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*\*In 1982, the name of the International Superphosphate Manufacturers' Associations (ISMA) was changed to International Fertilizer Industry Association (IFA).*

URANIUM FROM PHOSPHORIC ACID - THE EARTH SCIENCES PROCESS

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SUMMARY

In the wake of the current energy crisis, uranium is the most valuable by-product the phosphate fertiliser industry has the potential to produce, merely by extending existing operations and without creating significant environmental difficulties which are associated with new mining facilities. The main features of the process developed by Earth Sciences Incorporated and operated in its plant in Calgary, Alberta, Canada are outlined. The process is based on two cycle solvent extraction of uranium from wet processes phosphoric acid, using octyl phenyl acid phosphate (OPAP) in the first cycle and di-2-ethyl hexyl phosphoric acid (D2EHPA) with a synergistic additive in the second cycle. Process development systematically overcame problems encountered by others earlier, to give low first and operating costs so that the process is also suitable for the smaller plant. The ESI process is also suitable for uranium extraction from strong acid, produced by hemihydrate type of phosphoric acid processes, with many inherent advantages.

1. INTRODUCTION

Most of the world's phosphate rock deposits generally contain uranium in varying amounts. When the rock is reacted with sulphuric acid for the manufacture of phosphoric acid, this uranium is leached appearing as a minor impurity in the crude acid. During the last decade, the price of uranium for fueling atomic power reactors has risen rapidly, and its extraction from phosphate processing has become economically attractive. Uranium is therefore a valuable and, in the wake of the current energy crisis, possibly the most attractive by-product commercially which the phosphate industry has the potential to produce. At present the extraction of uranium from wet process phosphoric acid is either being carried out or about to commence in 9 plants in the United States and Canada. Altogether these plants have the capacity to supply about 2 million kilograms per year of  $U_3O_8$ , or approximately 14% of the current U.S requirements (1). There is one plant in operation in Europe and several others in various stages of planning or construction. The uranium is recovered as a by-product from existing operations, therefore the environmental difficulties are significantly less troublesome than those associated with new mining and milling facilities. It is highly likely that more of these extraction facilities will be therefore constructed all round the world, and it has been estimated (1) that this resource may supply about 10% of the world's uranium requirements.

In a recent extensive study (1) conducted by Earth Sciences Incorporated for the United States Department of Energy it is reported that the phosphate resources of the free world amount to about 1,300 billion tons, about a quarter of this being recoverable based on the present day technological and commercial yardsticks. The main purpose in developing these phosphate deposits around the world would be to make the phosphate available for fertiliser products, and the extraction of uranium from these deposits will be a secondary attraction. The ESI study estimated that the uranium present in the recoverable phosphate resources amounts to some 30 million tons.

During the early 1950's a considerable attention was given to the extraction of uranium directly from the phosphate rock by selective leaching methods (2). Alkaline leaching methods were ineffective whilst acid leaching although effective was not very selective and consequently was rather costly. At about the same time development work was also addressed to the extraction of uranium from phosphoric acid. Three commercial scale plants were built and operated successfully for several years. Two of these used solvent extraction and the third used a chemical precipitation method (3). These plants were located in the U.S.A. and were eventually unable to compete with the low cost uranium which subsequently became available from the mines in the western United States. Fortunately a very large proportion of the phosphate mined around the world is currently being converted into wet process phosphoric acid, and the trend is for this proportion to increase. Because of the more favourable production and market economics there is a resurgence of interest in the extraction of uranium from wet process phosphoric acid.

## 2. SOLVENT EXTRACTION TECHNOLOGY

Currently the basic extraction technology relies on solvent extraction and three main solvents have been identified, namely octyl pyrophosphoric acid (OPPA), di-2-ethyl hexyl phosphoric acid (D2EHPA) with a synergistic additive tri-octyl phosphoric oxide (TOPO), and octyl phenyl acid phosphate (OPAP). All processes appear to be capable of operating economically on a typical phosphoric acid product from a dihydrate process plant. All plants currently operating with these solvents extract uranium from 30%  $P_2O_5$  filter acid. For increasing  $P_2O_5$  content, the extraction coefficient from aqueous to organic phase falls hence more extraction steps are required. A process with the capability of extracting uranium from 45-54%  $P_2O_5$  acid will have several outstanding advantages discussed later in this paper. There is an optimum extraction temperature, in all cases generally lower than the supply acid temperature. Reducing the temperature of the acid generally leads to a lower dissolved content of gypsum in the solution, increase in the extraction coefficient, and reduction in the rate of degradation (if any) of the solvent, but it increases the time required for phase separation.

All processes employ an acid preparation system consisting of cooling, purification and uranium valence adjustment stages. All require a raffinate (uranium-barren acid) clean up system to ensure minimum extractant losses and to avoid deleterious effect on the rubber lining of phosphoric acid concentration equipment. All processes give rise to small quantities of liquid effluent which requires treatment and all require a system for fume and dust scrubbing in the final precipitation, drying and calcining, and bagging operations. All processes have the highest capital and operating cost associated with the first solvent extraction and stripping stages. Since the overall concentration of uranium over this operation is 30 to 50, the size of processing equipment and the consumption of reagents and utilities is much larger in this section than subsequent processing.

Earlier research and development work was carried out by the Oak Ridge National Laboratories (4) (5) (6). ESI evaluated the process requirements and economics of the three solvent systems, and determined to use the OPAP system for its plant in Calgary. ESI carried out extensive development work to solve some major problems identified by the earlier work. These problems were:

- i) Third phase formation (crud) in the solvent extraction unit steps.
- ii) Contamination of the  $U_3O_8$  - barren phosphoric acid returned to the acid plant.
- iii) Poisoning of the extractant.
- iv) Excessive phosphate, vandate, etc. contamination of the uranium product.

A pilot plant was constructed and operated. The plant demonstrated that the technology resulted in excellent uranium recovery and eliminated the above noted operating problems. The preferential loss of the mono ester reported by others (7) was not observed. The reason for hydrolisis of the OPAP reported in literature (3) was identified and a satisfactory solution developed. The data developed in the pilot plant was used to design the commercial scale plant which has come on stream recently.

### 3. EARTH SCIENCES URANIUM RECOVERY PROCESS

The solvent extraction technology developed by ESI is being applied in its own plant in Calgary, Alberta, Canada. The plant is located adjacent to the Western Co-operative Fertiliser Limited's phosphate complex and extracts uranium from wet process phosphoric acid manufactured by the Western Co-operative Fertilisers. It will recover in excess of 45,000 kg of  $U_3O_8$  per year from 29-30%  $P_2O_5$  phosphoric acid containing about 120 mg/l of  $U_3O_8$ . The acid treated in that plant is 'green' made from calcined western U.S. rock. The yellow cake product will meet purity standards of any conversion facility.

The ESI recovery process may, for clarity, be broken into five general process areas (Figure 1).

- i) Acid clean-up and preparation
- ii) Uranium extraction
- iii) Uranium purification
- iv) Product recovery
- v) Raffinate clean-up

#### Acid Clean-up and Preparation

The purposes of this area of the process are: to adjust chemically the oxidation potential of the acid to ensure that all the uranium is in the recoverable plus 4 valence; to clean the acid of material that will cause unwanted 'crud' formation during the subsequent uranium recovery by solvent extraction; and to lower the acid temperature to about 50°C and, thus, minimise operational problems during the solvent extraction process. A schematic flowsheet is given in Figure 2.

The acid is pumped from the surge tank through a water-cooled spiral heat exchanger, to lower its temperature to about 50°C, to a 17 foot diameter thickener which also receives a flocculant stock solution.

Underflow from the thickener at 15% solids is pumped to a drum filter. Filtrate is pumped to the thickener overflow tank and the gypsum filter cake is thoroughly washed with raffinate ( $U_3O_8$  - barren 30%  $P_2O_5$  acid) to recover any absorbed uranium. Washed filter cake (flocculated gypsum) is repulped with raffinate and pumped to the raffinate surge tank in the raffinate processing area. From there it goes back to the phosphoric acid plant with the uranium-barren acid. Thus, all gypsum entering the plant with the feed acid is sent back to the fertiliser complex. The thickener overflow is sent to the reduction circuit where iron powder is used to adjust chemically the EMF of the acid to about plus 200 mv and, thus, ensure that the uranium is all plus 4 valent. The uranium must be all plus 4 valent to assure maximum extraction by OPAP in the uranium-extraction circuit. About 0.5 grams of iron per litre of acid are required.

The acid contains about 0.2 g/l of suspended solids at this point and is sent to sand-type filtration units for final clarification. The filtrate is used to backwash and clean the filters and this stream is returned to the thickener.

After the phosphoric acid has passed through the acid clean-up and preparation circuit, it has:

1. an additional 0.5 gpl of iron,
2. had all crystalline and supersaturated gypsum removed,
3. been diluted with water by about 1%, and
4. been cooled to under 50°C

### Uranium Extraction

In this area of the plant uranium is recovered by solvent extraction with OPAP from the phosphoric acid, cleaned and prepared as described above. The uranium so recovered is stripped from the loaded organic with a 30%  $P_2O_5$  phosphoric acid solution containing hydrogen peroxide. The net result is recovery of greater than 90% of the uranium into a phosphoric acid solution containing about 10 gpl  $U_3O_8$ . Please see Figure 3.

Reduced phosphoric acid is pumped to the solvent extraction (SX) circuit. The circuit has five stages of extraction. A 0.3 molar octyl phenyl phosphoric acid (OPAP) dissolved in kerosene is used to extract the uranium from the acid. It is possible to produce a raffinate with as little as 5 mg/l  $U_3O_8$ .

The raffinate from the SX circuit is pumped to the raffinate clean-up area, discussed later. Loaded organic is pumped from the first stage extraction unit to an aqueous displacement tank. The recovered aqueous is pumped to the first stage of the extraction circuit.

The loaded OPAP solvent is pumped from the aqueous displacement tank to the solvent stripping circuit which consists of four stages of stripping. The uranium is stripped from the solvent using uranium-barren 30%  $P_2O_5$  acid and 50% hydrogen peroxide.

The stripped solvent, is pumped to a storage tank where entrained aqueous solution separates and is sent back to the solvent stripping circuit. Organic is recycle to the first stripping stage while the pregnant acid, containing about 10 gpl  $U_3O_8$ , is pumped to the uranium purification area.

By the time the phosphoric acid has passed through the uranium extraction circuit it:

1. has had most of the uranium removed leaving only about 5 mg/l  $U_3O_8$ , and
2. contains up to 500 parts per million entrained and dissolved solvent from the SX circuit.

### Uranium Purification

This area of the plant employs a solvent extraction process to produce a concentrated and purified uranyl carbonate solution from the pregnant phosphoric acid strip solution generated by the strip circuit in the uranium extraction area. Please see Figure 4.

Pregnant phosphoric acid strip solution containing about 10 gpl  $U_3O_8$  is diluted with water from 30%  $P_2O_5$  to about 28%  $P_2O_5$  prior to entering the solvent extraction circuit. The dilution is required to ensure efficient loading of the solvent which is a mixture of di-2-ethyl hexyl phosphoric acid (D2EHPA) and a synergistic additive dissolved in kerosene.

The diluted pregnant acid is pumped to the extraction circuit which consists of four stages of extraction. Raffinate from the extraction circuit still contains about 200 mg/l  $U_3O_8$  in the 6 valent state and is, thus, recycled to the reduction circuit in the acid clean-up and preparation area discussed above. Recycle of this stream allows the uranium to be recovered in the uranium extraction area. Prior to its recycle to the reduction circuit, the raffinate is sent to a bank of flotation cells, which remove the organics, and, thus, prevent contamination of the OPAP solvent with the D2EHPA solvent.

The D2EHPA solvent from the extraction circuit is loaded not only with  $U_3O_8$  but also many impurities, such as phosphate ion, and is sent to a scrub circuit for impurity removal. The solvent scrub circuit is comprised of three stages.

Scrubbing is performed with a mineral acid. Spent scrub solution (raffinate) from the scrub circuit containing the impurities is sent directly to the stripping circuit.

The scrubbed D2EHPA solvent is stripped of its uranium content in three stages of stripping using a 100 gpl sodium carbonate solution. The strip circuit yields a pregnant carbonate solution of approximately 50 gpl  $U_3O_8$ . The pregnant strip solution is stored in a surge tank prior to sending it to the acidification circuit in the product recovery area while stripped solvent is sent to the organic storage tank. This tank also serves as an aqueous displacement tank.

The stripped solvent, produced as described above, is in the sodium salt form and must be converted to the hydrogen salt prior to recycle to the solvent extraction circuit to prevent precipitation of sodium silicates. This solvent conversion is performed in two stages of regeneration using the scrub solution raffinate and a mineral acid. Any uranium in the scrub solution raffinate is recovered in the regeneration circuit. The organic is recycled to the extraction circuit and the spent scrub solution is sent to a coalescer for organic removal prior to collection in the tailings tank. The tailings tank solution, which is composed of spent scrub solution, contains about 50 gpl sodium sulphate, 15 gpl sulphuric acid, a small amount of ammonium sulphate, and impurities of iron, fluoride, etc. The stream ranges from 5 to 20 litres per minute depending on the size of the plant.

The uranium purification area produces three streams and the characteristics of each stream can be summarised as follows:

1. The extraction raffinate is an oxidised 30%  $P_2O_5$  acid solution which has been diluted with water to 28%  $P_2O_5$ . This stream returns 99.8% of the phosphate values entering the uranium purification area to the acid clean-up area as well as 3% of the uranium.
2. The pregnant carbonate strip solution contains 97% of the uranium entering the uranium purification area. The uranium contained in this 50 gpl solution is not significantly contaminated with impurities.
3. The spent regenerent solution is the impurity bleed stream for this area. It is essentially free of uranium and is sent to waste.

#### Product Recovery

In this area of the plant uranium is recovered from solution as yellowcake which is then dried and packaged in 55 gallon drums. The process is a conventional uranium recovery design and is shown in Figure 5.

Pregnant strip solution produced by the solvent strip circuit is pumped continuously to an acidification tank where sulphuric acid is added to lower the solution pH to between 6 and 7. At this pH iron and other impurities are precipitated from solution and the precipitate is removed by pressure filtration. The filter cake contains a significant amount of  $U_3O_8$  and is, therefore, sent to the dilution tank in the purification area for dissolution and ultimately, uranium recovery. Following filtration, the pregnant strip solution is further acidified with sulphuric acid to a pH of about 2 and heated to near boiling to completely expel the  $CO_2$  before the uranium is precipitated.

Uranium is recovered from the acidified solution as yellowcake by the addition of ammonia gas to both tanks of the precipitation circuit until a pH of 7 is reached. The yellowcake slurry so formed is flocculated and then thickened. Thickener underflow is pumped to the agitated centrifuge feed tank at about 25% solids while the thickener overflow is collected in a tank (along with two other streams) prior to recycle to the solvent scrub circuit in the uranium purification area.

The yellowcake centrifuging, drying, and product packaging areas run on a one shift per day, five day week schedule. The 25% solids yellowcake slurry is diluted to about 10% solids with water, centrifuged and simultaneously washed with more water. The 50% solids yellowcake slurry from the centrifuge is sent by screw conveyor to the dryer and the centrate is sent to the yellowcake thickener



overflow tank. The yellowcake is dried at 260°C. The cooled product discharged to a packaging unit fitted with a bagfilter. The product is then discharged to 250 litre drums which each hold 340 kg of yellowcake.

A few general comments can be made about the product recovery area as follows:

1. The two-stage acidification circuit removes impurities (notably iron and phosphate) from the uranium solution.
2. Recycle of the yellowcake thickener overflow, centrate, and venturi scrubber solutions to the scrub circuit in the uranium purification area bleeds impurities from the product recovery area. More importantly, recycle of these streams ensures recovery of the dissolved and entrained uranium.

#### Raffinate Clean-up

The raffinate phosphoric acid from the solvent extraction circuit in the uranium extraction area contains some entrained and dissolved organics. The raffinate clean-up area of the plant removes most of these organics from the raffinate (uranium barren 30% P<sub>2</sub>O<sub>5</sub> acid) prior to its return to the phosphoric acid plant, thus, recovering the solvent to prevent any organic-induced deterioration of rubber-lined equipment in the acid plant. Three steps are used by ESI in this area. The first two are a combination of coalescing and flotation. The third is a proprietary system. These are illustrated in Figure 6.

Raffinate from the uranium extraction area may contain as much as 500 ppm of entrained and dissolved process organics and is pumped to a multi-stage coalescer. The coalesced organics collect at the top and are periodically returned to the last uranium extraction stage. The coalescer efficiently removes entrained organic from the raffinate.

The remaining organics in the raffinate are typically in the order of 100 ppm and a large part of these organics can be recovered by froth flotation using conventional air-sparged flotation cells. The froth overflowing the cells consists of kerosene and OPAP left in the raffinate. The flotation process reduces the organics to between 20 and 50 ppm. The third step which ESI uses at Calgary can reduce organic levels to about 1 ppm. This may not always be necessary, but can be achieved at a reasonable cost. Once the raffinate is cleaned it is heated to 70°C and returned to the phosphoric acid plant.

Following the raffinate clean-up the raffinate:

1. Contains about 1 ppm of process introduced organics.
2. Is at the same temperature as it was received and is ready for evaporation.

#### 4. ADVANTAGES OF ESI'S TECHNOLOGY

ESI began its uranium recovery work on a relatively small phosphoric acid plant, 165,000 STPY  $P_2O_5$ . This size was considered by many in the industry to be too small to economically support uranium recovery. Partially due to this and to other process factors, ESI was able to develop extremely cost efficient methods to process the phosphoric acid and the resulting uranium. Some of these methods arise from the general nature of the OPAP process; others are related to pretreating the phosphoric acid, purifying the uranium stream, and removing process introduced organics from the raffinate efficiently.

In addition to the development efforts on the green acid of WCFL, ESI has conducted research into handling the organic-rich acids (black acids) and acids containing in excess of 30%  $P_2O_5$ . This work has yielded further potential advantages of ESI's technology for uranium recovery.

The use of OPAP offers potential operating cost savings in a variety of areas. The first relates to the initial solvent extraction stage. In most phosphoric acids, the bulk of the uranium is present in the reduced state or plus 4 valence. Reduction of the acid for OPAP extraction may be done at a reasonable cost with a variety of reductants. ESI's experience has suggested that it normally is less expensive to reduce the uranium for extraction by OPAP than to oxidise it for extraction by D2EHPA-TOPO.

The uranium can be stripped into a 30% phosphoric acid solution under oxidising conditions rather than into the concentrated acid specified in the literature, saving expense and process difficulties. Taking this strip solution directly to a D2EHPA extraction circuit for uranium purification, without any additional valance adjustment, allows further cost savings. From this point in the process conventional uranium recovery technologies may be used to obtain a final product. The specific arrangement of the purification circuit allows the production of an extremely high quality product, capable of meeting the specifications of any converter in the free world.

One of the chief concerns of phosphoric acid manufacturers has been the adverse effects of residual kerosene on rubber-lined evaporators. While the industry in the U.S. has seemed to set 50 ppm as an 'acceptable' level for kerosene, we are unable to verify that such a level is adequately low to afford protection to manufacturers. Knowledgeable suppliers of rubber lining have recommended the complete removal of kerosene. The lining is liable to absorb even traces of it and suffer degradation. Early in the development process, ESI was asked to delivery a raffinate with essentially no kerosene and a system was developed to produce such a clean raffinate. ESI's proprietary system can remove kerosene and OPAP levels to about 1 ppm. The analytical techniques necessary to generate reproducible results

were developed as well. There is a cost associated with the removal of the last 20 to 50 ppm, but it is a reasonable cost to pay as insurance against damage to the rubber lining in evaporation equipment. The cost generally will run to about 5 to 7% of the total plant cost and about \$0.50 to \$1.00 per lb of  $U_3O_8$ .

As black acids are common worldwide, ESI undertook the task of developing a system to remove the organics to allow solvent extraction. This proprietary process produces a 'beer' coloured acid which is easily handled in solvent extraction systems without serious emulsion formation. The capital cost of the system generally is about 10 to 15% of the total cost of the uranium recovery plant. The operating costs can range from \$0.50 to \$2.00 per pound of  $U_3O_8$ . These costs appear to be quite competitive with several other methods for cleaning black acid.

Rising energy costs have brought about a renewal of interest in the double crystallisation hemihydrate-dihydrate and the single stage hemihydrate high strength phosphoric acid processes. At the same time the ability to extract uranium from acid with higher than 30%  $P_2O_5$  concentration by the use of existing extraction technology has been seriously questioned. This led ESI to carry out in 1977 basic research in extraction from acid ranging from 38% to 54%  $P_2O_5$  content. The results of this work indicated that ESI's OPAP process is applicable to strong acid with only a relatively small reduction in stage extraction efficiencies, whilst several potential advantages are self-evident and significant.

First and foremost, of course, is the scope to reduce energy consumption in the production of concentrated fertiliser grade phosphoric acid without sacrificing the ability to extract the valuable uranium by-product. The very much reduced volume of the stronger acid (approximately halved for 50%  $P_2O_5$  acid in comparison to 30%  $P_2O_5$  acid) would enable a significant reduction in equipment size in the front end of the plant. The higher uranium concentration in the acid would further contribute to this saving, and the capital and operating costs of acid clean-up and first extraction sections, two of the three major sections of the plant, are consequently reduced. Unlike the 30%  $P_2O_5$  acid, which is invariably concentrated after uranium extraction, the strong acid produced from hemihydrate and double crystallisation processes is often produced at a concentration required for fertiliser production. The stringent requirement of raffinate clean-up to remove practically any trace of kerosene is therefore not applicable to strong acid, and the capital and operating costs of yet another major section of the plant are reduced significantly.

In spite of the lower extraction coefficient in the first cycle, the several advantages inherent in extraction of uranium from strong acid appear to be so overwhelmingly favourable that uranium extraction cost from 50%  $P_2O_5$  acid is estimated to be between 20 to 25% lower than the cost of extraction from 30%  $P_2O_5$  acid. The findings of this basic research would however need to be confirmed through further pilot plant work directed specifically at the first cycle extraction and phase separation.

Overall ESI has developed a variety of process characteristics which allow its technology to operate at costs lower than those resulting with many alternative systems. The process is therefore applicable equally to the smaller plant adding considerable to its profitability.

## 5. OPERATING REQUIREMENTS

The ESI plant at Calgary is currently in the commissioning phase. Although specification grade yellow cake has been produced, the extraction plant has not had sufficient acid feed for an adequate period of time for it to have attained steady operation. It has not therefore been possible to verify plant performance. So far there does not, however, appear to be any reason to suppose that the performance is, in any respect, significantly different from that expected.

The following table of reagent and utility consumptions is derived from published information (1) and applies to a process based on two cycle OPAP/D2EHPA extraction and may be assumed to represent average requirements for a plant treating green acid containing 140 mg/litre  $U_3O_8$ . The plant capacity is about 150,000 tons per year of  $P_2O_5$ , and the consumption is per kilogramme of  $U_3O_8$  produced.

Iron powder	4.0 kg
Flocculant	0.25 kg
OPAP	0.1 kg
Kerosene	0.4 kg
$H_2O_2$ 100%	1.5 kg
D2EHPA	5.0 g
Synergistic Additive	1.5 g
$H_2SO_4$	7.2 kg
Sodium Carbonate	3.0 kg

The consumption of process water is anticipated to be about 240 litres per kg of  $U_3O_8$  product and electricity usage about 30 KWh per kg of  $U_3O_8$ . Iron, Hydrogen peroxide and electric power are the cost significant items.

The cost of maintenance labour and materials is expected not to exceed 7% of the direct capital cost of the plant.

The labour force recommended for normal day to day operation of the plant is:

Plant Manager	1
Supervisor	1
Plant Engineer	1
Chemist	1
Operators	18
	<hr/>
Total	22

#### 6. CAPITAL COST

We estimate that for a Western European locality the erected cost of a battery limits plant for extracting  $U_3O_8$  from 150,000 tonnes  $P_2O_5$  per year green phosphoric acid containing around 140 mg per litre  $U_3O_8$  will be about 6.0 million Pounds Sterling, based on mid 1980 costs. This does not include for land cost but has a 10% contingency added to the estimated direct cost.

REFERENCES

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- (5) Hurst F.J., D.J. Crouse 'Recovery of Uranium from Wet Process Phosphoric Acid by Extraction with Octyl Phenyl Phosphoric Acid' ibid July 1974 No.12 p.286.
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- (7) Arnold W.D. 'Recent Studies of Uranium Recovery from Wet Process Phosphoric Acid with Octyl Phenyl Acid Phosphate' ACS Annual Meeting, Miami Beach, Florida, Sept. 13 1978.

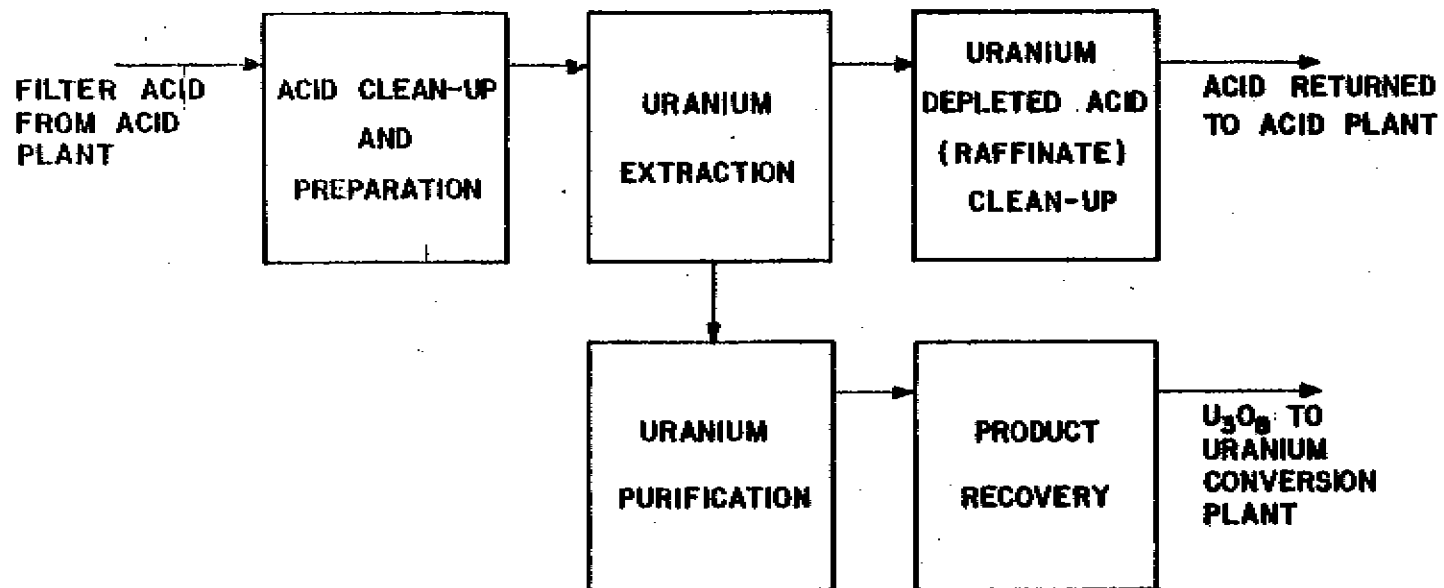


Figure 1 : Generalized Flowsheet for Uranium Recovery from Phosphoric Acid.

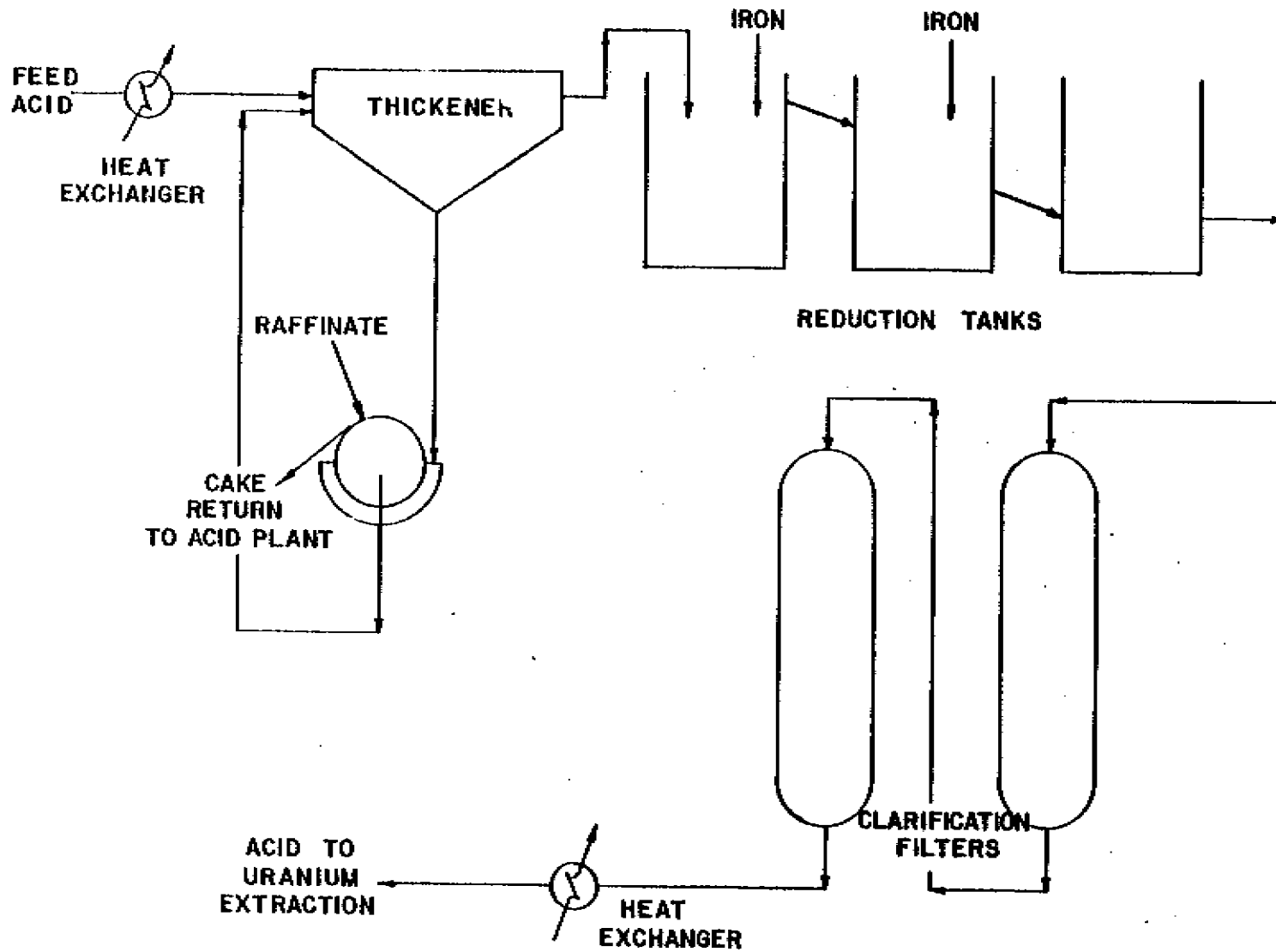


Figure 2.: Acid Clean-up and Preparation.



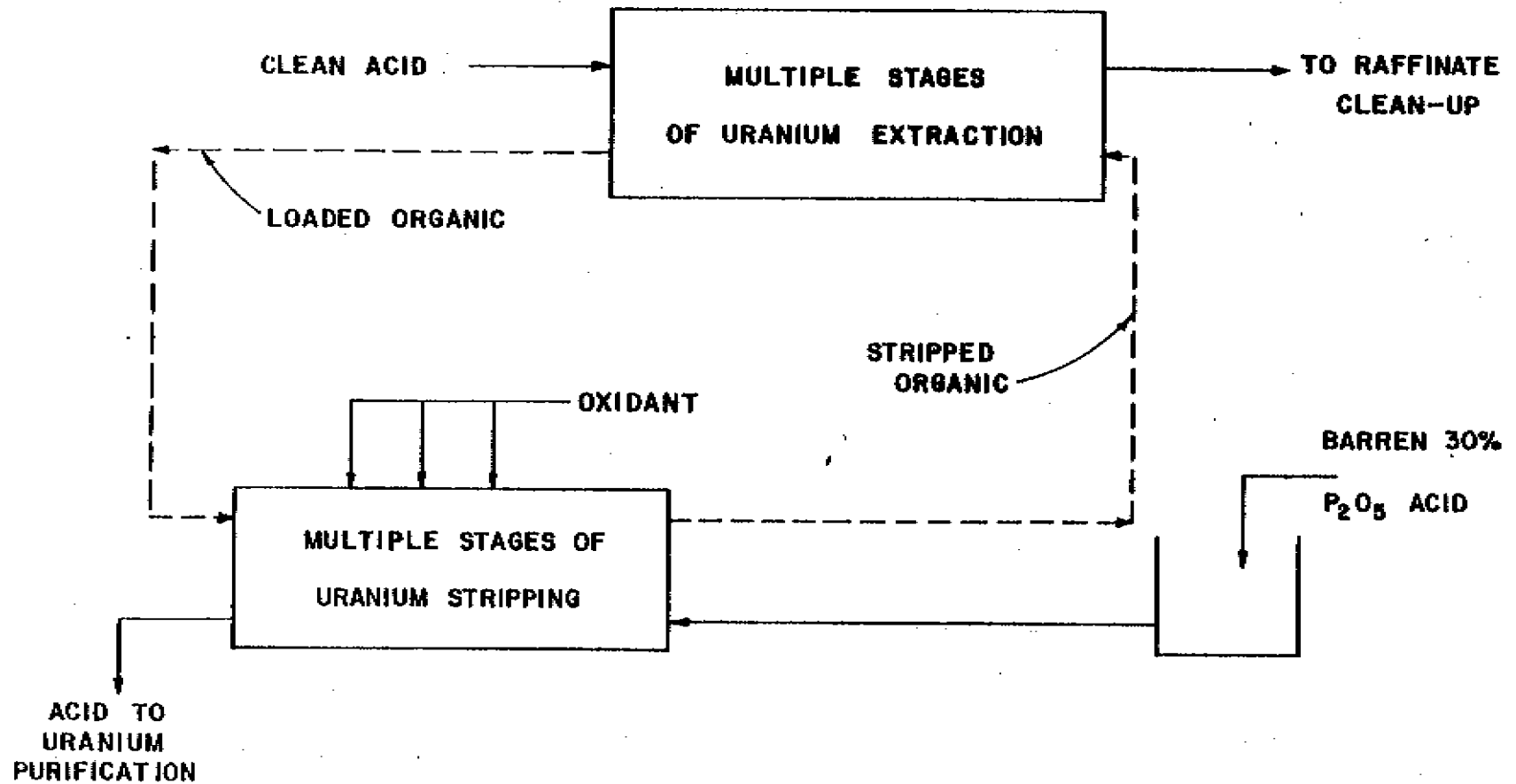


Figure 3 : Uranium Extraction.

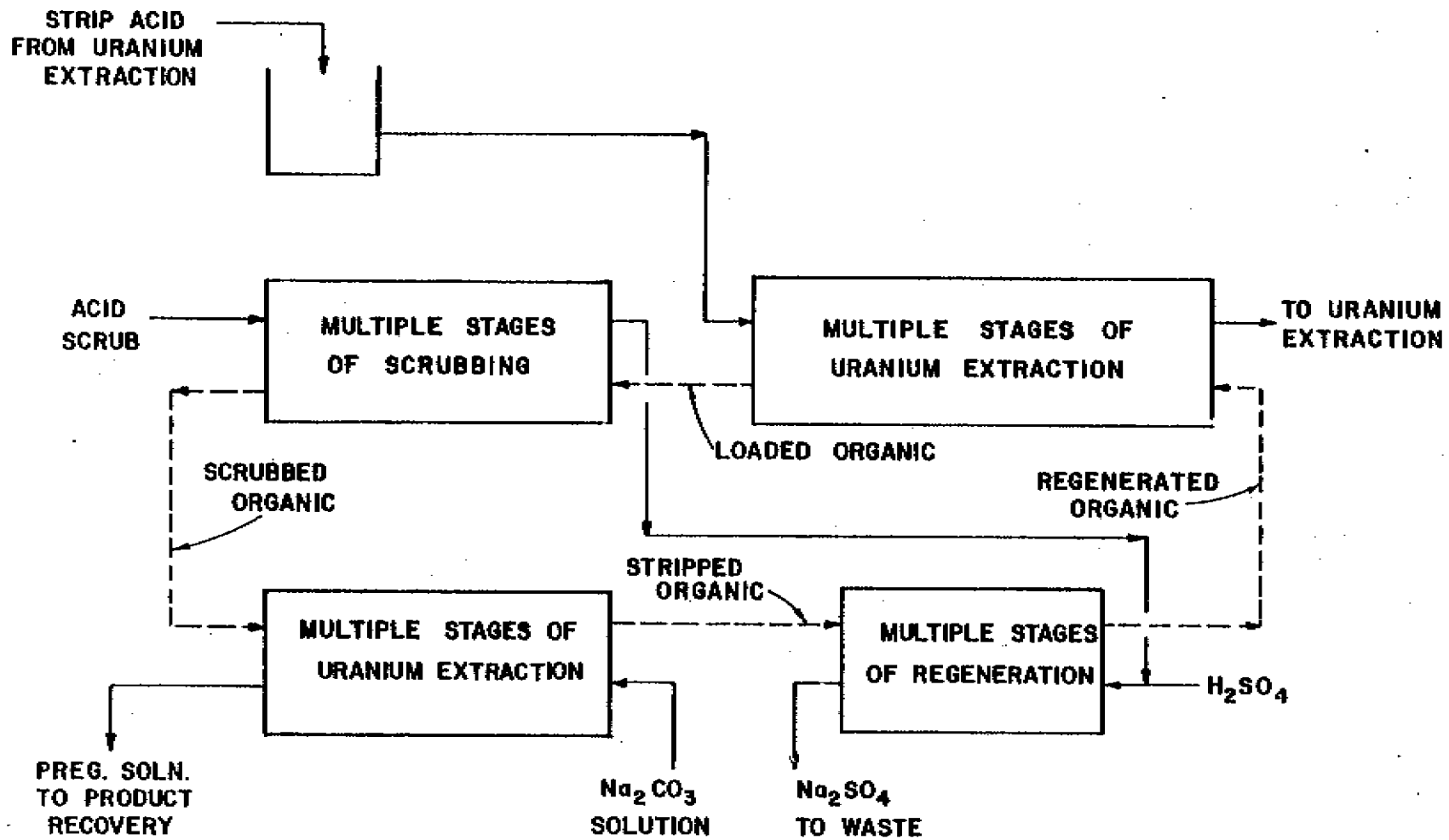


Figure 4 : Uranium Purification.

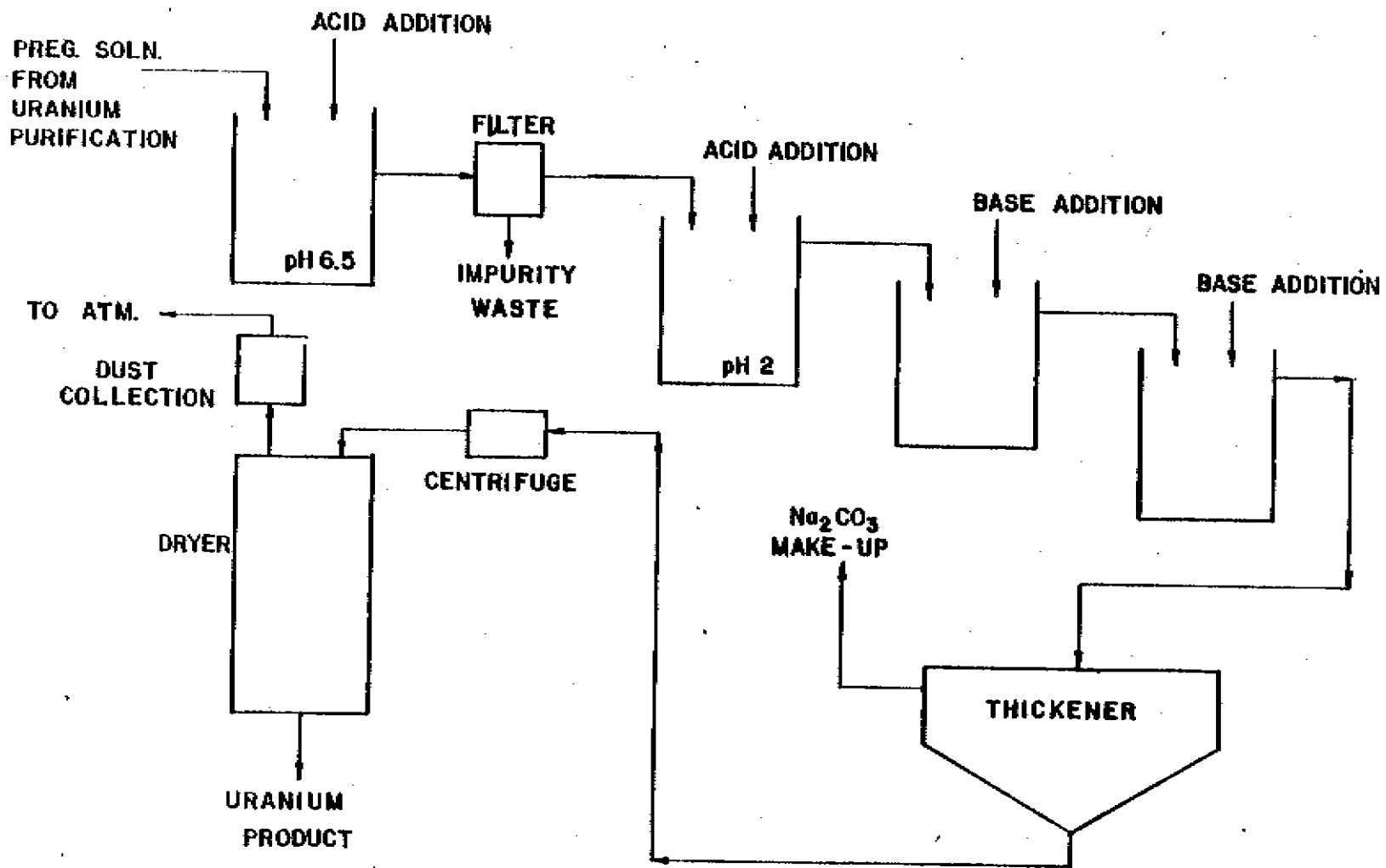


Figure 5 : Product Recovery.

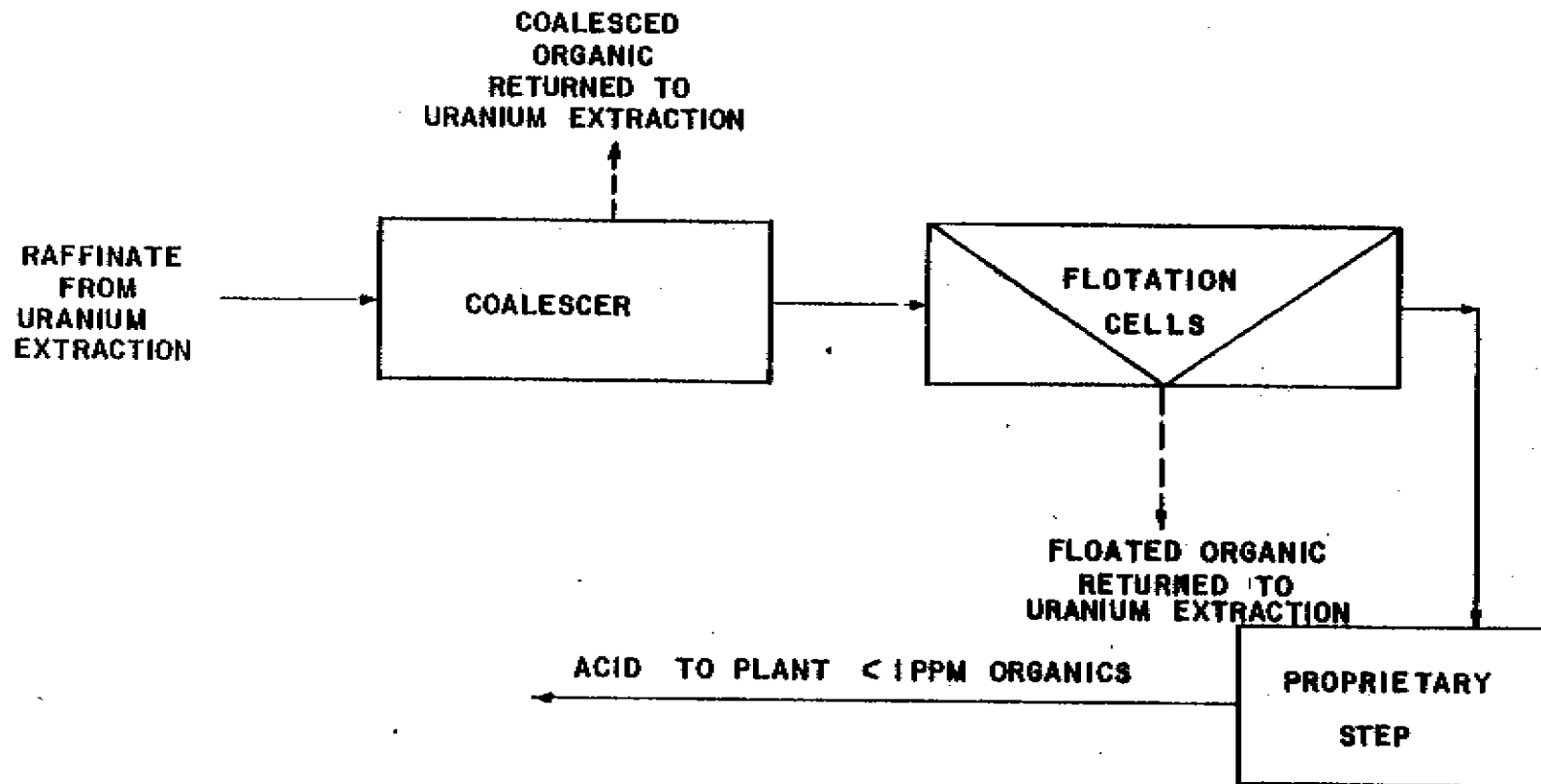


Figure 6 : Raffinate Clean-up.

TA/80/8 Uranium from phosphoric acid - The Earth Sciences process by  
I.S. Mangat, Humphreys & Glasgow, United Kingdom, J.H. Viellenave,  
Earth Sciences Inc., United States

DISCUSSION : (Rapporteur D.W. Leyshon, Jacobs Engineering, United States)

Q - Mr. R. van HARDEVELD, D.S.M., Netherlands

- 1) On page 10 of your paper you state that ESI's OPAP-process is applicable to strong acid with only a relatively small reduction in extraction efficiency.

Can you indicate what is the OPAP loss as a function of the acid concentration? Are any data available on OPAP hydrolysis as a function of acid strength and acid temperature? Can you give the change of distribution coefficient?

- 2) Did I understand from your paper correctly that you do not use any flocculant or active carbon treatment in acid clean up? If so, are you not faced with coalescence problems in the settlers?

A - 1) The figure 1 used as a slide in the presentation in Vienna illustrates the significantly higher uranium extraction coefficient which OPAP has compared to the D2T system. The data is taken from work carried out by ESI for the United States Department of Energy. (See figure following page).

- 2) Flocculants are used in two separations, first on the gypsum slurry filtration and also in the clarification of yellow cake. No carbon treatment is used in the ESI process; the Calgary plant processes green acid from calcined rock. ESI has a proprietary process for cleaning up "black" acid, as mentioned earlier.

Q - Mr. E. JEMAA, ICM, Tunisia

My question concerns both authors who presented their processes for extracting uranium from phosphoric acid. Is it possible to have a full chemical analysis of the uranium concentrate extracted from phosphoric acid and what is the efficiency of the two solvents used (DEHPA-TOPO and OPAP) vis à vis the extraction of rare earths (in particular thorium) contained in phosphoric acid?

A - I cannot answer at this time with a complete analysis. The uranium peroxide  $U_3O_8$  content is about 90%.  $SO_4^-$  is a main impurity. Thorium is low, well below industry specifications. Vanadium goes out in liquid effluent which is mainly  $Na_2SO_4$  with free sulfuric acid.

Answer by F. NIELSSON, IMC, United States

A typical analysis for the product is given in paper 7 indicating a 68.2% U which is equivalent to about 86.5%  $U_3O_8$ .

Q - Mr. I. EZAHR, OCP, Morocco

- 1) How often can OPAP be recycled?
- 2) What method of analysis do you use for residual solvent?
- 3) Does the amount of 20-50 ppm kerosene raise any problem in acid concentration?

- A -
- 1) Makeup is done infrequently. The OPAP consumption given in the paper is correct.
  - 2) The method of analysis for residual solvent is an ESI proprietary method. It is reproducible to 1 ppm.
  - 3) The U.S. seems to have accepted 50 ppm as permissible, but some evaporator suppliers are reluctant to concede this figure. ESI has a proprietary system to reduce the organics well below that figure.

Q - Mr. M. SMANI, CERPHOS, Morocco

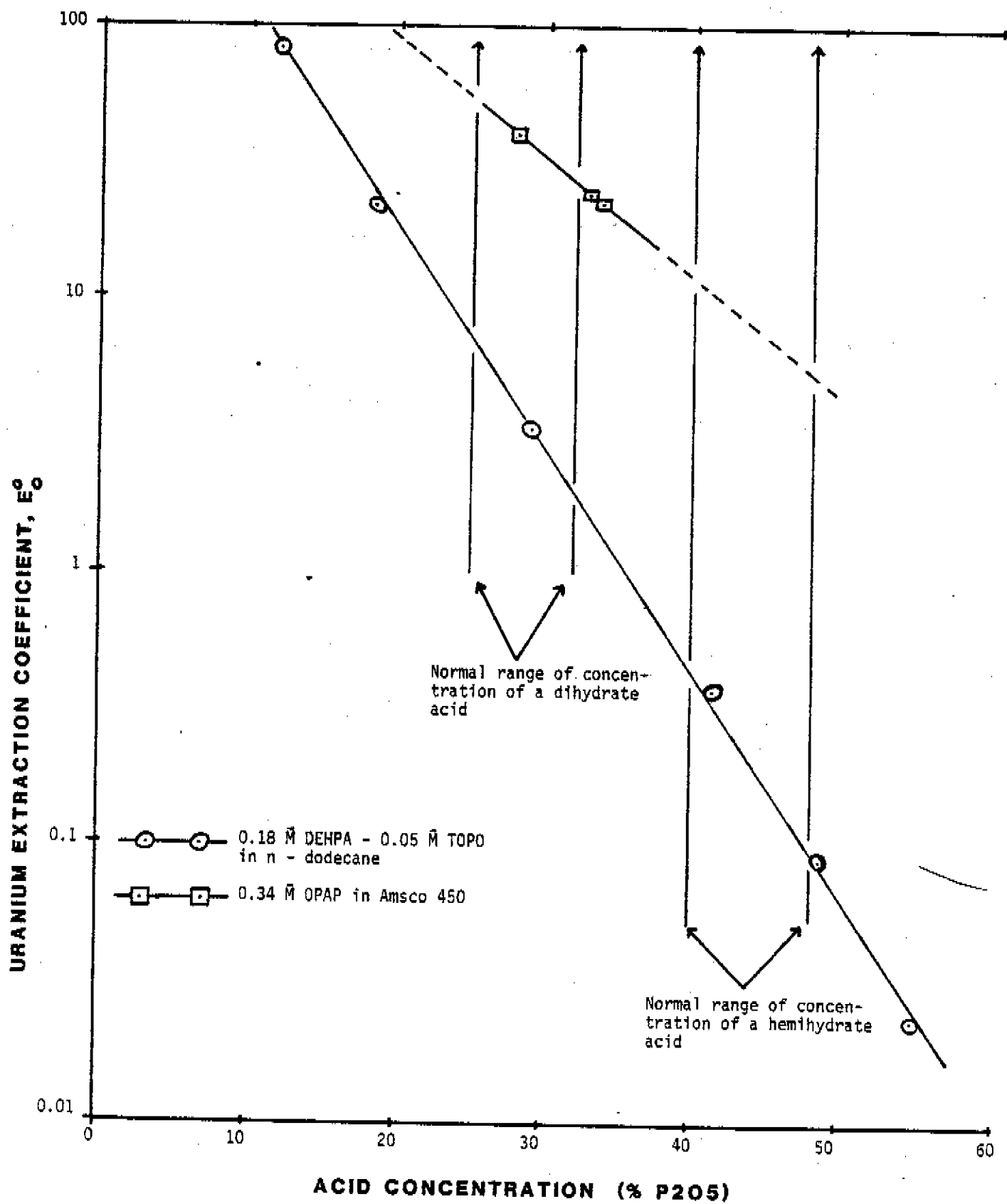
How did you determine that 200 mv was the potential indicating that all the iron is + 2 valence? What is the reference electrode used to give that value?

- A -
- The level of 200 mv has been established in practice as that required for good extraction. I do not have the details of the analytical determination, but it is fairly conventional.

Q - Mr. M. BERGERET, Gardinier, France

What would be the additional cost of an 150,000 t/yr  $P_2O_5$  plant using black acid from Florida instead of green acid?

- A -
- 10% to 15% extra (as stated in paper).



Uranium extraction coefficients as a function of acid concentration for two extractant systems