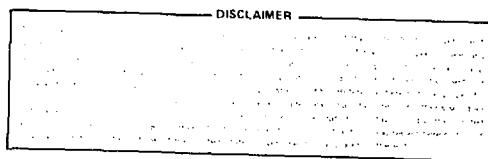


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URANIUM CONTROL IN PHOSPHOGYPSUM*

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Uranium Control in Phosphogypsum*

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Wet-process phosphoric acid plants produce about 4.5 tons of gypsum for each ton of P_2O_5 . About 24 million tons of gypsum is produced at phosphoric acid plants in the United States each year, and about 225 million tons is contained in stockpiles from production in previous years. While most of the uranium initially present in phosphate rock is dissolved when the rock is acidulated to produce phosphoric acid, a fraction of it remains undissolved and the gypsum contains 0.03-0.08 pounds of uranium per ton. Uranium concentrations in filter cakes produced by the hemihydrate process are significantly higher (as much as 0.20 lb/ton). These cakes hydrate to gypsum as they age and most of the uranium becomes readily soluble in the process; the concentration of insoluble uranium in the hydrated cake is about the same as that in filter cake produced by the dihydrate process. Even at these low concentrations, the gypsum produced at phosphoric acid plants each year contains 0.9 to 2.4 million pounds of uranium, and the stockpiled gypsum may contain a total of 7 to 14 million pounds.

Economical recovery of uranium from gypsum appears very unlikely in the foreseeable future. However, the loss to the filter cake can possibly be reduced by operating the phosphoric acid plant under conditions that maximize uranium dissolution during rock acidulation. In studies of this

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problem at ORNL and at other places, the major variable that affected the distribution of uranium between phosphoric acid and gypsum during the manufacture of wet-process H_3PO_4 was the redox potential of the slurry during acidulation. Other variables that affected the uranium distribution included rock size, digestion temperature, and concentrations of excess H_2SO_4 and fluoride. This paper describes some of the problem areas and possible approaches to their solution. Much additional information is needed.

Introduction

The more than 50 million tons of phosphate rock processed in Florida during 1980 is estimated to contain over 10 million lbs of uranium. By 1981, about half of this uranium will be recovered in six wet-process phosphoric acid plants.¹ Recovery of this uranium is very difficult and costly and can be done economically only as a by-product of wet-process phosphoric acid production. Thus, it seems only logical to try to dissolve as much uranium as possible during rock acidulation. Previous data, obtained during the 1950s when 3 plants recovered uranium from wet-process phosphoric acid showed that only 60 to 80% of the uranium originally present in the phosphate rock reported to the acid and that the remainder reported to the gypsum residue.

This paper reviews the early data, much of which had limited distribution, with emphasis on the variables that were considered to affect uranium distribution between the acid and the gypsum. It also includes more recent test results that confirm the early data and describes an alternative route that may be particularly attractive for hemihydrate processes.

Description of the Problem

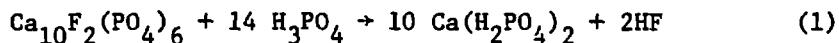
The current stockpile of phosphogypsum in Florida has been estimated at ~225 million tons, and it is growing at the rate of ~24 million tons per year.² No one knows how much uranium is contained in this stockpile of gypsum, but a reasonable estimate may be made by assuming 60 to 80% dissolution of uranium indicated by the results of early studies and the results of a few analyses of gypsum performed recently at ORNL. On this basis, we estimate a concentration range of 15 to 30 ppm uranium, which indicates 7 to 14 million lb of uranium in the stockpile. Assuming a uranium price of \$30/lb, the value of this uranium is \$0.90 to \$1.80/ton of gypsum.

It is very doubtful that this uranium can be recovered economically once it is incorporated into the gypsum. Thus, it becomes very important to divert it all to the acid (or to the gypsum, for subsequent recovery) during the rock acidulation.

Chemistry of the Process

The production of wet-process acid involves digesting a slurry of phosphate rock with sulfuric acid and separating the resulting phosphoric acid from the solid products of the reaction by filtration. The two major methods in use today are the dihydrate and hemihydrate processes, so named for the mode of calcium sulfate precipitation. The dihydrate process is by far the most widely used, but interest in hemihydrate processes is growing because of large potential savings in energy and capital costs.³

The overall reactions of the dihydrate and hemihydrate processes are essentially the same and may be represented as a two-step reaction. Equation 1 shows the dissolution of the phosphate rock in phosphoric acid to form monocalcium phosphate solution,



and Equation 2 shows the reaction of sulfuric acid with the monocalcium phosphate to produce a hydrated calcium sulfate which can then be separated from the phosphoric acid by filtration.



Depending on the operating conditions selected, the calcium sulfate can be crystallized as the dihydrate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) or as the hemihydrate ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$). In the first case the liquid phase will contain 28 to 30% P_2O_5 and in the latter case, it will contain 40 to 50% P_2O_5 . As we will see later, the mode of crystallization has a very important bearing on the distribution of uranium between the acid and the cake.

Early Work

As early as 1954, Shaw⁴ reported that in most phosphate plants only 60 to 80% of the uranium originally present in phosphate rock reported to the acid during the manufacture of wet-process phosphoric acid, and that the remainder reported to the gypsum residue. This high distribution of uranium to the gypsum residue led the Dow Chemical Company into an investigation of when and how the uranium was precipitated with the gypsum during acidulation. As the first step in their study, the rock dissolution step (Equation 1) and the crystallization step (Equation 2) of the acidulation reaction were studied separately using both oxidizing and reducing conditions. The tests were then repeated with the two reactions being carried out simultaneously. The effects of excess fluoride and excess sulfate were also studied.

Figure 1 summarizes the Dow acidulation tests: "As the reaction proceeded under oxidizing conditions, the uranium recovery into solution paralleled the phosphate recovery. Under normal conditions, however, the uranium recovery lagged far behind the phosphate recovery, being only 40% at 88% recovery of P_2O_5 . Under reducing conditions, the uranium recovery was worse. Only 3% to 5% of the uranium was recovered at 70% P_2O_5 recovery, and only 31% at 88% P_2O_5 recovery."

Dow concluded that uranium is present in phosphate rock primarily as U(IV) and that uranium losses to the filter cake are caused by gypsum coating of unreacted rock particles and substitution of uranium in the crystal lattice of the gypsum. To improve uranium dissolution, Dow recommended finer grinding of the rock, minimizing the local excess of sulfuric acid during acidulation and maintaining oxidizing conditions during acidulation.

The Blockson Chemical Company studied the distribution of uranium in their process for producing technical grade sodium phosphates⁵. On the basis of these studies, they also concluded that oxidized uranium is more soluble than reduced uranium in phosphate solutions. They reported over 90% dissolution of uranium with oxidizing conditions in acidulation, and over 95% if a small quantity of nitric acid was substituted for an equivalent quantity of sulfuric acid during the digestion.

Blockson calcined their rock before digestion to destroy organic matter. They discovered that oxygen was scavenged from the system during this step and produced reducing conditions. This increased the distribution of uranium to the gypsum to over 30% when the calcined rock was digested. They concluded that this was caused by the substitution of U(IV) for Ca in the crystal lattice of the gypsum. Subsequent leaching tests indicated that recovery of uranium from gypsum required complete dissolution, and that the costs for this step were higher than the value of the uranium.

Blockson investigated two approaches to minimize the distribution of uranium to gypsum. Their first approach was to maintain oxidizing conditions during digestion of the rock. The oxidizing agents tested were air, oxygen, ozone, chlorine, nitric acid, permanganates, persulfates, chromates, hydrogen peroxide, and chlorates. All were effective, some

more than others, but uranium oxidation was not selective. Thus, all ions present in a reduced state plus any organic matter also had to be oxidized. This increased operating costs to a point that they offset the value of the extra uranium recovered. Their second approach was to calcine the rock in an oxidizing environment. Under optimum conditions, about 85% of the uranium reported to the acid. The costs of increasing recovery to 95% was more than the value of the extra 10% uranium recovered.

In 1968, the Chemical Separations Corporation reported a study in which they tried to divert the uranium to the gypsum by acidulating phosphate rock under reducing conditions.⁶ Once the uranium was distributed to the gypsum, they planned to recover it from a gypsum-water slurry using Resin-In-Pulp ion exchange.

In one experiment they mixed two 10g samples of Florida phosphate rock with 50% sulfuric acid for one hour after adding an iron nail to one sample and 1 gram of sodium chlorate to the other. After filtration, washing, and drying, the gypsum from the test made under reducing conditions contained 165 ppm uranium compared to only 15 ppm in the gypsum from the test made under oxidizing conditions. Although this was a simple test, it further confirms and emphasizes the importance of redox potential on the distribution of uranium between the acid and the filter cake.

The data in Table 1 shows the wide day-to-day variation obtained on the distribution of uranium between the acid and the gypsum at a phosphate plant during the month of December 1952.⁷ In this period, as much as 92% and as little as 51% of the uranium was found in the acid. The average distribution was 73% which is within the range reported by Dow.⁴

Distribution Profile in a Phosphate Plant

Figure 2 shows recent data on the distribution of uranium to gypsum in a phosphate plant in Florida. This plant has two identical trains for producing wet-process acid which are fed from a common rock supply. Operating conditions are reportedly the same for the two trains. Yet, in spite of these similarities, the concentration of uranium in the gypsum from the south train is approximately twice that from the north train (~34 ppm U compared to ~17 ppm U on an as-received basis). To date, no reason has been found for this anomaly. The results indicate the need for additional study in order to get a better understanding of the factors that control uranium distribution in a plant.

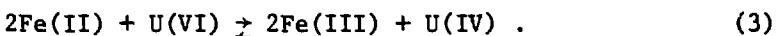
Uranium in Apatite

The key to the erratic distribution of uranium to filter cake may be related to the nature of its occurrence in the phosphate rock or apatite. Altschuler⁸ found that tetravalent uranium was the predominant species in eleven apatite samples examined (Table 2). From 40 to 91% of the uranium (average 65%) was present as U(IV), with the remainder presumed to be present as U(VI). The ionic radius of U(IV) (0.97 Å) is almost identical to that of Ca(II) (0.99 Å) and it is assumed that U(IV) substitutes for Ca(II) in the apatite structure. Altschuler points out that a uranium content of 0.01% in apatite is equivalent to only one atom of uranium for every 26,620 calcium atoms. Furthermore, the positive charge excess can easily be compensated by other known replacements of greater magnitude.

Altschuler⁸ also pointed out that U(VI) can be held in apatite by chemisorption as $\text{UO}_2 \text{HPO}_4$ or by exchange with surface calcium. In the latter mode, it is assumed that 2 moles of calcium are displaced for each mole of UO_2^{+2} taken from solution and 2 moles of phosphate are rendered

nonexchangeable. This would require the uranium to exist as a pyro-phosphate, $\text{UO}_2(\text{HPO}_4)_2$, which is less likely than the chemisorption theory.

As apatite is decomposed and dissolved in a phosphoric acid -- sulfuric acid media, phosphate ions go into solution and U(IV), U(VI), Fe(II), and Fe(III) ions are released. Once in solution the relative amount of these ions are controlled by the following relationships:



The work of Baes,⁹ showed that ferrous iron can readily reduce U(VI) to U(IV) especially at the concentration of phosphoric acid in the attack tank and that the reduction is catalyzed by fluoride ion from the rock. Since Fe(III) and U(IV) form very stable complexes with fluoride and orthophosphate ions, there is a strong tendency for more U(IV) ions to form in addition to those present in the rock. These factors make it easier for uranium to substitute for Ca ions which are being released and become available for reaction with sulfate ions to form $\text{CaSO}_4 \cdot \text{XH}_2\text{O}$ crystals. Free or excess sulfate and fluoride ions can also influence the cocrystallization of uranium in the CaSO_4 crystal. Obviously the mechanism of cocrystallization is very complicated and needs additional study.

Hemihydrate Processes

Uranium recovery from the more concentrated (40 to 50% P_2O_5) acids produced by hemihydrate processes is much more difficult than recovery from the conventional (28 to 30% P_2O_5) acids produced by dihydrate processes. For example, in extraction of uranium from phosphoric acid with DEPA-TOPO,^{10,11} the extractant of choice for most operations involving uranium recovery from dihydrate acids, the uranium extraction coefficient decreases as

the inverse 5th power of the acid concentration. The data in Figure 3 shows that it is necessary to use a very high (and expensive) extractant concentration ($\sim 1\text{M}$ DEPA- 0.25M TOPO) to obtain coefficients in the minimum usable range of 1 to 2 when extracting from $\sim 40\%$ P_2O_5 acids. Since, coefficients for the upper (50% P_2O_5) range are less than one, DEPA-TOPO is not an effective extractant for uranium from these strong acids.

Preliminary tests with our alternate OPAP (octylphenyl acid phosphate) extractant indicate that it has sufficient extraction power to be an effective extractant from these acids at least the lower concentration range of hemihydrate process acids. For example, the data in Figure 4 shows that the extraction power for 0.5 M OPAP is about a factor of ten higher than that obtained with 1M DEPA- 0.25 M TOPO. However, the OPAP extraction system is plagued by stability problems that need to be resolved before an effective process can be realized.¹² Our program on OPAP development at ORNL has been terminated, but is being continued by TVA at Muscle Schoals, Alabama. Also, Earth Sciences is operating a uranium recovery facility at Western Coop's phosphate complex in Calgary which uses OPAP in the first cycle of extraction. Hopefully, this work will lead to a resolution of these problems.

During our initial testing of hemihydrate process acids, we observed that the concentration of uranium was significantly below the levels expected. A further analysis of the problem led to the discovery of unusually large quantities of uranium in hemihydrate process filter cakes from the hemi-process. For examples, the data in Table 3 shows 61 to 101 ppm uranium in filter cakes from two hemihydrate process plants, as compared to 15 to 36 ppm in filter cakes from four dihydrate process plants.

In an effort to resolve this variance, we carried out a few cursory tests to determine the variables that may affect the distribution of uranium between the acid and the filter cake during the manufacture of hemihydrate acid. On the basis of past information, the redox potential was considered to be the most important variable. However, in view of the higher distribution of uranium to hemi-cakes than di-cakes, other factors such as temperature, crystal habit, and crystal size distribution may be involved. In addition, hemihydrate can precipitate in clusters or agglomerates which may tend to carry down more of the uranium than the dihydrate.

These preliminary batch tests were made following the procedure used in TVA's foam process.¹³ Our test conditions were:

1. Mix 15 g of finely ground phosphate rock with 30 g (23 mL) of 26% P_2O_5 wet-process acid in a 200 mL Berzelius beaker immersed in a heating bath.
2. Add an oxidant ($NaClO_3$) or a reductant (iron metal).
3. Add 7 ml 98% H_2SO_4 dropwise over a period of 30 min (mole ratio - $H_2SO_4:CaO=1:1$).
4. Allow 1 h for reaction and digestion.
5. Filter the slurry on a 5.5 cm Whatman #40 filter paper and Buchner funnel.
6. Wash the cake with water (or ethanol).
7. Air-dry the cake.

The data in Table 3 shows that as the digestion temperature of the phosphate rock in sulfuric acid was increased from 65° (dihydrate temperature) to 98°C (hemihydrate temperature), the fraction of uranium that reported to the cake increased from 12 to 31%.

In subsequent tests made at 98°C, only 20% of the uranium reported to the acid when the reaction was made under strongly reducing conditions compared to 98% when the reaction was made under strongly oxidizing conditions (Table 4). In the tests made under oxidizing conditions, most of the organic matter was decomposed during digestion and a very clean acid was produced. The use of strongly oxidizing conditions in the attack tank could minimize the acid pretreatment required prior to uranium recovery by solvent extraction.

In other tests at 98°C, 55% of the uranium remained in the cake after it was washed with ethanol compared to 31% when it was washed with water, indicating that some uranium is released from the cake as it is hydrated. This phenomenon was also observed when a sample of wet filter cake that initially contained 101 ppm uranium was filtered to remove solution that had separated after the cake had aged for 5 months. The cake, after air-drying, contained 46 ppm uranium compared to 101 ppm uranium initially, and the solution removed from the cake contained 197 ppm uranium and 180 g/L phosphate.

Following this analysis, we made a few tests to determine the ease in which uranium could be leached from plant samples of hemihydrate filter cake. The data in Figure 5 shows that approximately 60% of the uranium was easily washed from air-dried filter cake with water or dilute phosphoric acid. Additional tests at variable temperature with 3 M phosphoric acid, which gave slightly better dissolution than water or 5 to 7 M acid, showed no change in solubility over the 15° to 75°C range tested (Figure 6). The dissolution was increased to 80% by increasing the digestion time from 1 to 8 h but there was little, if any, improvement beyond 8 h (Figure 7). Although we have no tests to prove it, we assume that recovery of the final 20% of the uranium would require complete dissolution of the cake.

Because of the difficulty of recovering uranium from the stronger acids and the higher distribution of uranium to the filter cake in hemihydrate processes, there may be a potential process advantage if most of the uranium could be diverted to the hemihydrate cake rather than the acid. The uranium would be dissolved subsequently in a dilute phosphoric acid wash stream which could be easily processed to recover the uranium. This possible alternate route to uranium recovery is shown in Figure 8¹⁴ as a revision of the Nisson Hemihydrate process. To be attractive, additional research is needed to improve the distribution of uranium to the cake and to increase its release from the cake on hydration.

Conclusions

Both previous and recent test results show that uranium dissolution from phosphate rock is significantly higher when the rock is acidulated under oxidizing conditions than under reducing conditions. Excess sulfate and excess fluoride further enhance the distribution of uranium to the cake. Apparently the U(IV) present in the crystal lattice of the apatite plus that formed by reduction of U(VI) by Fe(II) during acidulation is trapped or carried into the crystal lattice of the calcium sulfate crystals as they form and grow. The amount of uranium that distributes to hemihydrate filter cake is up to seven times higher than the amount that distributes to the dihydrate cake. About 60% of the uranium in hemihydrate cakes can be readily leached after hydration of the cake, but the residual uranium (20-30%) is very difficult to remove economically. Much additional research is needed to develop methods for minimizing uranium losses to calcium filter cakes.

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ORNL WS-13703

Table 1

DISTRIBUTION OF U IN A COMMERCIAL PLANT

DATE	FRACTION OF U IN ACID
12-3	0.92
5	0.91
7	0.67
12	0.55
13	0.51
14	0.56
15	0.81
16	0.83
17	0.82
AVERAGE	0.73

Table 2

U(IV) IN BONE VALLEY APATITE SAMPLES¹

SAMPLE	U CONCENTRATION (%)	FRACTION OF U AS U(IV)
BLACK PEBBLE	0.010	0.91
DARK PEBBLE	0.0089	0.63
FINE PEBBLE COMPOSITE	0.016	0.81
4-8 mm PEBBLE COMPOSITE	0.032	0.50
	0.022	0.50
	0.015	0.42
	0.021	0.48
APATITE PELLETS	0.011	0.45
	0.009	0.67
	0.0075	0.40
	0.007	0.43

¹Z. S. ALTSCHULER, R. S. CLARKE, JR., AND E. J. YOUNG,
GEOLOGICAL SURVEY PROFESSIONAL PAPER 314-D.

Table 3

**IN WET-PROCESS PHOSPHORIC ACID PLANTS, MORE
URANIUM DISTRIBUTES TO HEMIHYDRATE FILTER
CAKES THAN TO DIHYDRATE FILTER CAKES**

PLANT	TYPE	U IN FILTER CAKE (ppm)
A	Di	15
B		10
C-1		12
C-2		13
D-1		18
D-2		36
X	HEMI	101
Y		61

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Table 4

EFFECT OF DIGESTION TEMPERATURE ON U DISTRIBUTION

DIGESTION TEMPERATURE (°C)	U IN FILTER CAKE	
	CONC., ppm	% OF TOTAL
65	26	12
85	43	21
98	74	31

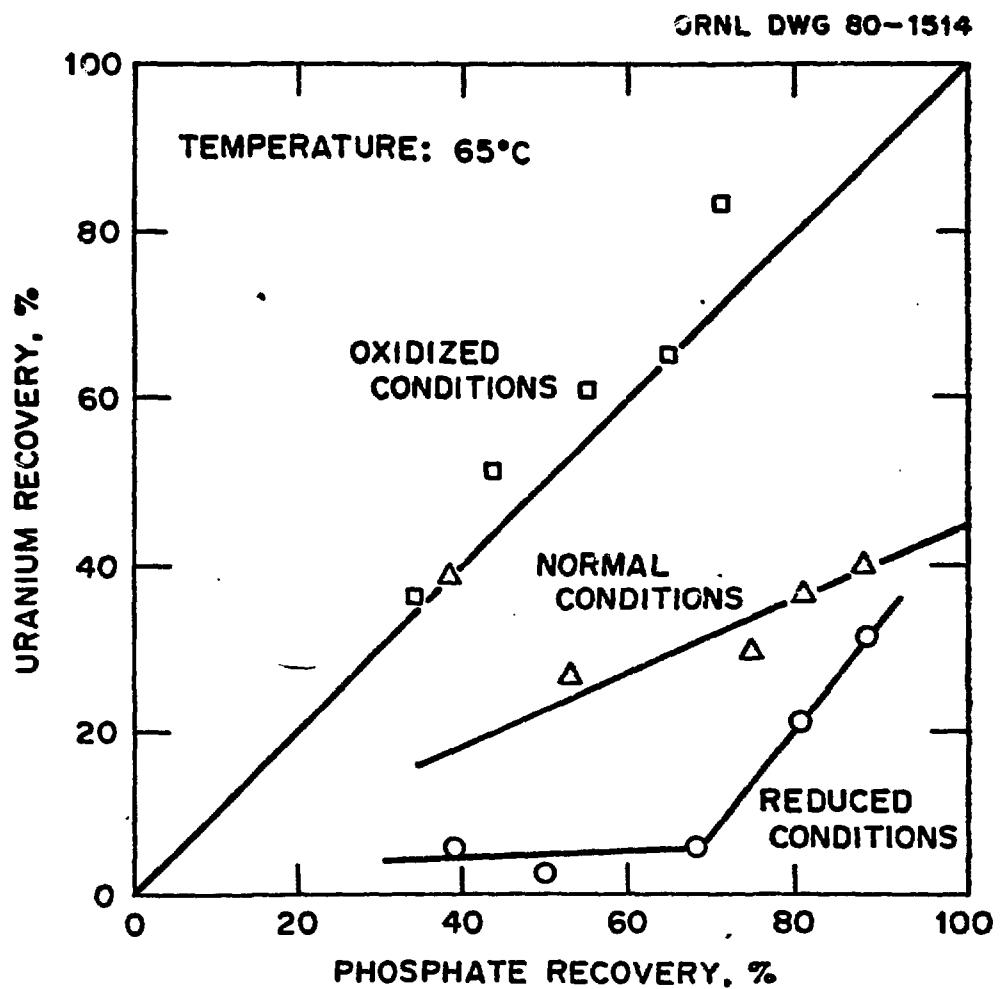
Table 45.

EFFECT OF REDOX POTENTIAL ON U DISTRIBUTION

ADDITIVE	EMF ^a	CONC., mg/ml		U IN FILTER CAKE	
		Fe ²⁺	Fe ³⁺	CONC., ppm	% OF TOTAL
NaClO ₃ , 10%	+0.950	<0.0001	15.9	5.0	2
NaClO ₃ , 2%	+0.860	<0.0001	19.4	5.7	2
NONE	+0.260	1.2	15.3	74	31
Fe°, 0.33%	+0.245	5.7	13.2	73	29
Fe°, 7%	+0.110	42.3	0.4	238	79

^aGOLD vs Ag-AgCl

DIGESTION TEMPERATURE - 98°C



K. G. Shaw, DOW-111 (1954)

~~Figure 1~~ RECOVERY OF PHOSPHATE AND URANIUM
DURING ACIDULATION OF PHOSPHATE ROCK

ORNL-DWG 80-1531

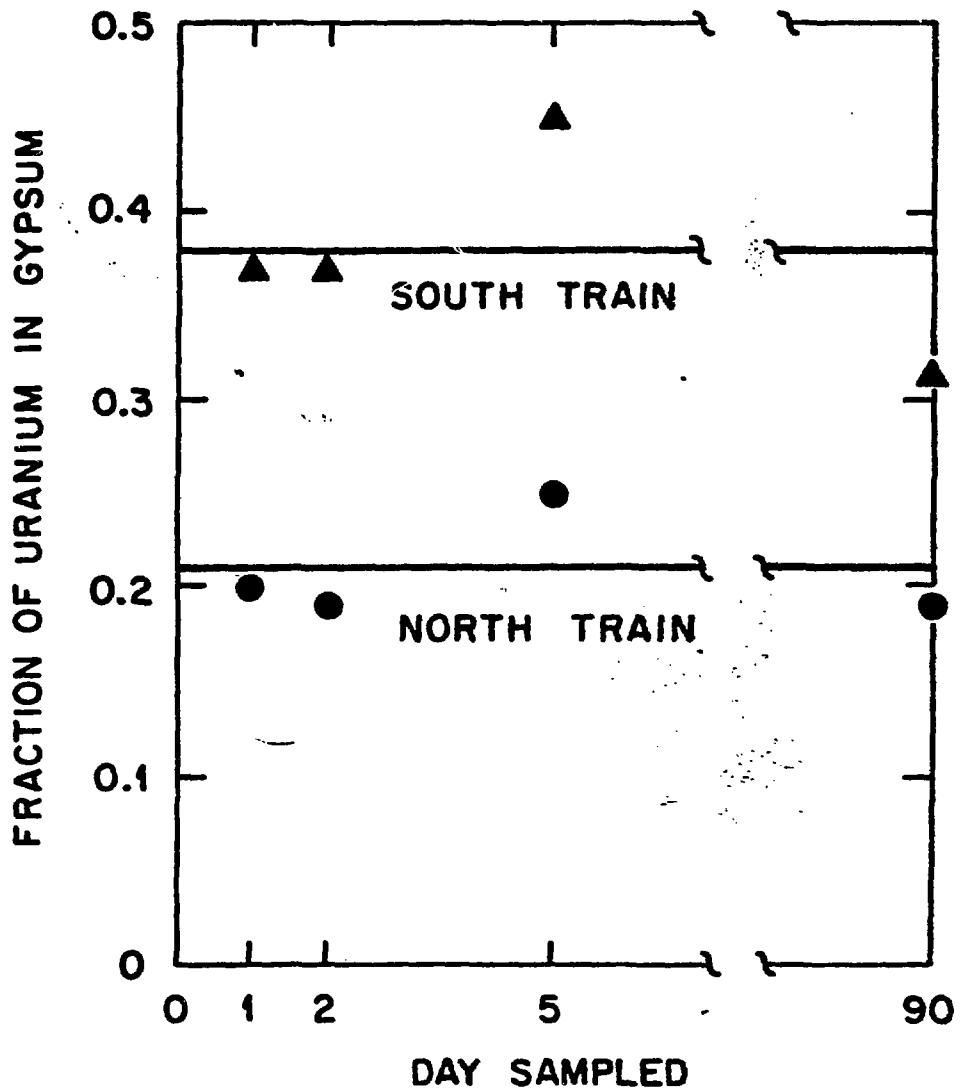


Figure 3: URANIUM DISTRIBUTION TO FILTER CAKE FROM A WET-PROCESS PHOSPHORIC ACID PLANT

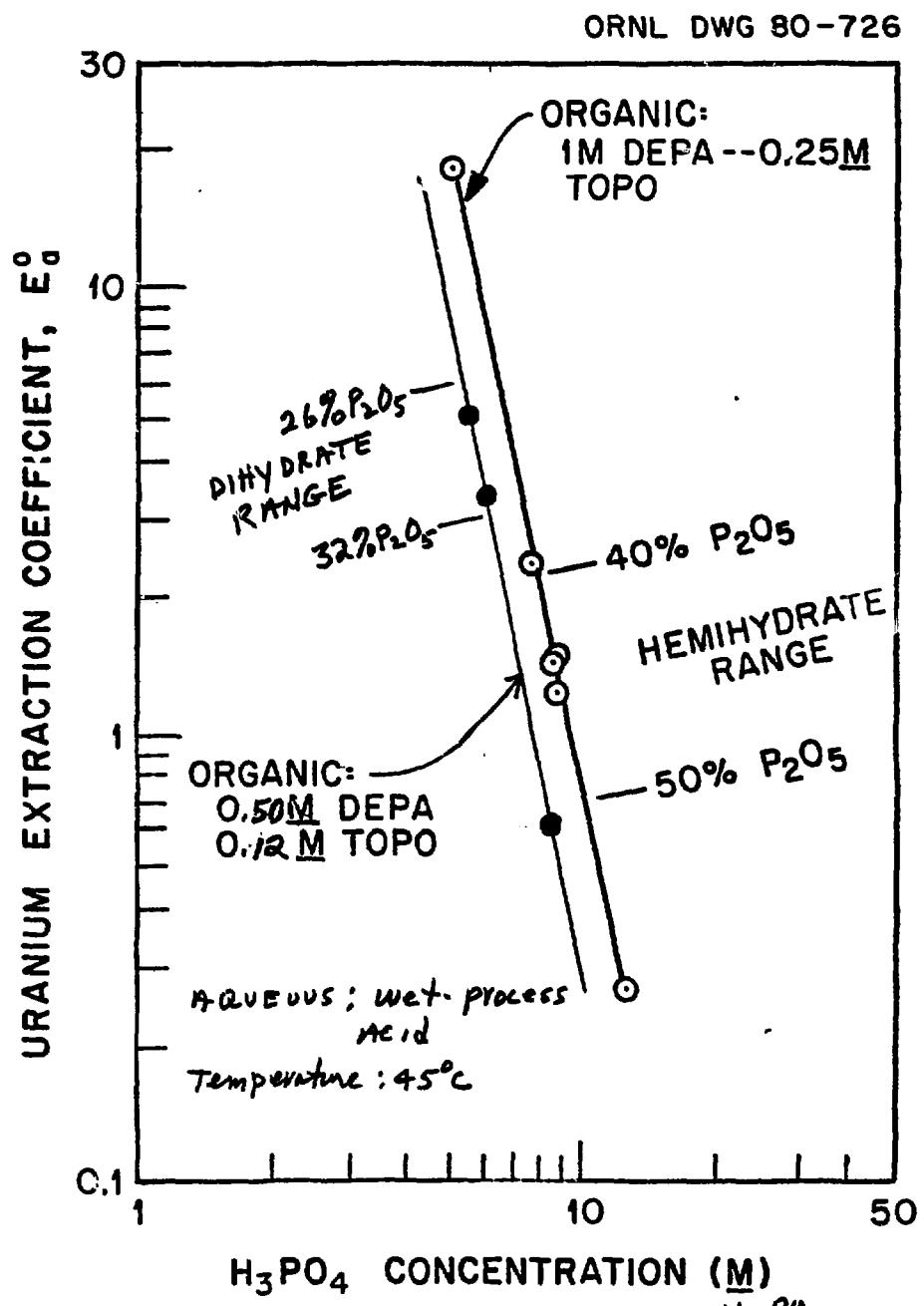
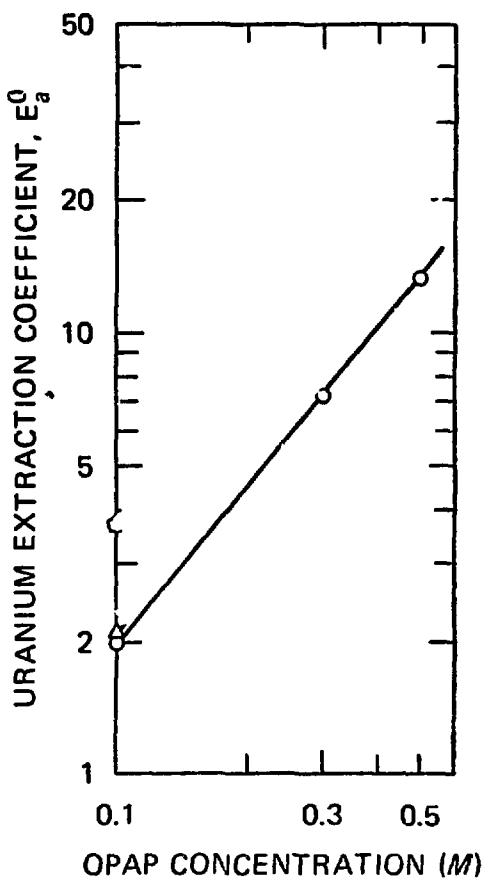


Figure 3. Effect of phosphoric acid concentration on uranium Extraction by DEPA - TOPO



~~Fig. 4~~ ~~URANIUM EXTRACTION WITH OPAP~~ ~~Temp.~~
~~ORGANIC PHASE~~ ~~OPAP IN N-DODECANE~~
~~AQUEOUS PHASE~~ ~~WET PROCESS PHOSPHORIC ACID~~
~~○ HEMIHYDRATE PROCESS, 38% P_2O_5 , 0.11 gU/liter, 12 gFe/liter~~
~~△ DIHYDRATE PROCESS, 30% P_2O_5 , 0.17 gU/liter, 13 gFe/liter~~
~~○ DIHYDRATE PROCESS, 30% P_2O_5 , 0.12 gU/liter, 8.6 gFe/liter~~
or 8

Figure 4. Effect of OPAP Concentration
 (38% P_2O_5)
 on uranium Extraction from wet-process H_3PO_4
 produced by Hemihydrate Process

ORNL DWG 80-1516

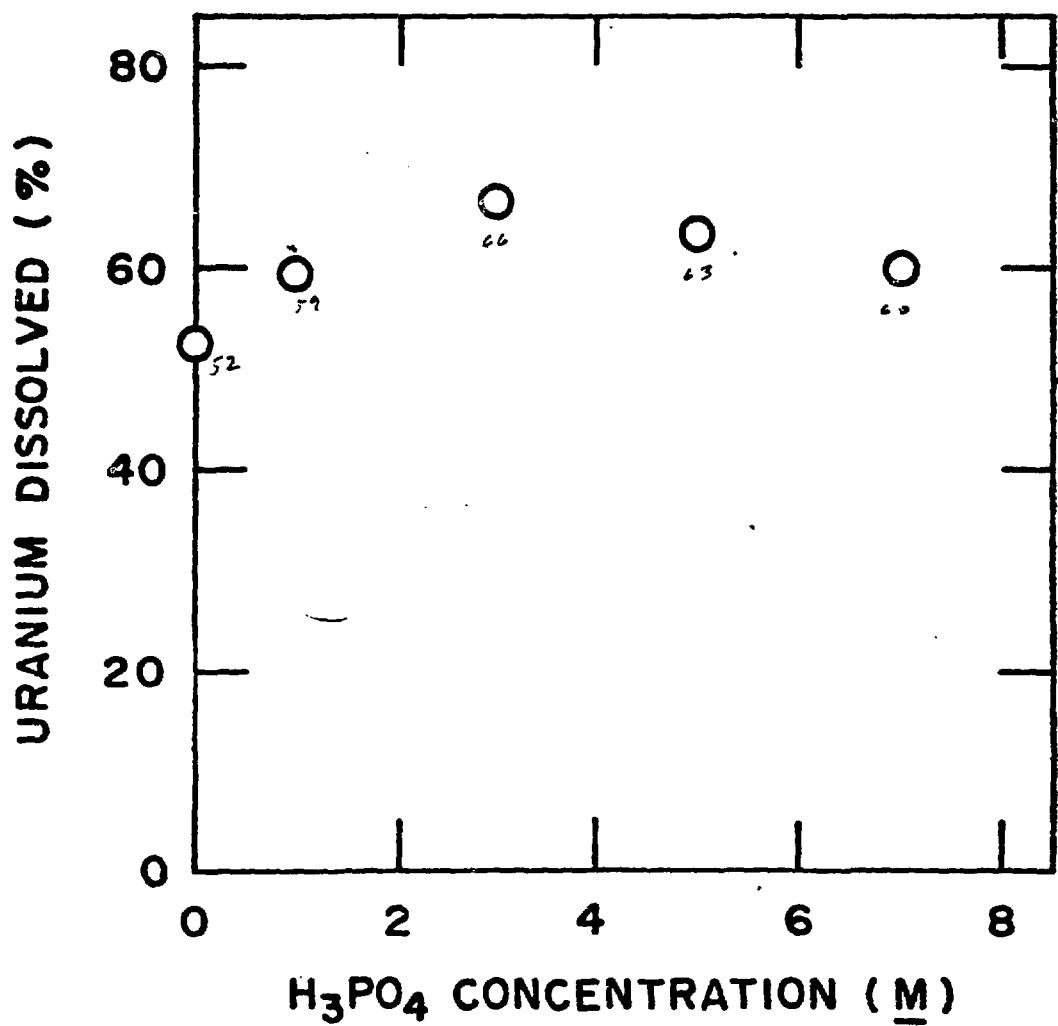


Figure 5 EFFECT OF H_3PO_4 CONCENTRATION

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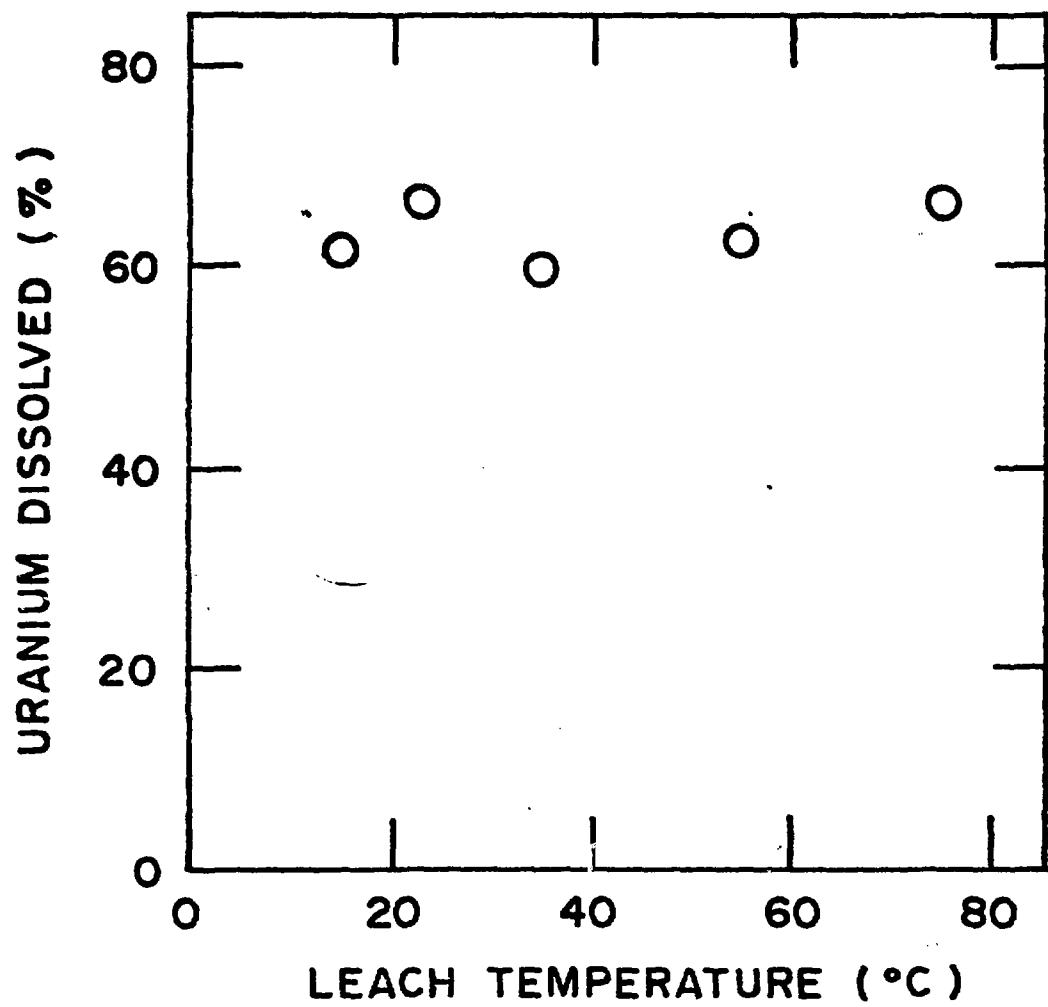


Figure 6. EFFECT OF TEMPERATURE

219"

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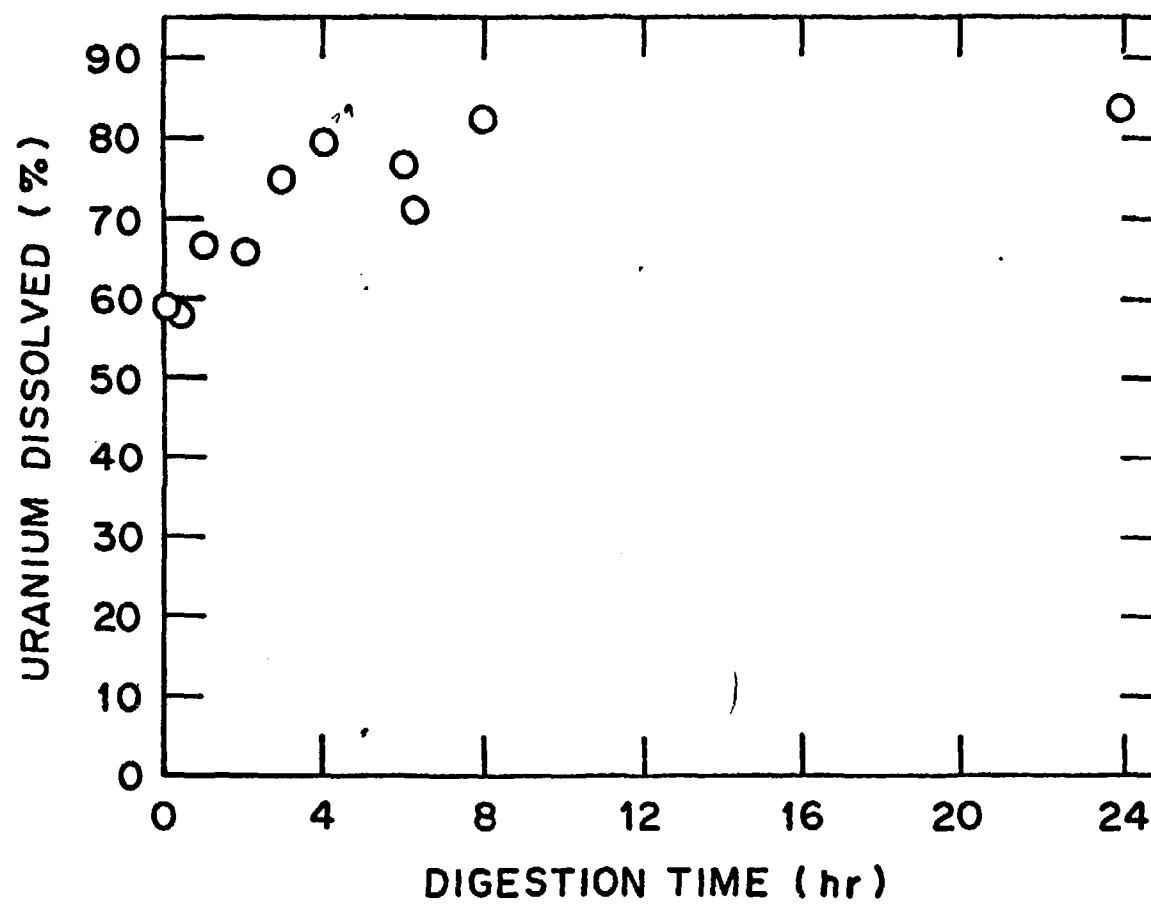
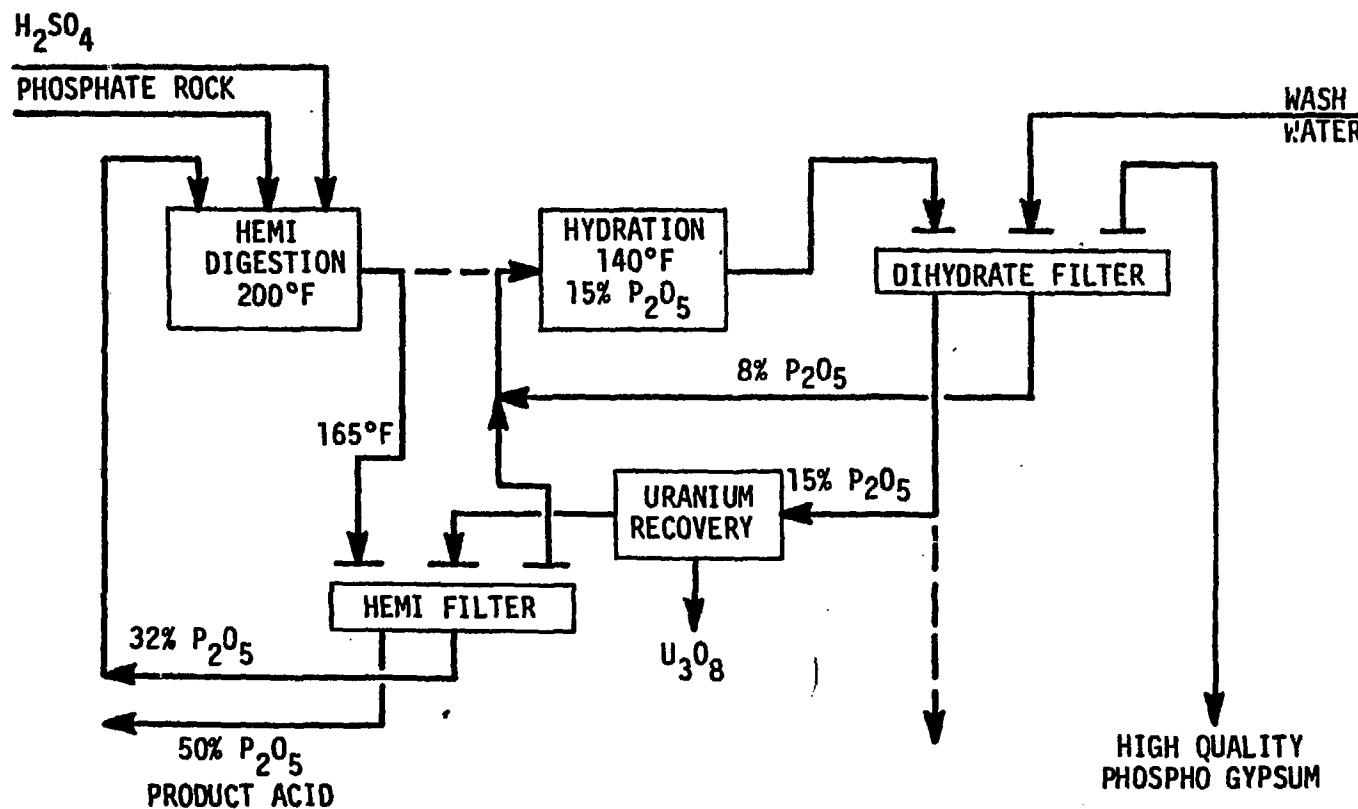


Figure 7 EFFECT OF DIGESTION TIME

fig 8



W. E. Goers, Fertilizer Round Table (1978)

Figure 8 ALTERNATE ROUTE FOR URANIUM
RECOVERY FROM HEMI-HYDRATE ACID

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