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TECHNOLOGY—FEED MATERIALS

21430

PURIFICATION OF PHOSPHATE RESIDUE FROM

PREVIOUS DIGESTIONS OF URANIUM OXIDES FOR ETHER EXTRACTION

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PURIFICATION OF PHOSPHATE RESIDUE FROM
PREVIOUS DIGESTIONS OF URANIUM OXIDES FOR ETHER EXTRACTION

I. INTRODUCTION

In the production of UO_2 of extreme purity from UO_3 , sodium uranates and other commercial products, certain insolubles containing uranium in considerable quantities were accumulated as filter cake. These products consisted chiefly of uranyl phosphate (probably UO_2HPO_4), due to the phosphate content of the raw material, and of sodium uranate, largely $Na_2U_2O_7$. Large quantities of these products have been accumulated varying from 30 to 60% in uranium content. These products will be referred to hereinafter as 306 residues. The recovery and purification of this uranium is of utmost importance. This paper describes laboratory determinations of applicable reactions and pilot plant operations of some of these methods by the Mallinckrodt Chemical Works.

A consideration of the reactions of UO_2HPO_4 and of $Na_2U_2O_7$ or Na_2UO_4 indicates that the 306 Residues could be dissolved by digestion in HNO_3 , or that the phosphate portion could be dissolved in $NaOH$, filtered off and the residual soda salt dissolved in HNO_3 . The all-acid digestion required larger quantities of expensive acid and a high-acid filtration requiring Vinyon. The alkali-acid procedure, however, required two digestions and a strong alkali filtration, thus doubling the processing labor and offering equal filtration difficulties.

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Laboratory investigation by a different group (1) indicated that the alkali procedure could be used, but the double digestion and filtration process made the procedure undesirable for the present plant; hence, it was not tried in the pilot plant. This work has been fully reported elsewhere. This material varies in composition from 2 to 9% $\text{PO}_4^{=}$ and from 2% to 7% Na^+ . The remainder consists of SiO_2 , ferric compounds and other impurities. Six lots were selected representing residues produced at different times from different raw materials. The composition of these six lots of material is presented in Table 1.

TABLE 1

Composition

	<u>X</u>	<u>PO_4</u>
P-4186VH49	33.87	6.90
P-4188NG36	58.57	9.17
P-4188AT05	47.74	7.70
N-4171RB36	57.23	6.13
N-4171DA06	48.57	2.37
N-4171VE49	59.85	4.98

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Laboratory trials showed that virtually complete solution of the uranium constituents of the residue could be accomplished with 3 to 5 moles excess of HNO_3 over the stoichiometric equivalent of all reactants present with quantities of materials which produced the desired concentration of 250 g/l of uranium. Less than 3N excess of HNO_3 resulted in the recovery of only 60 to 80% of the uranium present in the residue. Recoveries of 95 to 99% were achieved with 3 to 5 N acid. Since filtration at such an acidity is not possible on cotton duck, the solution was partially neutralized with lime. The extent of neutralization and the mode of addition of the lime was found to be quite critical. Neutralization below 2N caused precipitation of UO_2HPO_4 if $\text{PO}_4^{=}$ was present in quantities of 8% or more in the raw material. The resulting $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ could then be purified by ether extraction.

This process was then carried out on the pilot plant scale, as described in the next section of this report, and variations were made in the procedure until it was possible to extract the solution without emulsion or sedimentation difficulties at a normal rate of flow. Freedom from precipitation of phosphate during the extraction process was found to be related to acidity of digestion liquor, acidity of feed, and extent of heating during digestion and subsequent boil-down. Freedom from sedimentation or precipitation on standing was related to extent of neutralization after digestion.

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Emulsification resulted from sedimentation before or during extraction and from inadequate digestion or lack of boildown. Each of these relations will be shown in the treatment of the experimental data, and the procedure employed in the final digestion process described.

II. EXPERIMENTAL

The extraction was conducted by the two-stage ether extraction process employed in the purification of AAA ores, described elsewhere (2).

The first lots of residue used for pilot plant extraction were digested in sufficient nitric acid to produce a slurry 5 molar in excess acid and to contain 2 to 2-1/2 pounds of uranium per gallon of filtered solution (extraction column feed). The acid was brought to a temperature of 180 to 210°F, the residue added, the temperature raised to 200°F to boiling in various cases (see Table IIA) and heated for 30 minutes to 1 hour. Sufficient water was added to produce the desired concentrations of acid and of uranium.

Runs R-4 through R-7 were made in the production unit, where it was necessary to have volumes of 700 to 1000 gallons; hence, filtrations were made on the production presses. It was desirable, therefore, to use cotton duck as the filtration medium. The solution was cooled to about 90°F and filtered on cotton duck. At this temperature and acidity about two sets of duck were ruined per fil-

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tration. Subsequent filtrations were made in the pilot plant and filtered on Vinyon. The washed cake contained from 1.5 to 5% uranium.

The filtrate was then neutralized with lime to produce a solution from 1.2 to 1.8N in HNO_3 . Sufficient $\text{Ca}(\text{NO}_3)_2$ was added to raise the $\text{Ca}(\text{NO}_3)_2$ concentration due to the neutralization to a total concentration of 150 to 300 g/l in various experiments. In some cases $\text{Fe}(\text{NO}_3)_3$ was added as shown in Table 3B.

The adjusted filtrate was extracted with ether by mixing co-currently in the ratio of two volumes of ether, which was 1N in HNO_3 , with 1 volume of feed, decanting off the ether extract and completing the extraction with acid ether in a counter-current packed column. The extract from this column was then used in the co-current mixer. The extract from this acid stage was re-extracted with distilled water, evaporated to molten uranyl nitrate hexahydrate, extracted with neutral ether, and again re-extracted with distilled water. This process has been fully described in a previous report (2).

The extraction feed liquor prepared as above (Experiment R4 through R7) was introduced into the acidified ether-feed mixer at the rate of 20 to 30 gallons per hour. Some lots passed through the acid extraction unit without great difficulty, as shown in Table III, while others emulsified to such an extent that they could be run at only 20 gallons per hour, and even at that rate

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difficulty was experienced with the introduction of too much ether into the de-etherizer, due to slow separation and consequent entrainment of emulsion by the water extract.

Particular difficulty was experienced with feeds which had stood for any length of time. Some precipitation of uranyl phosphate usually occurred in the storage tank, resulting in a cloudy feed. Further precipitation occurred during extraction in the packed column. This precipitate always resulted in emulsification in the column, in some cases producing such a viscous milky emulsion that flow in the columns was practically stopped, resulting in a complete lack of separation of ether extract and raffinate and the passage of emulsion into both the raffinate and extract receivers.

The emulsions formed on precipitated phosphate were ether in water dispersion which became so thick in some cases that gels resulted on standing even momentarily. These milky ether-in-water emulsions were also formed when sulfate precipitates formed in AAA ore and were contrasted with the clear, globular water-in-ether emulsions sometimes produced mechanically or by dissolved stabilizing agents.

As a result of the reduced rate of operation, the lack of purification and the high loss of uranium in the raffinate due to these emulsions, digestion procedures were sought which would avoid

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the precipitation of phosphates and the resulting water-ether emulsions and gels in the column and which would prevent the formation of the ether-water emulsions formed without precipitation.

Several conditions in the digestion procedure were found to be involved in these results, namely, the acidity of the feed in the precipitation of phosphate and the acidity and time of digestion and the maximum temperature reached during the final adjustment of the feed. The following experiments established these relations and determined the optimum conditions for the digestion of residues.

It had been observed in the treatment of AAA ores that a concentration of the feed to molten uranyl nitrate hexahydrate by evaporating until a boiling point of 243°F had been reached prevented the formation of the clear, globular water-in-ether type of emulsion. Hence, a boil-down, or concentration by evaporation to a boiling point of 243°F was introduced following the digestion. $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ was added in varying quantities as the salting out agent and the solutions varying from 5.2 to 5.4N in acidity were neutralized to acidities of 1.4 to 2.14 N.

The addition of lime in one lot caused local neutralization to the point where complete precipitation of the phosphate occurred and the digestion was lost. Further additions of lime were made by small increments, slowly, with stirring.

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The slurries prepared as above were almost unfilterable, the lime causing local alkaline concentrations sufficient to precipitate ferric and other hydrous oxides in very slimy, gelatinous form. It was necessary to break down the one-foot press a number of times for a single filtration. The cake could not be washed at all.

To prevent the formation of a gelatinous slurry by the hydrous oxides, it was filtered before neutralization in Experiments R-12 through R-16. Although slow, the resulting filtration was satisfactory, the cake being granular and washable. Due to the high acidity (ca 5N) filtration was conducted on Vinyon. The filtrate was then concentrated to a boiling point of 245°F , neutralized with $\text{Ca}(\text{OH})_2$ to an acidity of 1 to 1.6 N and again filtered through vinyon on the press.

The solutions adjusted as above for uranium content, acidity, and salting-out agent, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, were extracted by the two-stage acid-neutral extraction process described above. The boil-down prevented the formation of water-in-ether emulsions and some of the feeds were extracted at the normal rate of 30 gallons per hour. However, some feeds which were held over longer between digestion and extraction could not be fed into the columns even at 20 gallons per hour due to phosphate precipitation which had occurred and which caused milky ether-in-water emulsification. To test this hypothesis, a double lot was digested by the last procedure described

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above; half of the lot was extracted immediately and one half permitted to stand for two days before extraction. The first half was extracted at 30 gallons per hour with no difficulty whatever, while the portion which had stood over could not be run even at 20 gallons per hour because of emulsification.

III. DISCUSSION

It has become more and more obvious that success in the purification of uranium by ether extraction and by a continuous process is dependent on the proper preparation of the feed liquor to be extracted. In the experiments described in this report, the following relations to digestion have been established:

Acidity: It is necessary that the digestion medium have an excess acidity of about 5N at the completion of digestion in order to dissolve the phosphate and uranate. The calculation of the acid required was made as follows assuming the reactions indicated:

(The acid salts are indicated by analysis of precipitate.)

Let R pounds be the weight of residue to be digested.

X%	% by weight of uranium ion
P%	% by weight of phosphate ion
S%	% by weight of sodium ion
N%	% by weight of HNO ₃ in acid used

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$$R \text{ lbs.} \times \frac{P\%}{100} \times \frac{\text{UO}_2\text{HPO}_4}{\text{PO}_4} \times \frac{2\text{HNO}_3}{\text{UO}_2\text{HPO}_4} \times \frac{100}{N} \times \frac{1 \text{ gal.}}{12.5 \text{ lbs.}} = A' \text{ gallons acid for phosphate.}$$

$$R \text{ lbs.} \times \frac{S}{100} \times \frac{\text{Na}_2\text{U}_2\text{O}_7}{2\text{Na}} \times \frac{6\text{HNO}_3}{\text{Na}_2\text{U}_2\text{O}_7} \times \frac{100}{N} \times \frac{1 \text{ gal.}}{12.5 \text{ lbs.}} = S' \text{ gallons acid for sodium salt.}$$

The final volume to contain 2.5 pounds of uranium per gallon is

$$R \text{ lbs.} \times \frac{X\%}{100} \times \frac{1 \text{ gal.}}{25 \text{ lbs.}} = X' \text{ gallons of solution.}$$

The acid required to produce a 5N excess in solution is

$$X' \text{ gal.} \times 5 \times 63 \times 0.0083 \text{ lbs./gal.} \times \frac{100}{N} \times \frac{1 \text{ gal.}}{12.5 \text{ lbs.}} = A \text{ gallons of acid.}$$

The total volume of acid required is $A' + S' + A = T$ gallons of acid of $N\%$ HNO_3 required for the digestion. The total volume X' gallons is completed by the necessary dilution with water.

Temperature: Most complete digestion of uranium compounds with less loss in residue was achieved by digestion with boiling. The resulting slurry was also more filterable.

Time: One hour of digestion produced more complete extraction of uranium and more filterable slurry than shorter times. Two hours of digestion improved the filterability of the slurry and the washability of the cake.

Filtration: The cake obtained on filtering residue digest liquors consisted largely of silica, ferric and other hydrous oxides, and undigested phosphate, if any. This is a rather gelatinous cake and filters with difficulty. Neutralization before

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filtration renders the latter almost impossible, while filtration in very high acidity is hard on the filtering medium. However, Vinyon at 90°F or less stands up fairly well under the 5N solution.

Filtration was greatly improved by filtering before neutralization. The formation of gelatinous hydrous oxides was prevented and more granular solids were obtained.

Concentration: In the ether extraction of feed liquors prepared from pitchblende ores, from sodium uranate or from the above phosphate-uranate residues, clear, globular dispersions of water in ether were frequently formed. These dispersions are usually sufficiently rapid in breaking that no difficulty in extraction is caused. At times, however, the dispersion is so well stabilized that flow through the columns is greatly hindered or even stopped. Whether this is due to the presence of a single component, or to a number of different stabilizers, has not been established. It has been found, in all cases, however, (3) that the stabilizing agent is destroyed, or its formation prevented by concentrating the feed liquor until its boiling point is about that of uranyl nitrate hexahydrate, 243°F. It will be observed from Tables II and III that in Experiments R-4 through R-7, in which the maximum temperature reached in the digestion was 200 to 220°F, such emulsions formed, while in Experiments R-9 to R-15, in which the solutions were boiled down to 243°F, no emulsions of this type were encountered.

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Salting-out Agents and Recovery on Extraction: While the greater portion of this work was done with $\text{Ca}(\text{NO}_3)_2$ as the salting out agent, some work was done with $\text{Fe}(\text{NO}_3)_3$. The relative efficiencies were not tested quantitatively, but both salts in the range of 1-1/2 to 3 pounds per gallon of feed were found to give 95 to 99% extraction. The loss of uranium held in ether emulsion in the raffinate was the chief source of difference in recovery. Ferric nitrate was somewhat undesirable, however, due to the hydrolysis occurring on standing and during extraction, even at 1 to 1.5 N, resulting in considerable fouling of the columns and packing. On continuous operation this behavior would not be permissible.

Efficiency of Extraction

The total recovery indicated in the final process is 97 to 99% in the digestion procedure (Table IV), varying with the washability of the reslurry cake and 96 to 99% in the extraction procedure, varying with emulsion in the raffinate. Overall recovery with continuous operation should run from 92 to 98%.

Purification Achieved

The purification achieved is comparable with that of the AAA ore in the acid extraction. For this reason no neutral extraction of 306 Residues was run.

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Final Process Recommended

As a result of the program described above, it is recommended that 306 Residue be digested in sufficient nitric acid to produce a 5N slurry, cooled to about 90°F, filtered on acid-resisting Vinyon, the filtrate neutralized with lime to about 2N in nitric acid, the solution boiled down to a boiling point of 243°F, then sufficient water, $\text{Ca}(\text{NO}_3)_2$ and nitric acid added (if necessary) to produce a feed having about 300 g/l of X, 300 g/l of $\text{Ca}(\text{NO}_3)_2$ and about 0.5 N in HNO_3 .

This solution should then be extracted by the usual acid and neutral stage ether extraction described above (2).

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TABLE 2

DIGESTION AND FILTRATION OF RESIDUUSA. Without Boil-Down (Concentration)

Date	Exp. No.	Wt. Charge lbs	X Content lbs	X Phos. Content lbs	Sodium Iodate lbs	HNO ₃ 38° lbs	Maximum Temp. °F	Digest Time Hrs.	Filtration	Neut. Ca(OH) ₂ lbs	Neutra- lisation Ca(OH) ₂ lbs
9-11-45	R-1										
9-13-45	R-2										
9-13-45	R-3										
11-14-45	R-4	3,500	983			5760	220° F	1		By batches	dry
11-23-45	R-5	3,200	1,800			3600	220° F	1 1/4		By batches	dry
12-19-45	R-6	8,400	3,040			5760	200° F	1		By batches	ss mud wet
1-31-46	R-7	10,695	2,650			6920	200° F	1	100° F on cotton	850	850 wet cake and 6. mud

B.

B.									With Boil-Down But Neutralisation Before Filtration					
									Boil-Down Temp. °F	CaNO ₃ Sp.Gr. 1.54, gal.	Normality	Ca(OH) ₂ Added lbs	Filtration	Final Norm.
2-28-46	R-8	256	150	60	193	450	211	1	243	11	5.16	70	*	-- whole chg ppt
3-18-46	R-9	256	160	60	193	450	210	1	243	5.5	5.40	59	50 gal/hr	1.73
3- 2-46	R-10	256	160	60	193	450	212	1	243	5.5	5.36	69	Very slowly	2.14
3- 4-46	R-11	256	160	60	193	592	212	1	243	0	5.20	52	Very slowly	1.40

C.

C.									With Boil-Down But Filtration Before Neutralisation					
									Filtration	Boil-Down Temp. °F	Normality	Ca(OH) ₂ Added lbs	Normality	Filtration
3- 6-46	R-12	314	160	93	192	460	212	1	Very poor	243	6.25	42	1.02	Good
3- 7-46	R-13	314	150	93	192	460	220	2	Very poor	243	5.31	43.5	1.56	Good
3- 8-46	R-14	314	160	93	192	450	220	2	<15 gal/hr	243	4.41	22	1.39	Good
3-12-46	R-15	496	237	147	304	710	222	2	<20 gal/hr	243	4.60	34		Good

* The Ca(OH)₂ in R-8 was added as the powder from a scoop, the last 50 lbs. being added at once from the bag. At the last addition the solution precipitated solidly and had to be blown out with a hose to the recovery system.

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TABLE 3

CONTINUOUS EXTRACTION OF SO6 RESIDUES IN PILOT PLANT

Exp.No.	Feed sp.gr.	Feed Composition			Ppt. on Standing		Operational Data			Maximum Feed Rate	Comments
		Assay lbs/gal	Excess HNO ₃ N	Calcium Nitrate lbs/gal	Time Hours	Amt. (Quan)	Emulsification		Nature		
							Decant	Columns			
R4A	1.42	---	---	---	---	---	None			30	Extracted at 30 gph without emulsification.
R5A	1.52	2.24	1.62	1.27	---	---	None			30	No emul. noted during run - same material used 3 days later emulsified.
R6A6	1.425	1.39	1.22	1.94	---	---	None			30	Exp's in R6 series (determined effect of iron salting) & mixing pump operation.
R7A	1.50	1.58	1.55	2.50	---	---	Not noted	Both	Fish egg	20	Extracted at 20 rate despite emul.
R9	1.620	1.88	1.75	3.52	20	---	Slight	1st	Sed.	30	Starting rate 30 gph emulsified. Reduced to 20 ran good - up to 30 gph - continued good. Sedimentary deposit in column 44% x 14% PO ₄ .
R10	1.685	1.50	2.14	3.71	47	Great	Not noted	1st	---	30	Raffinate slightly emul. emul. prod. sed. type. 18 hours later same material ran with heavy deposition of PO ₄ accompanied by emulsion.
R11	1.675	1.98	1.40	2.91	20	---	None	None	None	30	No emulsification - excellent run.
R12	1.657	2.15	1.02	3.19	22	---	Slight	Mod.	Sed.	20	Heavy yellow ppt. - reduced rate permitted operation.
R14	1.618			1.17	70	None	Mod.	Mod.	Not noted	20	Emulsified - operation however not hindered.
R15				1.68	2 1/2	None	None	Mod.	Fish egg	30	
R15A				1.68							

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TABLE 4EFFICIENCY OF EXTRACTION AND RECOVERY~~SECRET~~

Exp.No.	Feed Rate gph	Emulsi- fication	Recovery		Purification						I Content Raffinate lbs/gal
			Loss in Raffinate % U	Recovery % U	MoO ₃		P ₂ O ₅		SiO ₂		
					Feed ppm	NOK ppm	Feed ppm	NOK ppm	Feed ppm	NOK ppm	
R4A	30	Not noted	4.6	95.4	---	68	---	1800	---	---	0.053
R5A	30	None	5.1	94.9	---	---	46400	---	---	---	0.111
R6A6	30	Note noted	9.3	90.7	---	---	---	---	---	---	0.130
R7A	20	Heavy	0.95	99.0	8160	1360	70000	4390	2210	43	0.016
R9	30	Moderate	5.7	94.3	725	73	94600	2820	27	---	0.107
R10	30	Slight	1.7	98.3	722	83	147000	6100	155	---	0.025
R11	30	None	3.5	96.5	884	75	123000	8700	151	---	0.070
R12	20	Moderate	2.9	97.1			128500	14600	2280	---	0.062
R14	20										
R15											
R15A											

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BIBLIOGRAPHY

- 1) Robles, Edward G., Jr., January 1946: "A Report on the Recovery of X from Residues Containing Large Amounts of Phosphate."
- 2) Opie, John V., Yeager, Harold, and Keller, W. H., "Development of a Continuous Ether Extraction Process for Purification of Uranium Derived Directly from Pitchblende Ore", Mallinckrodt Chemical Works Research Report No. 19.

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