent from the data obtained during the previous report period that there were several statistical blanks in the information concerning the precipitation of uranyl ammonium phosphate from the acid digest filtrates of the Vitro sodium carbonate residues. Again it was fairly obvious that certain factors of precipitation and their influence on quantitative recovery had not been completely assayed. Consequently, an extensive series of experiments was concluded designed to correlate the influence of pH of precipitation with optimum quantities of monobasic ammonium phosphate necessary for quantitative or satisfactory recovery. 100 gram samples of Vitro carbonate residues were calcined at 1300° F, slurried in 200 ml of H₂O, digested under optimum conditions in HCl at pH 0.8-0.9, filtered, and the pregnant acid liquor obtained was used for experimentation. UAP was precipitated from the clarified filtrate by neutralizing excess acid with dilute NH₄OH to pH values ranging from 1.5 to 2.5 in the presense of varying amounts of the precipitant ranging from 200% to 800% of the theoretical requirements. Data obtained in this manner indicates that with elevation of pH of precipitation, precipitant requirements were appreciably lowered. It was found that at pH 2.5 only 200% of the theoretical requirements for quantitative precipitation

were necessary. The purity of the resultant residue obtained at elevated pH values was not found to markedly differ from previous optimum values when larger quantities of precipitant were used at lower pH values. Economically, under these conditions, the procedure becomes somewhat more desirable. Complete representative data may be found in Table I.

In addition, extensive experiments were completed based on the assumption that it is necessary that the pregnant solution contain at least 0.5 molar ammonium ion before quantitative precipitation is obtained. Experiments were completed in which uranyl ammonium phosphate was precipitated at pH values ranging from 1.5 to 2.5 by neutralization of excess acid with dilute NH_4OH , using monobasic ammonium phosphate as a precipitating agent in quantities ranging from 200% to 800% of the theoretical requirements after first preparing the solution to be precipitated with excess ammonium ion to concentrations of at least 0.5 molar. 100 gram samples of Vitro sodium carbonate residues assaying approximately .26% U_3O_8 were calcined at optimum temperatures approximating 1300°F, slurried in 200 ml of H_2O and digested in hydrochloric acid under optimum conditions at pH 0.8-0.9. After clarification of the slurry by filtration, the acid digest filtrates were used for experimentation. The solution was prepared for precipitation by adding 0.5 molar excess hydrochloric acid and neutralizing with an equivalent weight of ammonium hydroxide.

From the data obtained it may be readily observed that, although the presence of excess ammonium ion, prior to precipitation, has considerable influence on quantitative recovery, elevation of the pH of precipitation is undoubtedly the most economical means of lowering the phosphate requirements necessary for satisfactory extraction. No additional work is contemplated at this time on this phase of the procedure insofar as laboratory operations are concerned. A pilot operation will be established as soon as feasible in order to determine whether or not the process, as outlined, will be practical on a large scale. Representative data is presented in Table II.

An investigation conducted to determine the feasibility of using phosphate compounds other than monobasic ammonium phosphate was concluded. It is evident from the data obtained that while di-basic ammonium phosphate may be used to practical advantage, no other phosphate compound is readily apparent that will bring about quantitative extraction of contained uranium values. 100 gram samples of Vitro sodium carbonate residues, assaying approximately .26% U₃O₈ were calcined at 1300° F, slurried in 200 ml of H₂O and digested in hydrochloric acid, using established optimum digestive factors at pH 0.8 to 0.9. The clarified acid digest filtrates resulting from this procedure were tested with di-basic ammonium phosphate, phosphoric acid and tri-sodium phosphate in the presence of excess ammonium ion. Satisfactory and quantitative recovery was obtained only through the use of di-basic ammonium phosphate. Since experimentation has established that approximately 200% of the theoretical requirements will effect maximum recovery, and because these figures are not too advantageous on an economic basis a more extensive search for other precipitant forms is not indicated. Data is found in Table III.

It was found to be possible to further purify those residues resulting from the UAP refining procedure by using the established Vitro purification process consisting of sulphuric acid digestion, removal of iron and aluminum by neutralization of excess acid with sodium carbonate followed by precipitation of uranium from the clarified carbonate filtrate by the addition of sodium hydroxide. In this manner a high grade product assaying between 75 and 80% U₃O₈ may be obtained. This data confirms previous experimentation. Complete data is presented in Table IV.

Additional experiments were conducted in which the crude uranyl ammonium phosphate residue obtained by use of optimum critical factors was digested at 90° F in strong (1:1) sodium hydroxide solution. In this fashion, phosphate ion and excess aluminum are removed as soluble salts leaving an iron cake averaging about 22-25% U₃O₈, thus confirming data already obtained. Whether or not this is, or would be, a satisfactory end product is not known at this time. No further experiments of a refining nature are indicated at this time until it is

definitely established what constitutes a satisfactory end product. Data is tabulated in Table V.

As previously reported, the Vitro sodium carbonate residues must be calcined before satisfactory extraction of uranium may be effected. The calcination phase was reviewed from 1000° F - 1600° F to further evaluate optimum temperatures. 100 gram samples were calcined for one hour at varied temperatures and the fired residues digested in HCl at pH 0.8-0.9. Analysis of the insoluble residue indicates that temperatures of the order of 1200-1300° F will be satisfactory in effecting optimum extraction of contained values. Data is presented in Table VI.

9. Extraction of Uranium as a Soluble Carbonate From Ammonium Salts Precipitated from the Digest Liquor of Sodium Carbonate Process Residues

Previous experimentation had indicated that it is possible to extract contained uranium values from the Vitro carbonate residues by neutralization of the hydrochloric acid digest filtrate with sodium carbonate followed by removal of the insoluble residue by filtration. However, it was definitely established that such a procedure left residues containing uranium values in excess of .05% U3O8. In order to reduce the uranium levels of these residues to less than .05% U3O8, it was necessary to carry the resultant carbonate residue through a series of repulp washes. This procedure, while effective, resulted in the accumulation of large quantities of liquor containing very low uranium concentrations. Apparently the sodium carbonate residue is of such physical characteristics that uranium values are readily occluded. To avoid this hazard, preliminary experiments were conducted in which the acid digest filtrate resulting from digestion of the Vitro sodium carbonate residues was neutralized with ammonia to pH values approximating 6.0 and the resultant ammonium salts removed by filtration. The residue thus obtained was slurried in H₂O and digested in the presence of various carbonate compounds such as sodium carbonate, ammonium carbonate and sodium bicarbonate. After digestion at elevated temperatures up to 90° C for periods of time up to two hours,

the insoluble residue was removed by filtration, washed and analyzed and found to contain less than .05% U₃O₈ except when (NH₄)₂CO₃ was used. No ready explanation is at hand why this treatment should be effective and direct carbonation of the acid filtrate ineffectual. Presumably the physical characteristics of the two residues are somewhat dissimilar and the ammonia cake does not so readily occlude contained values. Additional experiments of this nature are planned to obtain confirmation of these results. Preliminary data may be found in Table VII.

An additional series of experiments, duplicating experiments already conducted, was completed in which the acid filtrate resulting from digestion of Vitro sodium carbonate residues was processed by direct carbonation. These experiments were run in parallel with the carbonate digestion of ammonia salts in order to obtain comparative data. In every instance the insoluble residue obtained from a treatment of this nature contained uranium values in excess of .10% U₃O₈. Only by a process of repulp washing was it possible to reduce the uranium content below maximum specifications. Average data is presented in Table VII A.

E X H I B I T S

15

TABLE I

DETERMINATION OF INFLUENCE OF PH OF PRECIPITATION ON EXTRACTION OF URANYL AMMONIUM PHOSPHATE

MATERIAL: VITRO SODIUM CARBONATE RESIDUES

ASSAY: .26% U308

DIGEST CONDITIONS: 1/2 HR. AT 90°C AT PH 0.8 - 0.9

	<u>Experiment No.</u>					
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Weight of sample	100	100	100	100	100	100
Volume of filtrate & wash (ml)	300	300	300	300	300	300
pH prior to precipitation	1.05	1.00	1.00	1.10	1.05	1.05
pH of precipitation	1.5	2.0	2.5	1.5	2.0	2.5
NH ₄ H ₂ PO ₄ added (gm)	0.5	0.5	0.5	1.0	1.0	1.0
Residue (gm)	2.5	2.5	3.0	3.0	3.5	3.5
Residue (%U308)	3.57	4.48	9.52	6.9	Traces	.0007
Discard Effluent (% U308)	.049	.040	Traces	.0014	7.77	7.87
Recovery (%)	36.4	47.9	100	98.6	100	99.8

16

TABLE I (CONTINUED)

DETERMINATION OF INFLUENCE OF PH OF PRECIPITATION ON EXTRACTION OF URANYL AMMONIUM PHOSPHATE

MATERIAL: VITRO SODIUM CARBONATE RESIDUES

ASSAY: .26% U308

DIGEST CONDITIONS: 1/2 HR. AT 90°C AT PH 0.8 - 0.9

Experiment No.

	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>
Weight of sample (gm)	100	100	100	100	100	100
Volume of filtrate and wash (ml)	300	300	300	300	300	300
pH prior to precipitation	1.10	1.05	1.05	1.05	1.025	1.05
pH of precipitation	1.5	2.0	2.5	1.5	2.0	2.5
NH ₄ H ₂ PO ₄ added (gm)	1.5	1.5	1.5	2.0	2.0	2.0
Residue (gm)	3.0	3.5	4.0	3.0	3.5	4.0
Residue (% U308)	7.36	6.8	6.23	7.14	7.02	6.6
Discard Effluent (% U308)	.001	.001	Traces	.0003	Traces	.0005
Recovery (%)	99.2	99.8	100	99.5	100	99.4

17

TABLE II

DETERMINATION OF THE EFFECT OF ADDITION OF EXCESS NH_3 ION

ON THE PRECIPITATION OF URANYL AMMONIUM PHOSPHATE

MATERIAL: VITRO SODIUM CARBONATE RESIDUES

ASSAY: .26% U_3O_8

	<u>Experiment No.</u>					
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Weight of sample (gm)	100	100	100	100	100	100
Volume of Filtrate and Wash (ml)	300	300	300	300	300	300
Excess HCl added (molar)	0.5	0.5	0.5	0.5	0.5	0.5
pH of Precipitation	1.5	2.4	2.5	1.2	2.0	2.5
$\text{NH}_4\text{H}_2\text{PO}_4$ added (gm)	0.5	0.5	0.5	1.0	1.0	1.0
Residue (gm)	3.5	4.0	3.5	3.5	3.0	4.0
Residue (% U_3O_8)	4.5	5.11	7.21	6.83	8.1	6.86
Discard Effluent (% U_3O_8)	.016	.0017	Traces	.0007	.0001	.0007
Recovery (%)	76.1	99.8	100	99.9	99.9	99.9

18

TABLE II (CONTINUED)

DETERMINATION OF THE EFFECT OF ADDITION OF EXCESS NH_3 ION

ON THE PRECIPITATION OF URANYL AMMONIUM PHOSPHATE

M/TERI-L: VITRO SODIUM CARBONATE RESIDUES

ASSAY: .26% U308

	<u>Experiment No.</u>					
	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>
Weight of sample (gm)	100	100	100	100	100	100
Volume of Filtrate & wash (ml)	300	300	300	300	300	300
Excess HCl added (molar)	0.5	0.5	0.5	0.5	0.5	0.5
pH of Precipitation	1.5	2.0	2.5	1.5	2.0	2.5
$\text{NH}_4\text{H}_2\text{PO}_4$ added (gm)	1.5	1.5	1.5	2.0	2.0	2.0
Residue (gm)	4.0	4.0	4.75	3.5	3.5	4.0
Residue (%U308)	6.58	6.61	5.5	7.14	7.02	6.82
Discard Effluent (%U308)	.0001	Traces	.0007	.0003	Traces	Traces
Recovery (%)	99.9	100	99.9	99.9	100	100

19

TABLE III

EVALUATION OF PHOSPHATE PRECIPITANTS

MATERIAL: VITRO SODIUM CARBONATE RESIDUES

ASSAY: .26% U308

DIGEST CONDITIONS: 1/2 HR. AT 90°C AT PH 0.8 - 0.9

	<u>Experiment No.</u>					
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Weight of sample (gm)	100	100	100	100	100	100
Excess HCl added (molar)	-	-	-	-	0.5	0.5
pH of Precipitation	1.7	1.7	2.5	2.5	2.5	2.5
Precipitant added	(NH ₄) ₂ H PO ₄	(NH ₄) ₂ H PO ₄	H ₃ PO ₄	H ₃ PO ₄	Na ₃ PO ₄	Na ₃ PO ₄
Residue (% U308)	7.88	7.74	.35	.55	-	-
Residue (gm)	3.0	3.0	2.0	2.0	-	-
Discard Effluent (% U308)	.0002	Traces	.09	.07	-	-
Recovery (%)	99.7	99.7	99.7	99.7	-	-

TABLE IV

REFINING DAT USING VITRO PURIFICATION PROCESS

MATERIAL: CRUDE UR. NYL AMMONIUM PHOSPHATE

ASSY: 7.78%

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of Phosphate Residue (gm)	30	30	30
Weight of H ₂ O (gm)	100	100	100
Weight of H ₂ SO ₄ (gm)	18.5	18.5	18.5
Digest pH	0.0	0.0	0.0
Digest Time (h .)	2	2	2
Digest Temperature (°C)	80	80	80
Na ₂ CO ₃ added to filtrate (gm)	25	25	25
Digest Time (hr)	1	1	1
Digest Temperature (°C)	80	80	80
NaOH added to Filtrate (gm)	3.5	3.5	3.5
Digest Time (hr.)	1/2	1/2	1/2
Digest Temperature (°C)	90	90	90
Residue (gm)	2.90	3.00	2.85
Residue (% U308)	78.2	77.6	79.8
Discard Effluent (% U308)	.001	.001	.002

21

TABLE V

REFINING DATA USING NaOH DIGESTION

MATERIAL: CRUDE URANYL AMMONIUM PHOSPHATE

ASSAY: 7.42% U308

	<u>Experiment No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
Weight of sample (gm)	25	25	25
Weight of H ₂ O (gm)	100	100	100
Weight of NaOH (gm)	100	100	100
Digest Time (hr)	2		
Digest Temperature (°C)	95	95	95
Residue Weight (gm)	7.85	7.40	7.75
Discard Effluent (%U308)	Traces	Traces	.0001
Residue (% U308)	23.6	25.2	24.2

22

TABLE VI

DETERMINATION OF CALCINATION TEMPERATURE

MATERIAL: VITRO SODIUM CARBONATE RESIDUES

ASSAY: .26% U308

MESH: 85% -100

Experiment No.

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Weight of sample (gm)	100	100	100	100	100	100
Weight of H ₂ O (gm)	200	200	200	200	200	200
Weight of HCl (gm)	100	100	100	100	100	100
Calcination Temperature (°F)	1000	1100	1200	1300	1400	1500
Calcination Time (hr.)	1	1	1	1	1	1
Digest Time (hr.)	1/2	1/2	1/2	1/2	1/2	1/2
Digest pH	0.8-0.9	0.8-0.9	0.8-0.9	0.8-0.9	0.8-0.9	0.8-0.9
Digest Temperature (°C)	90	90	90	90	90	90
Residue (gm)	84.3	85.4	84.0	84.5	84.6	83.8
Residue (%U308)	.1	.1	.050	.135	.050	.017
Recovery (%)	63.9	68.7	84.9	88.9	84.6	94.7

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NO-1154
Page: 22

TABLE VII

EXTRACTION OF URANIUM FROM AMMONIUM SALTS AS A

SOLUBLE CARBONATE

MATERIAL: VITRO SODIUM CARBONATE RESIDUE

ASSAY: .26% U3O8

DIGEST CONDITIONS: 1/2 HR. AT 90°C AT 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
pH of precipitation of Ammonia salts	6.0	6.0	6.0	6.0
Discard Effluent (% U3O8)	.0007	.0004	.0006	.0006
H2O to slurry (gm)	200	200	200	200
Digest compound	(NH4)2CO3	Na2CO3	K2CO3	NaHCO3
Weight of Digest Compound (gm)	20	20	20	20
Digest Time (hr.)	2	2	2	2
Digest Temperature (°C)	60	90	90	90
Residue Wash (ml)	100	100	100	100
Residue (% U3O8)	.12	.035	.017	.055

2/2

2/1

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YD-2151
Page: 24

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TABLE VII-A

EXTRACTION OF URANIUM AS A SOLUBLE CARBONATE
BY NEUTRALIZATION OF FREE ACID WITH SODIUM CARBONATE
MATERIAL: VITRO SODIUM CARBONATE RESIDUE

ASSAY: .26% U308

DIGEST CONDITIONS: 1/2 HR. AT 90°C AT PH 0.8 - 0.9

	<u>Experiment No.</u>	
	<u>1</u>	<u>2</u>
Weight of sample (gm)	100	100
Na ₂ CO ₃ added to neutralize (gm)	25	25
Digest Time (hr.)	2	2
Digest Temperature (°C)	90	90
Carbonate Residue (% U308)	.13	.12

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TO F. M. Belmore

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