



SPECIFICATION

Lab-Scale Continuous Flow Pilot-Plant for Recovery of Uranium from Phosphoric Acid

1. Scope

- 1.1. This specification describes the requirements for a lab-scale continuous flow pilot plant for recovery of uranium from phosphoric acid (hereinafter referred to as the “System”).
- 1.2. The End-User of the System is the Nuclear Materials Research Section Philippine Nuclear Research Institute, Quezon City (hereinafter referred to as the “End-User”).

2. Requirements

The System shall meet the following functional and performance requirements:

- 2.1. Have the following power requirements: 220 to 240 volts AC (60Hz) and have the proper electrical connection to support operation in the Philippines; and
- 2.2. The System shall be capable of meeting the design and performance characteristics as described in the attached report “Background, Design Basis and Operational Philosophy for a Continuous Flow Lab-Scale Pilot Process”.

3. Marking

The System shall have all safety markings in the English language.

4. Packing

- 4.1. The System, for the shipment by air to the End-User, shall be packed in accordance with international standards that are applicable for the shipment by air of this kind of equipment.

5. Quality Requirements

- 5.1. The System shall be manufactured, packed and installed in accordance with the Supplier’s ISO quality assurance system or an equivalent quality assurance system.
- 5.2. The Supplier shall document the compliance with this quality assurance system.

6. Testing and Acceptance

- 6.1. The System, prior to shipment, shall be tested for conformance of the System with manufacturer’s performance specifications and the minimum requirements specified herein.
- 6.2. The System, after installation, shall be tested by the Supplier together with the End-User to demonstrate that the performance meets the manufacturer’s



performance specifications and the minimum requirements specified herein as determined by the IAEA and the End-User.

6.3. The results of the testing of the System shall be documented by the Supplier in an acceptance protocol that shall be signed by the End-User.


7. Installation and Training

7.1. The Supplier shall install the System at the End-User's site.

7.2. The Supplier shall provide five days of training for up to three staff of the End-User in the safe start-up, operation, troubleshooting and shutdown of the System at the End-User's location immediately after the installation of the System.

8. Deliverable Data Items

8.1. The Supplier shall provide two (2) complete sets of operation and servicing manuals and technical drawings (where applicable) in the English language.



BACKGROUND, DESIGN BASIS AND OPERATIONAL PHILOSOPHY FOR A CONTINUOUS FLOW LAB- SCALE PILOT PROCESS



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I. INTRODUCTION

Phosphate rock is the main source of phosphorus used in the production of phosphate fertilizers and is also a potential source of uranium, rare earth elements, as well as thorium and valuable elements such as scandium, vanadium, etc. About 1.97 million tonnes of phosphate ore is processed annually by Philippine Phosphate Fertilizer Corporation (PHILPHOS), the largest and leading phosphate fertilizer plant in the Philippines for the production of phosphate-based fertilizers. During the “wet process” production, phosphate-bearing ore is leached by sulfuric acid to produce phosphoric acid and phosphogypsum by-product. During this process majority of the elements such as uranium, vanadium and other industrial elements in the phosphate rock reports to the phosphoric acid. The rare earth elements primarily report to the by-product phosphogypsum. The phosphoric acid is then further processed into fertilizers. It was observed that uranium in phosphate rocks (71-141 ppm) and phosphoric acid (108 ppm) were transferred and enhanced into the phosphatic fertilizer in which uranium content reached up to 228 ppm (found in NPK fertilizer 18-46-0). This is significantly higher than the global median uranium content in soils and rocks, which is 1.3 ppm (UNSCEAR, 2008). Continuous application of contaminated fertilizers therefore may pose negative environmental impacts, as well as, result to loss of a valuable energy resource. This project is a continuation of the static laboratory testing for developing a recovery system of uranium, rare earths and other valuable elements that may be derived from phosphate processing in the Philippines.

II. SYSTEM DESCRIPTION

The lab-scale continuous flow solvent extraction and precipitation process (herein referred to as the ‘System’) is a demonstration setup for the conduct of a continuous counter-current liquid-liquid extraction of uranium from wet phosphoric acid using a synergistic mixture of di-ethyl hexyl phosphoric acid (DEHPA) – trioctyl phosphine oxide (TOPO) in kerosene. The System is designed to handle feed phosphoric acid (27-32% P₂O₅ content) at a rate of 5 L/hr. Uranium extraction and enrichment from feed phosphoric acid (aqueous phase) is carried out by solvent extraction using 0.5 M DEHPA-0.125 M TOPO in kerosene (solvent phase).

The System layout will include an acid storage area for the collected phosphoric acid from the manufacturing plant and return acid (uranium barren acid), a phosphoric acid pretreatment area, the main extraction unit, solvent preparation, and the analytical laboratory as support. The equipment in this facility is composed of holding tanks, oxidation and reduction tanks, pumps and mixer-settlers covering the pretreatment of phosphoric acid, and the 1st cycle extraction-stripping (6 extraction stages and 6 stripping stages) of uranium. The main extraction-stripping equipment will be housed in a 2-tier 6m x 3m platform. The mixer-settlers will be located on 2nd tier while its corresponding pumps and day tanks will be located on the first tier. The facility will run continuously (24x7) for each trial operation to collect enough uranium rich strong phosphoric acid (strip acid), which will be kept in a storage tank for subsequent operation of the second cycle extraction-stripping.

For the second cycle extraction-stripping, the same mixer-settlers will be used for 2nd cycle extraction (6 stages), scrubbing (4 stages) and 2nd cycle carbonate stripping (2 stages). Precipitation of uranium yellowcake from the uranium-rich sodium carbonate solution will be done batch wise.



III. GOAL OF THE SYSTEM

The primary goal of the System is to acquire process parameters for conducting scaled up/pilot or commercial plant recovery of uranium. Specific objectives of the program include the following:

1. Determine the kinetics and equilibrium isotherms for extraction and stripping of uranium in a continuous counter-current liquid-liquid extraction.
2. Study the effects of varying operating parameters such as optical density, oxidation reduction potential, temperature, and aqueous to organic phase ratio on the overall recovery of uranium from phosphoric acid.
3. Determine effectiveness of pretreatment steps of the raw phosphoric acid.
4. Determine the optimum process parameters for the recovery of uranium.
5. Identify possible bottlenecks and process conditions that might arise during the operation of a scaled-up setup.
6. Gain confidence on the dynamic and steady-state operation on a continuous basis.

IV. EXPECTED OUTCOME OF THE INTEGRATED FACILITY

1. Equilibrium data of a continuous liquid-liquid extraction process of uranium from phosphoric acid covering start-up, steady phase and to the shutdown.
2. Optimum parameters for scaled-up continuous uranium extraction systems.
3. Performance of industrial scale equipment.
4. Input for development of revised process operation manual.
5. Preliminary techno-economic analysis for feasibility study.
6. Production yellowcake for subsequent analysis of its purity and use in development of purification steps.

V. OPERATIONAL DESCRIPTION

Uranium is recovered from phosphoric acid using the synergistic solvent mixture DEHPA and TOPO in kerosene. The method described by IAEA (1989) and Hurst et al. (1972) was adapted and modified. The raw phosphoric acid from the fertilizer plant undergoes pretreatment, followed by two cycles of extraction and stripping until final precipitation of uranium yellow cake. The general flowsheet of the extraction of uranium using DEHPA-TOPO is shown in Figure 1.

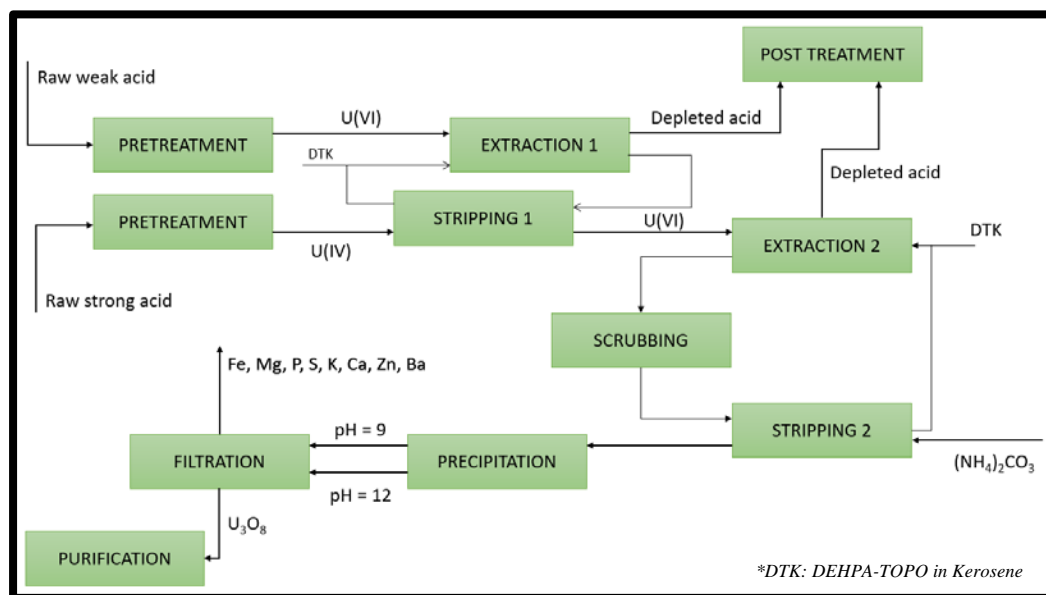


Figure 1. Simplified flowsheet for the recovery of uranium using DEHPA-TOPO Process.

Pretreatment of weak phosphoric acid

Pretreatment of weak phosphoric acid involves cooling to precipitate Na_2SiF_6 and CaSO_4 and improve SX efficiency; flocculation-filtration to remove the precipitates, suspended solids, and dissolved organic matter; and adjustment of acid oxidation-reduction potential to oxidize U prior to U extraction. Phosphoric acid of 27-32% P_2O_5 content coming from the phosphoric acid plant is pumped into flash-coolers to reduce temperature to around 30-40 deg C. The liquid-liquid extraction of uranium using DEHPA-TOPO, according to preliminary laboratory results is determined to be an exothermic reaction (Figure 2). Thus, some cooling is necessary to attain reasonable extraction efficiency.

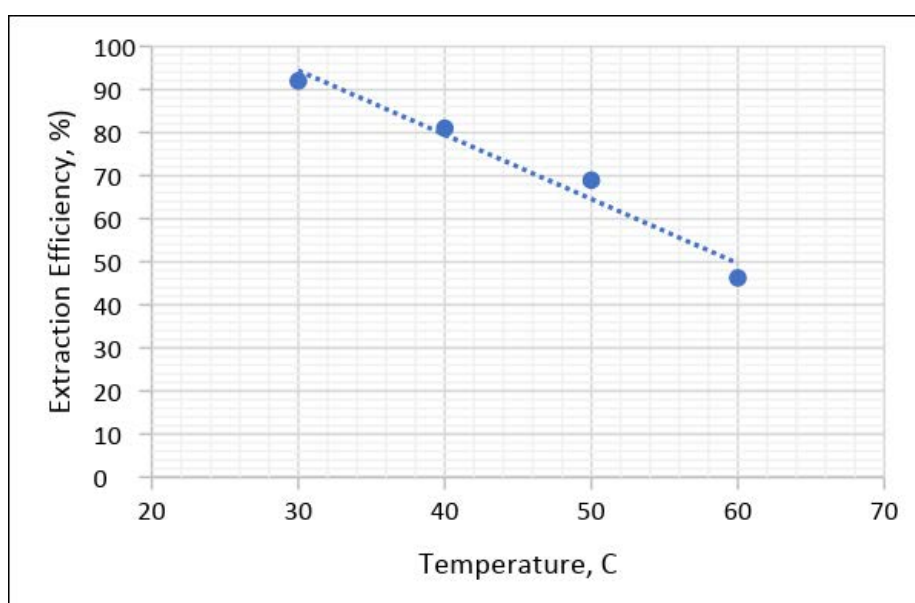


Figure 2. Variation of extraction efficiency with temperature.



After cooling the acid, the feed then goes into the activated carbon column to remove suspended solids and dissolved organic matter. The degree of pretreatment of the acid is monitored by measuring the optical density of the acid at 408 nm wavelength in a UV-Vis spectrophotometer. The exiting optical density should be checked to be in a range of 0.5 – 1.5. It is very important to note that the effect of optical density on extraction efficiency is significant (Figure 3), since the dissolve organic materials contaminate the solvent extractant (DEHPA-TOPO in kerosene) and reduce the extraction efficiency.

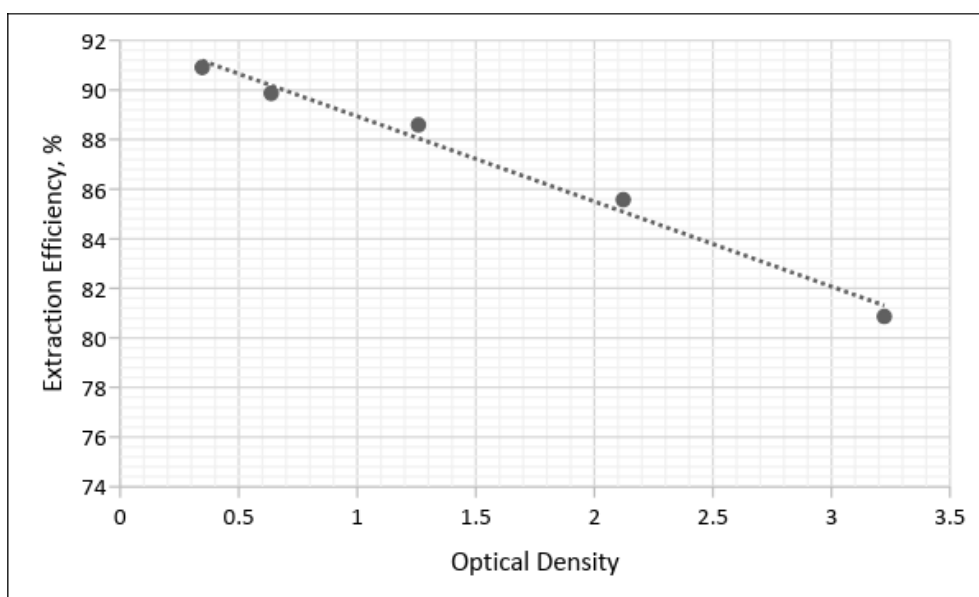
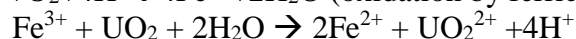
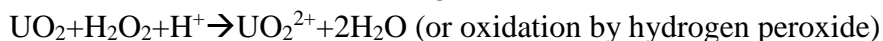


Figure 3. Effect of the variation of optical density of phosphoric acid on the uranium extraction efficiency.

After the removal of dissolved organic matter, the oxidation-reduction potential (ORP) of the acid is then raised to >650 mV by addition of an oxidant (i.e. H₂O₂). The oxidation of uranium is shown below:



Or

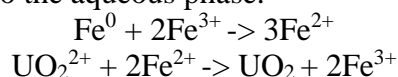


Ferrous iron in phosphoric acid is first oxidized to ferric iron then this ferric iron oxidize uranium IV to uranium VI. In the second case, hydrogen peroxide can also directly oxidize uranium in the phosphoric acid into extractable uranium VI. Oxidation is necessary since the solvent extractant forms a chelate or complex only with uranium in the hexavalent state otherwise extraction efficiency could be too low. The ORP of the acid is an indication of the degree of conversion. At >650 mV most of the uranium in the phosphoric acid is in hexavalent state.



Pretreatment of strong phosphoric acid

The strong phosphoric acid, which is the main feed solution for the first cycle stripping step, is used to back extract uranium in the loaded organic phase coming from the first cycle extraction. It is important to note that the condition of the acid like temperature and ORP should be the opposite of the pretreatment of the weak phosphoric acid to encourage release of uranium ions from the solvent phase back into the aqueous phase.



The ORP of the acid is reduced to a recommended <200 mV and temperature of the acid is raised to around 40-50 deg C. The ORP of the acid is lowered by adding iron to reduce uranium VI to uranium IV.

1st Cycle extraction

The chemical equilibrium for the extraction of uranium in the phosphoric acid using DEHPA-TOPO solvent extractant is as follows:



Uranium is extracted with a mixed solution of 0.5 M DEHPA-0.125 M TOPO in kerosene. During mixing, uranium in the aqueous phosphoric acid forms a complex with DEHPA in the organic phase effectively removing uranium from the phosphoric acid. While TOPO is known to assist the formation of the Uranium-DEHPA complex. Factors that affect this reaction are ORP of the acid, temperature, optical density, aqueous to organic phase ratio and contact time. Pretreatment of phosphoric acid plays an important role as most of these extraction parameters are to be met prior to the contact/mixing of the phosphoric acid and the solvent extractant.

The phosphoric acid plant produces two types of phosphoric acid, namely, the weak phosphoric acid (27-32% P₂O₅ content) and the strong phosphoric acid (>50% P₂O₅ content). Based on the chemical equilibrium for the extraction, less acidic feed source would favor extraction (by Le Chatelier's principle). Recovery of uranium is best conducted on weak phosphoric acid as also depicted in the preliminary batch testing (Figure 4).

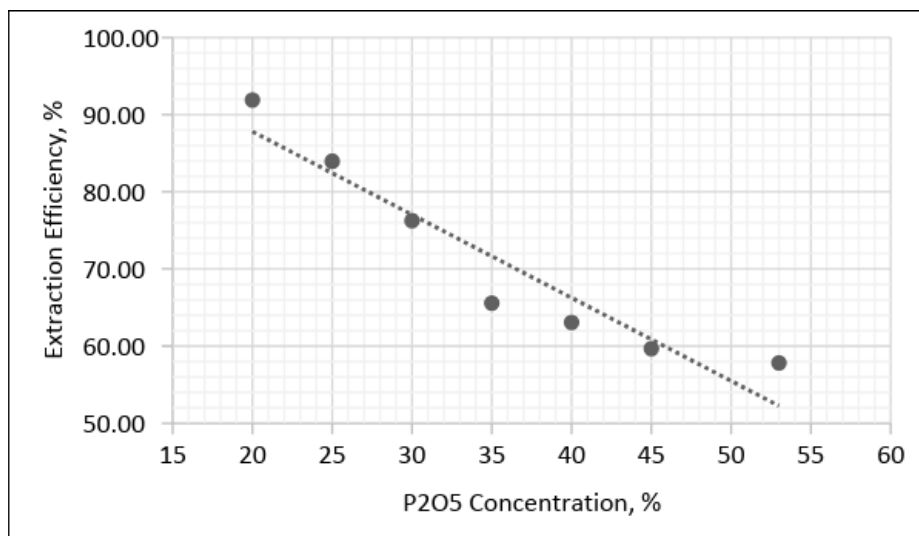


Figure 4. Effect of the variation of phosphoric acid content (P2O5) on the uranium extraction efficiency.

Liquid-liquid extraction of uranium in the scaled-up facility will be conducted in a series of mixer-settlers where the phosphoric acid (as aqueous phase) and organic extractant (as solvent phase), two immiscible liquids continuously flow in a counter-current manner (Figure 5).

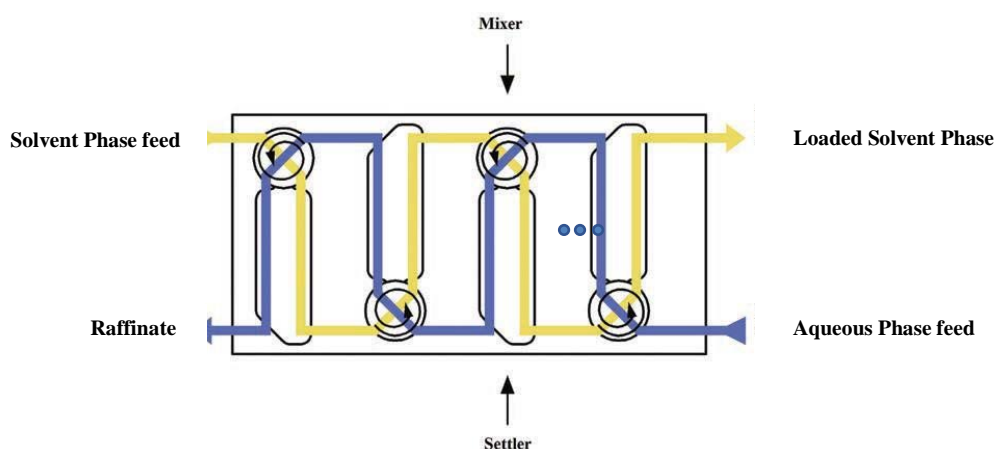


Figure 5. A representation of counter-current liquid-liquid extraction using mixer-settler.

During contact, a portion of uranium from the feed aqueous phosphoric acid (aqueous phase) is transferred into the organic extractant 0.5 M DEHPA-0.125 M TOPO (solvent phase) with each stage (mixer-settler) it passes through. This mass transfer is described by the Kremser equation:

$$N = \frac{\log \left[\left(\frac{x_f - y_s/K_d}{x_n - y_s/K_d} \right) \left(1 - \frac{1}{E} \right) + \frac{1}{E} \right]}{\log E}$$



where N = number of stages of extraction

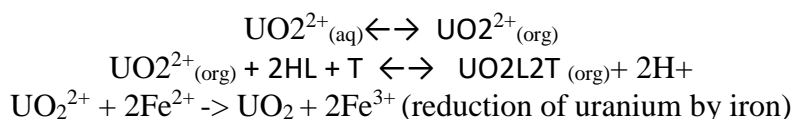
x_f	=	Conc. of uranium in feed aqueous phase
x_n	=	Conc. of uranium in raffinate
y_s	=	Conc. of solute in loaded solvent phase
K_d	=	Distribution coefficient
E	=	$K_d \times O:A \text{ ratio} = (K_d)(\text{flow rate solvent phase}/\text{Flow rate aqueous phase})$

phase)

From the equation, the number of stages (corresponding to the number of mixer-settler) can be calculated by varying the A/O volume ratio, the enrichment of uranium in the light phase and uranium in the raffinate heavy phase. Conversely, values of each can be calculated by fixing values of the other variables.

1st Cycle Stripping

The first cycle stripping is to back extract uranium from the loaded light phase from the 1st cycle extraction. The purpose of this step is to free the solvent phase of uranium so that it can be recycled back for use in the 1st cycle extraction and at the same time concentrate the uranium in aqueous phase.



There is a dynamic equilibrium between uranium in the organic phase and uranium in the aqueous phase, with the uranium in the organic phase also in equilibrium with the DEHPA-TOPO solvent extractant. To strip uranium from the light phase, uranium in the DEHPA-TOPO must be released, which is done by increasing the acidity of the solution. The next step is to reduce the released uranium VI into uranium IV. This is done by contacting the loaded light phase with strong phosphoric acid with low ORP. The process reduces uranium VI into uranium IV in the light phase and effectively transfers uranium to the heavy phase. The stripping process is done in a mixer-settler as discussed in the first cycle extraction.

2nd Cycle Extraction

Prior to entering extraction circuit, the strip acid from the first cycle stripping is diluted to 27-32% phosphoric acid by adding water. The second cycle extraction operates with the same principles of the first cycle extraction. The loaded strip acid coming from the 1st cycle stripping is diluted to reduce the acidity and followed by increasing the ORP of the solution back to >650 mV by addition of oxidants (H₂O₂). In this step, a low concentration solvent is used to extract, which upon verification in the laboratory proves to be more efficient (Figure 7).

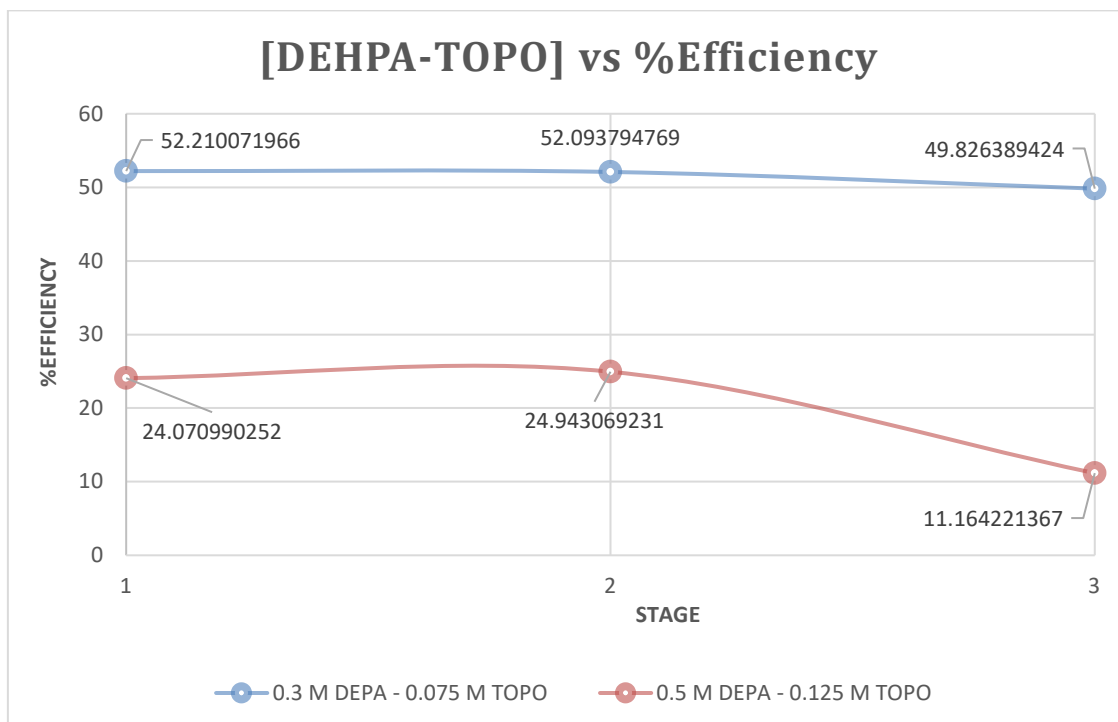


Figure 7. Comparison of the extraction efficiency on of 0.5M DEHPA-0.125M TOPO and 0.3M DEHPA-0.075M TOPO as solvent in the 2nd cycle extraction

In preliminary laboratory experiments, the distribution constant (K_d) of uranium ~27% Phosphoric acid and the 0.3 M DEHPA-0.075 TOPO was determined to be 7.33.

Scrubbing

During extraction of uranium, other elements particularly iron, trace barium, magnesium etc. among others gets coextracted in the process. Solvent scrubbing is necessary to remove other elements as to not contaminate the uranium yellowcake in the following precipitation step. The process is made by contacting loaded light phase in 4-stage mixer-setter: 2 stages of sulfuric acid scrubbing and 2 stages of washing with water). This process precipitates iron, calcium and barium sulfates.

2nd Cycle Stripping

The second cycle stripping is an almost quantitative stripping of uranium from the loaded 0.3 M DEHPA-0.075 M TOPO to the sodium carbonate solution as represented by the following equation:



This process completely removed uranium from the extractant. The light phase can then be recycled back into the 2nd cycle extraction.



Product Precipitation

A two-stage precipitation procedure is conducted. First precipitation is conducted at pH 9, by adding sodium hydroxide into the uranium loaded sodium carbonate solution to precipitate other coextracted elements that is not removed by scrubbing step. A second precipitation is done by further increasing the pH to pH 12 where uranium yellowcake precipitates.

VI. LIST OF EQUIPMENT

Equipment no.	Name	Duty	Size (L)	Material of Construction	Remarks
MT-1	Mixing tank	To mix the raw strong phosphoric acid with the flocculant	500.00	Polypropylene	Provision for level indicator
MT-2	Mixing tank	To mix the DEHPA-TOPO in kerosene mixture for 1st cycle extraction and serves as day tank. Reused for the same purpose in the 2nd cycles extraction.	50	Polypropylene	Provision for level indicator
MT-4	Mixing tank	To precipitate the sodium diuranate and the other compounds	50	Polypropylene	Provision for level indicator
ST-1	Storage tank	To serve as the reservoir for the raw phosphoric acid from PHILPHOS	1000	Polypropylene	
ST-2	Storage tank	To store treated strong phosphoric acid from the activated carbon column	500		
ST-3	Storage tank	To store the waste solvent after cleanup and shutdown	1000	Polypropylene	
ST-4	Storage tank	To store the kerosene washings	500	Polypropylene	



		after cleanup and shutdown			
FT-1	Feed tank/Da y tank	To store the pretreated weak phosphoric acid. Also, used to store the diluted strip acid for 2nd cycle extraction.	400	Polypropylene	Provision for level indicator
FT-2	Feed tank/Da y tank	To store the pretreated strong phosphoric acid and also the store sodium carbonate	50	Polypropylene	Provision for level indicator
FT-3	Feed tank/Da y tank	To store the 1M sulfuric acid for scrubbing	150	Polypropylene	Provision for level indicator
FT-4	Feed tank/Da y tank	to store the process water for scrubbing	150	Polypropylene	Provision for level indicator
PFFP-1	Plate and Frame Filter Press	To filter the suspended solids after flocculation of raw weak phosphoric acid	452	Polypropylene	
ACC-1	Activated Carbon Column	To decrease the optical density of the raw weak phosphoric acid by adsorption of dissolved organic matter.	27.6 kg	Polytetrafluoroethylene	
DT-1	Oxidation tank	To dilute and oxidize(increase ORP) of the treated strong phosphoric acid.	400	Polypropylene	
RT-1	Reduction tank	To decrease ORP of the raw strong phosphoric acid.	50	Polypropylene	
HT-1	Holding tank	To hold the loaded solvent after the 1st cycle extraction for exigencies	50	Polypropylene	Manual level indicators will be introduced to control



					the pump and valve
HT-2	Holding tank	To hold the loaded solvent after the 2nd cycle extraction for exigencies	50	Polypropylene	Manual level indicators will be introduced to control the pump and valve
PT-1	Product Tank	To store the barren weak phosphoric acid from the 1st cycle extraction. Reused as a product tank for the barren phosphoric acid in the 2nd cycle	900	Polypropylene	*based on the accumulation of 180 L strip acid to run a 3-day trial for 2nd cycle extraction at 5 L/hr. Provision for level indicator
PT-1A	Product Tank	To store the barren phosphoric acid from the 2nd cycle extraction			
PT-2	Product Tank	To store the strip phosphoric acid from the 1st cycle stripping	500	Polypropylene	Provision for level indicator
PT-3	Product Tank	To store the barren strip acid from the 2nd cycle extraction	400	Polypropylene	Provision for level indicator
PT-4	Product Tank	To store the scrubbing sulfuric acid from the 2nd cycle scrubbing	150	Polypropylene	Provision for level indicator
PT-5	Product Tank	To store the scrubbing water from the 2nd cycle scrubbing	150	Polypropylene	Provision for level indicator
DT-1	Dilution tank	To dilute the purified acid for	500		



		the 1st cycle extraction			
DT-2	Dilution tank	To dilute the strip acid from the 1st cycle extraction	500	Polypropylene	Provision for level indicator
MSE-1	Mixer-Settler	1st cycle extract: to extract the uranium from the treated weak phosphoric acid using DEHPA-TOPO solvent	Dimensions and internal arrangements to be finalised by the supplier.	Polytetrafluoroethylene	Provision for visualization of settling behaviour of the aqueous and solvent phase. Built-in opening for clean-up and crud removal.
MSE-2	Mixer-Settler	2nd cycle extraction: to extract uranium from diluted strip acid from 1st cycle using DEHPA-TOPO solvent	Dimensions and internal arrangements to be finalised by the supplier.	Polytetrafluoroethylene	Provision for visualization of settling behaviour of the aqueous and solvent phase. Built-in opening for clean-up and crud removal.
MSS-1	Mixer-Settler	1st cycle stripping: To strip the uranium from loaded solvent using treated strong phosphoric acid.	Dimensions and internal arrangements to be finalised by the supplier.	Polytetrafluoroethylene	Provision for visualization of settling behaviour of the aqueous and solvent phase.



					Built-in opening for clean-up and crud removal.
MSS-2	Mixer-Settler	2nd cycle stripping: To strip the uranium from the loaded solvent using ammonium carbonate	Dimensions and internal arrangements to be finalised by the supplier.	Polytetrafluoroethylene	Provision for visualization of settling behaviour of the aqueous and solvent phase. Built-in opening for clean-up and crud removal.
MSSC-1	Mixer-Settler	To scrub the loaded solvent using 1M sulfuric acid removing impurities that will affect the quality of the uranium	Dimensions and internal arrangements to be finalised by the supplier.	Polytetrafluoroethylene	Provision for visualization of settling behaviour of the aqueous and solvent phase. Built-in opening for clean-up and crud removal.
MSSC-2	Mixer-Settler	To scrub the loaded solvent using water to remove entrained acid in the solvent.	Dimensions and internal arrangements to be finalised by the supplier.	Polytetrafluoroethylene	Provision for visualization of settling behaviour of the aqueous and solvent



					phase. Built-in opening for clean- up and crud removal.
HE-1	Water bath	To heat the loaded solvent to 50C before going to 1st cycle stripping	15 L	Stainless Steel 316	
HE-2	Water bath	To cool the recycled solvent to 25C before going back to the 1st cycle extraction	15 L	Stainless Steel 316	
P-1	Pump	To pump the raw phosphoric acid in the reservoir to the pretreatment mixing tank	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-2	Variable Peristalt ic pump	To pump the flocculated weak acid to the filter press	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-3	Variable Peristalt ic pump	To pump the filtered weak acid to the activated carbon column	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-4	Variable Peristalt ic pump	To pump filtered acid from activated carbon column to the storage tank of treated strong phosphoric acid.	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-5	Pump	To pump the treated strong phosphoric acid into the dilution tank and the reduction tank.	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr



P-6	Pump	To pump the treated weak phosphoric acid into the feed tank for first cycle extraction.	Size and dimensions to be finalised by the supplier	Polytetraflouroethylene	Flowrate range is 0.1 - 6 L/hr
P-7	Pump	To pump the strong phosphoric acid into the 1st cycle stripping feed tank	Size and dimensions to be finalised by the supplier	Polytetraflouroethylene	Flowrate range is 0.1 - 6 L/hr
P-8	Variable Peristaltic pump	To pump the weak phosphoric acid into the 1st cycle extraction. Also, used to pump the diluted acid for 2nd cycle extraction	Size and dimensions to be finalised by the supplier	Polytetraflouroethylene	Flowrate range is 0.1 - 6 L/hr
P-9	Variable Peristaltic pump	To pump the barren weak phosphoric acid to the product tank. Also used to pump the barren diluted acid to the product tank	Size and dimensions to be finalised by the supplier	Polytetraflouroethylene	Flowrate range is 0.1 - 6 L/hr
P-10	Variable Peristaltic pump	To pump the solvent phase into to the 1st cycle extraction. Used also to pump the recycle solvent phase to the 2nd cycle extraction	Size and dimensions to be finalised by the supplier	Polytetraflouroethylene	Flowrate range is 0.1 - 6 L/hr
P-11	Pump	To pump the loaded solvent phase into the exigency tank. Also used for 2nd cycle exigency tank	Size and dimensions to be finalised by the supplier	Polytetraflouroethylene	Flowrate range is 0.1 - 6 L/hr
P-12	Variable Peristaltic pump	To pump the loaded solvent phase into the 1st cycle stripping	Size and dimensions to be finalised	Polytetraflouroethylene	Flowrate range is 0.1 - 6 L/hr



			by the supplier		
P-13	Variable Peristaltic pump	To pump to recycle barren solvent phase back to the 1st cycle extraction	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-14	Variable Peristaltic pump	To pump the treated strong phosphoric acid into the 1st cycle stripping	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-15	Variable Peristaltic pump	To pump the loaded strong phosphoric acid into the product tank	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-16	Pump	To pump the stock DEHPA into the mixing tank	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-17	Pump	To pump the kerosene into the mixing tank	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-18	Variable Peristaltic pump	To pump the diluted phosphoric acid into a feed tank for 2nd cycle	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-19	Variable Peristaltic pump	To pump the loaded solvent phase into the sulfuric acid scrubbing	Size and dimensions to be finalised by the supplier	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-20	Variable Peristaltic pump	To pump the 1 M sulfuric acid into the scrubber	Size and dimensions to be	Polytetraflouroethyl ene	Flowrate range is 0.1 - 6 L/hr



			finalised by the supplier		
P-21	Variable Peristaltic pump	To pump the loaded sulfuric acid into product tank	Size and dimensions to be finalised by the supplier	Polytetrafluoroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-22	Variable Peristaltic pump	To pump the process water to the 2nd set scrubbers	Size and dimensions to be finalised by the supplier	Polytetrafluoroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-23	Variable Peristaltic pump	To pump the wash water into the product tank	Size and dimensions to be finalised by the supplier	Polytetrafluoroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-24	Variable Peristaltic pump	To pump the scrubbed solvent phase into the 2nd cycle stripping	Size and dimensions to be finalised by the supplier	Polytetrafluoroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-25	Variable Peristaltic pump	To pump the recycle solvent phase back to the 2nd cycle extraction	Size and dimensions to be finalised by the supplier	Polytetrafluoroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-26	Variable Peristaltic pump	To pump the sodium carbonate solution into the 2nd cycle stripping	Size and dimensions to be finalised by the supplier	Polytetrafluoroethyl ene	Flowrate range is 0.1 - 6 L/hr
P-27	Variable Peristaltic pump	To pump the uranium-loaded sodium carbonate to the mixing tank	Size and dimensions to be finalised by the supplier	Polytetrafluoroethyl ene	Flowrate range is 0.1 - 6 L/hr



VII. LIST OF CHEMICALS

1. Process Reagent

***Note to SX System Vendor: For reference only to better understand the reagents that will be used in the Lab-Scale Pilot SX System.**

Reagent	Estimated Qty	Purpose
Di-ethylhexyl Phosphoric acid (DEHPA) Technical/Industrial Grade	200 L	Organic extractant for Uranium
Trioctyl Phosphine Oxide (TOPO) Technical/Industrial Grade	100 kgs	Organic extractant for Uranium
Kerosene (Technical/Industrial Grade)	500 L	Solvent for DEHPA and TOPO
Hydrogen peroxide (33-34% technical grade)	60 L	Oxidant
Iron metal	2 kg	Reductant
Sulfuric acid (technical grade)	30 L	Used in scrubbing of uranium loaded organic phase
Sodium carbonate (technical grade)	3 kg	Used in stripping uranium from loaded organic phase
Sodium hydroxide (technical grade), pellets	1 kg	Used in pH adjustment of solution during precipitation of uranium yellowcake.

2. Analytical Reagent

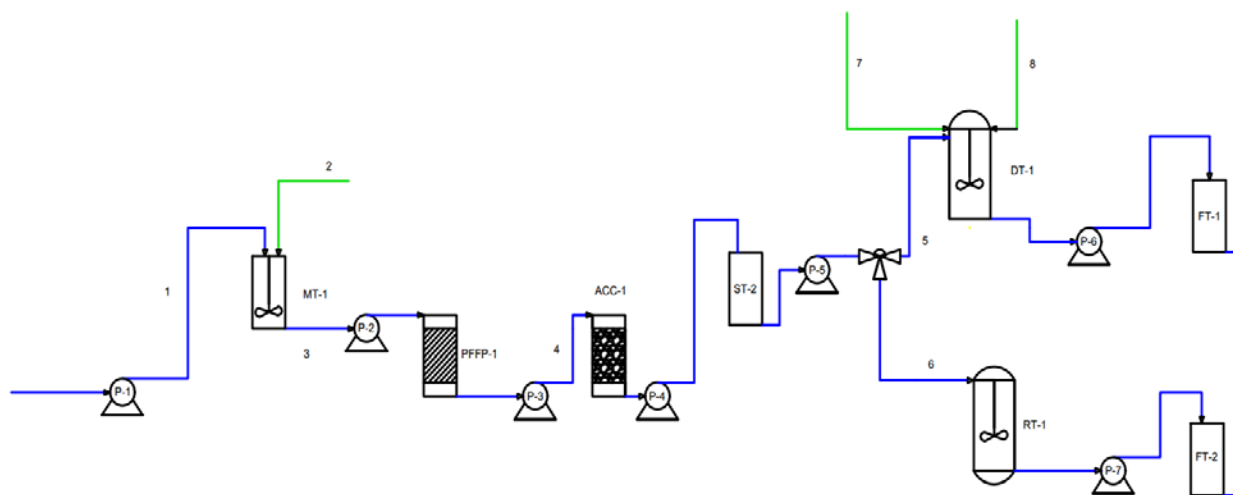
Reagent	Estimated Qty	Purpose
Arsenazo III (analytical reagent (AR) grade)	20 g	Chromogenic reagent used in the spectrophotometric determination of uranium
Hydrochloric acid (AR grade) 37%, 2.5 L	5 bottles	Used for acid digestion
Nitric acid (AR grade) 65%, 2.5 L	5 bottles	Used for acid digestion
Uranyl nitrate hexahydrate (AR grade), 25 g	2 bottles	Serves as uranium standard in chemical analysis

VIII. DETAILED PROCESS FLOW DIAGRAM AND MATERIAL MASS BALANCE



Please see attached pdf file “Process Flow Diagram, Pretreatment, First and Second Stages” for an overall view of the SX pilot process.

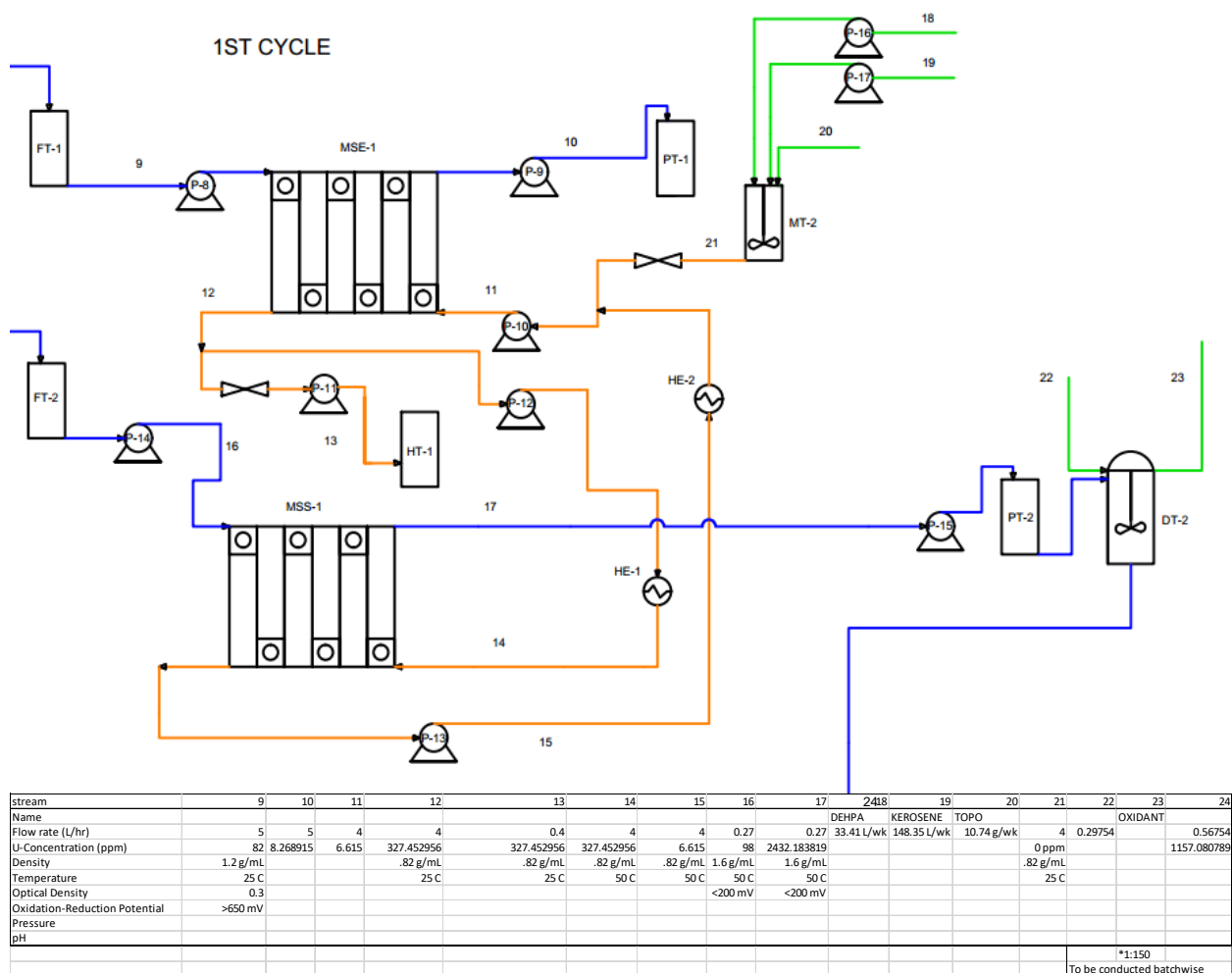
Pretreatment Flow Diagram and Material Mass Balance



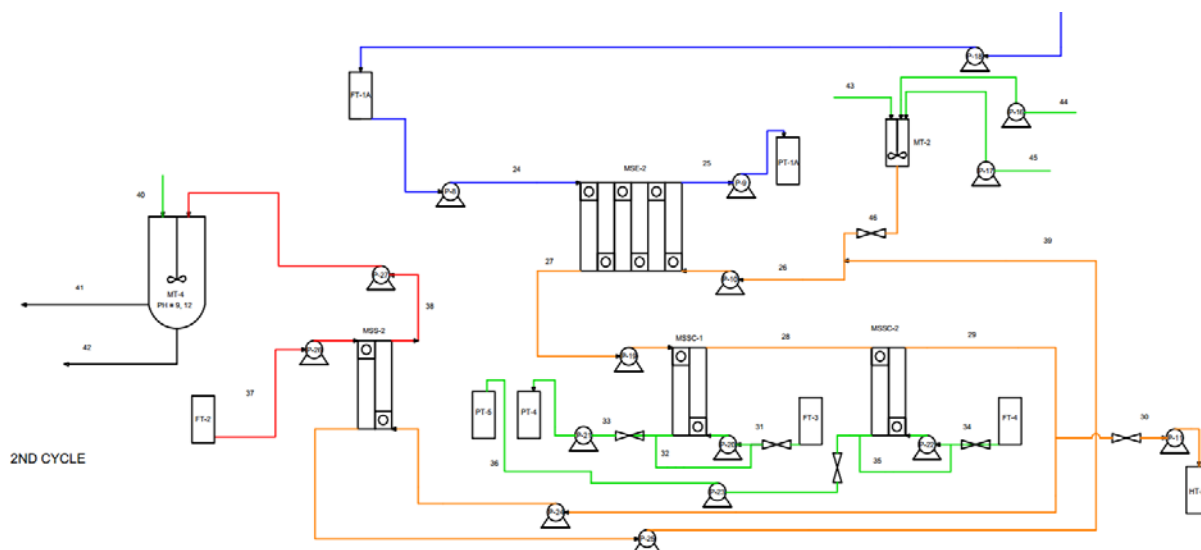
PRETREATMENT

stream	1	2	3	4	5	6	7	8	9	16	11	12	13	14
Name		Floculant				Oxidant	Water							
Flow rate	162.72 L	65.09 g*	162.72 L	162.72 L	142.72 L	20 L	0.95 L**	157.28 L	300 L	20 L	20.107 L/ 2 days	18.096 L/ 2 days	18.096 L/ 2 days	.27 L
U-Concentration	172 ppm		172 ppm	172 ppm	172 ppm	172 ppm			82 ppm	172 ppm				98 ppm
Density														1.6 g/mL
Temperature								25 C						50 C
Optical Density						0.3							0.5	<200 mV
Oxidation-Reduction Potential														
Pressure														
pH														
Process water														
Hydrogen Peroxide (oxidant)														
Magnafloc (Floculant)														
		*0.4 g Floculant per L acid					**1:150 Phos acid to hydrogen peroxide oxidant volume ratio							
Pretreatment is conducted batchwise every 2 days.														

First Cycle Flow Diagram and Material Mass Balance



Second Cycle Flow Diagram and Material Mass Balance



Stream	24	25	26	27	30	27	28	29	*31	*32	*33	**34	**35	**36	37	38	39	40	41	42
Name																				
Flow rate (per hour)	5 L	5 L	4 L	4 L	4 L	4 L	4 L	4 L	4 L	4 L	4 L	4 L	4 L	4 L	.27 L	.27 L	4 L			
U-Concentration	1157.080789	0.023604855	0	1446.32148	1446.32148	1446.32148	1446.32148	1446.32148	0 ppm	0 ppm	0 ppm	0 ppm	0 ppm	0 ppm	0 ppm	21422.34	0.313357			
Density	1.2 g/mL		.81 g/mL			.81 g/mL														
Temperature	25 C		25 C			25 C														
Optical Density	0.3																			
Oxidation-Reduction Potential	>650 mV																			
Pressure																				
pH																			9	12
Stream	43	44	45	46																
Name	TOPO	DEHPA	KEROSENE																	
Flow rate (per hour)	6.44 g/wk	20.04 L/wk	167.48 L/wk	4 L																
U-Concentration				0 ppm																
Density				0.81 ppm																
Temperature				25 C																
Optical Density																				
Oxidation-Reduction Potential																				
Pressure																				
pH																				

IX. PLOT PLAN

Please see attached pdf file with “SX Pilot System_General Arrangement Diagram”

X. OPERATIONAL PHILOSOPHY

Sample collection and Storage

Raw phosphoric acid (48-53%) is collected at the Philippine Phosphate Fertilizer Corporation (PHILPHOS), Isabel Leyte. Per experimental trial, a total of 900 L phosphoric acid enough to conduct a 7-day continuous operation will be collected. From the plant site, 300L of acid contained in 15 carboys (20 L capacity) will be transported by land to PNRI. A total of three (3) trips is needed to collect the entire 900L volume. The collected acid will then be transferred into the strong phosphoric acid storage (SPA) tank (1000 L capacity).

Solvent Preparation

In the conduct of 7-day continuous run of the scale-up facility, the following solvents are prepared from the following reagents:

DEHPA: density= 995 g/L; 97% purity; MW=322.43 g/mol



TOPO: 90% purity; MW=386.63 g/mol
H₂SO₄: 1.840 g/mL; 95% purity; MW=98.01 g/mol

Preparation of 0.5 M DEHPA – 0.125 M TOPO in kerosene

For use in the 1st cycle extraction a total of 50 L of solvent of 0.5 M DEHPA – 0.125 M TOPO in kerosene is prepared by pumping 8.35 L of DEHPA, 37.09 L kerosene and manually add 2.69 kg TOPO into a 70 L mixing tank. The solvent is mixed until TOPO completely dissolves.

Preparation of 0.3 M DEHPA - 0.075M TOPO in kerosene

For use in the 2nd cycle extraction, 50 L of 0.3 M DEHPA – 0.075 M TOPO in kerosene is prepared by pumping 5.01 L of DEHPA, 41.96 L kerosene and manually add 1.61 kg TOPO into a 70 L mixing tank. The solvent is mixed until TOPO completely dissolves.

Preparation of 1 M H₂SO₄ Scrubbing solution

For use in the 2nd cycle extraction-stripping, the 150 L of 1M H₂SO₄ is prepared by pumping 8.42 L of conc. H₂SO₄ and 134.54 L of water into a 300 L mixing tank.

Pretreatment of Strong Phosphoric acid

The feed acid for the 1st cycle extraction is 27-32% P₂O₅ content acid. The private company partner PHILPHOS currently does not produce the type of required acid but instead imports strong phosphoric acid (43-53% P₂O₅ content acid). The strong phosphoric acid will be used in place of the weak acid until PHILPHOS will resume production of weak acid.

From the SPA storage, the acid is stirred to disperse settled matter (to emulate the suspended matter in fresh acid from phosphoric acid plant), 300 L will be pumped into the mixing tank where the acid is added with 120 g of Magnafloc polyacrylamide flocculant (corresponding to 0.4 g flocculant per liter acid), mixed for 10 mins, settled for 20 mins and then pumped into the filter press. The acid is then clarified by passing through the activated carbon column. The optical density of the acid will be checked to fall in the range of 0.5-1.5 using a UV-VIS spectrophotometer at 408 nm. The initial ORP of the acid will then be measured. After the filter press, the acid will then be pumped into a holding tank where it is connected via three-way valve into the oxidation tank (leading to 1st cycle extraction) and the reduction tank (leading to 1st cycle stripping).

A target of 300L of treated 27% P₂O₅ content acid will be prepared as feed for the first cycle extraction, this volume will be adequate to sustain the extraction setup for 2.5 days. A volume of 142.72 L of clarified strong phosphoric acid will be pumped into the dilution tank and diluted to 300 L volume (1L of 48% P₂O₅ produces 2.1020 L of 27% P₂O₅ acid). The ORP will then be increased to >650 mV by adding 30% hydrogen peroxide solution. The amount of oxidant added, and the total time of oxidation will be recorded. The treated weak acid will then be pumped into the day tank (400 L feed tank) for 1st cycle extraction. Pretreatment of phosphoric acid is conducted every 2 days to sustain continuous operation of the setup.

From the clarified strong phosphoric acid storage, 20 L of phosphoric acid is pumped into the reduction tank (mixing tank containing iron balls) to reduce acid ORP to <200 mV. The initial ORP, final ORP and total time for reduction of ORP will be recorded. The reduced acid will then be pumped into the day tank for the 1st cycle stripping (50L holding tank).



1st cycle Extraction

From the treated weak phosphoric acid day tank, the aqueous phase is pumped into the mixer-settler (6-stages) at a flow rate of 5 L/hr. Simultaneously, the valve connecting the solvent phase storage is opened to allow pumping of solvent into mixer-settler at a flow rate of 4 L/hr resulting to an A/O ratio of 1:0.8. The stirring speed in the mixer will be set to 300 rpm. The uranium barren aqueous phase exiting the mixer-settlers will then be pumped into a holding tank for storage (400 L capacity). The loaded solvent phase will then be pumped into a heat exchanger (coils immersed in water bath) to increase temperature to 50 deg C before entering the 1st cycle stripping. Uranium barren solvent phase exiting the 1st cycle stripping is passed through a cooler then recycled back into the 1st cycle extraction mixer-settlers. The valve connecting the solvent phase storage will remain open until the solvent phase exigency storage tank (50 L capacity) is adequately filled.

A sample of the aqueous and solvent phase will be drawn from the settling area of each mixer-settler every 4 hrs. intervals for analysis.

1st cycle stripping

With the first stream of loaded solvent phase exiting the 1st cycle extraction, aqueous phase from the treated strong phosphoric acid day tank is then pumped into the mixer settler (6 stages) at a volume flow rate of 0.27 L/hr resulting to an O/A ratio of 15:1. This will present operational difficulties but will perform better enrichment of uranium in the aqueous phase. The exiting loaded aqueous phase will then be collected in a storage tank (200 L capacity) for subsequent use for the 2nd cycle extraction while the uranium barren solvent phase is recycled back into the 1st cycle extraction. A sample of the aqueous and solvent phase will be taken every 4 hrs. interval for laboratory analysis.

The whole of the 1st cycle extraction and stripping will be operated continuously for 7 days and will be monitored by at least 2 persons. During operation, the operator should check the levels of phosphoric acid in the day tank for both the strong and weak phosphoric acids. Once the level of phosphoric acid in the day tank reaches the 20L mark, the operator should immediately notify that pretreatment for a new batch of feed acid should commence if it has not started yet. Additionally, operator should record the amount of crud formation in the mixer-settlers every 3 hours to determine the rate of accumulation.

2nd cycle extraction-stripping

For the second cycle extraction-stripping of uranium, the same equipment from the first cycle circuit will be utilized and reconfigured. The following are needed:

1. A dilution tank for diluting the strip acid from the 1st cycle stripping back to 27% P₂O₅ content.
2. An oxidation tank for raising ORP of diluted strip acid.
3. Mixer-settler for 6-stages of 2nd cycle extraction, 4-stages of scrubbing and 2-stages of uranium stripping by sodium carbonate; and



4. Pumps and piping will be rearranged appropriately to follow process flowsheet for 2nd cycle extraction-stripping.

Dilution and ORP adjustment

After accumulation of 450 L of uranium loaded aqueous phase, the acid is then pumped into a mixing tank to reduce P₂O₅ content to 27% by addition of water. The resulting aqueous phase is then added with hydrogen peroxide to raise the ORP back to >650 mV. The initial ORP, final ORP and amount of oxidant will be recorded, and a sample will be taken for chemical analysis of the P₂O₅ and uranium content. The treated acid is then pumped into the day tank for the 2nd cycle extraction mixer-settler. This step is conducted batchwise, converting all collected acid into 27% P₂O₅ content acid prior to storing.

2nd Cycle Extraction

In this step, a low concentration solvent phase of 0.3 M DEHPA-0.075 M TOPO is used to extract uranium from aqueous phase. From the day tank, the aqueous phase is pumped into the 2nd cycle extraction (6-stages) at a flow rate of 5 L/hr. Simultaneously, the valve connecting the solvent phase (0.3M DEHPA-0.075M TOPO in kerosene) storage is pumped to the mixer-settlers at a flow rate of 4 L/hr, operating at A/O ratio equal to 1:0.8. The loaded solvent phase will then be pumped into the scrubbing and uranium stripping mixer-settler prior to recycling. The valve connecting the solvent phase storage will remain open until the solvent phase exigency storage tank (50 L capacity) is adequately filled (40L mark). The stirring speed in the mixer will be set to 300 rpm. A sample from the aqueous phase and solvent phase will be drawn every 4 hrs interval for analysis.

Scrubbing

The exiting loaded solvent phase from the 2nd cycle extraction is pumped into a 2-stage mixer settler for sulfuric acid scrubbing. The aqueous phase, which is 1 M sulfuric acid, will be pumped at the same rate of 4 L/hour operating at A/O ratio equal to 1. The scrubbed solvent phase is then pumped into a 2-stage scrubbing with water to remove trace sulfuric acid. Water is pumped into the mixer-settler at the same flow rate of 4L/hr. Sulfuric acid and water will be recycled to their respective mixer-settlers with a provision for drainage and addition of fresh acid and water. The exiting scrub solvent phase will then be pumped into the 2nd cycle stripping then recycled back into the 2nd cycle extraction mixer-settler. A sample will be drawn for laboratory analysis every 4 hrs interval.

2nd Cycle Stripping

The scrubbed loaded solvent phase is pumped into a 2-stage mixer-settler at a flow rate 4 L/hr and at the same time 30% (m/v) sodium carbonate stripping solution is pumped at a flow rate of 0.27 L/hr operating at a A:O ratio of 1:15. The solvent phase is recycled back into the 2nd cycle extraction. The loaded strip solution is then pumped into a storage tank for subsequent use in the precipitation of uranium yellowcake. A sample will be drawn every 4 hrs. interval for laboratory analysis.



During operation, the operator should check the levels of phosphoric acid in the day tank of weak phosphoric acid. Crud formation should be monitored in the 2nd cycle extraction. The pH and amount of precipitates in the scrubbing stages should also be monitored. The pH of the water should not reach the pH of 1M sulfuric acid in the first stage, otherwise, the water in its holding tank is partially drained and pumped with fresh water.

Product Precipitation

With the collected uranium-loaded carbonate solution, a two-stage precipitation procedure will be conducted in a mixing tank (50 L capacity). This will be conducted batchwise. First precipitation is conducted at pH 9, by adding 30% sodium hydroxide to precipitate other coextracted elements, followed by filtration to remove the precipitates. The second precipitation is done by further increasing the pH to pH 12. Once it reaches the target pH, the solution will be continuously stirred for 10 hours at 100 rpm to allow complete uranium yellowcake precipitation. The uranium yellowcake product will then be filtered and dried. The barren solution will then be stored, and a sample will be drawn for analysis.

XI. START-UP AND SHUTDOWN

1st cycle extraction and Stripping

Startup

The first step in starting up the recovery plant consists of preparing the service streams namely: 0.5 M DEHPA-0.125 M TOPO in kerosene (solvent phase for 1st cycle extraction), treated weak phosphoric acid (aqueous phase for 1st cycle extraction), and treated strong phosphoric acid (aqueous phase for 1st cycle stripping). Preparation of solvent phase and all aqueous phase starts batchwise where they are pump into their respective day tanks. Operator should inspect the volume of each liquid in the day tanks. Volume of solvent phase should be at the 200-L mark at the start of the operation. The volume of the treated weak phosphoric acid is at 300 L and the volume of treated strong phosphoric acid should be at 20-L mark. Once the feed solutions are available, inspection of pipes is done to insure proper connections and tight fittings. Pump flow rates are set according to the following:

Treated weak phosphoric acid to mixer-settler: 5 L/hr

Treated strong phosphoric acid to mixer-settler: 0.27 L/hr

Solvent phase to extraction and stripping mixer-settler: 4 L/hr

The valve for the solvent phase is then opened and the process is then commenced. Once solvent and aqueous phase are completely flowing in both extraction and stripping stages, the operator should check the flow meters to verify the flowrates of the aqueous phase and solvent phase. Depending on the reading of the flow meters the pumps are increased or decreased accordingly to achieve the desired flow rates. The setup is then allowed to run for 1 hour before opening the solvent exigency tank. Once the solvent exigency tank fills to 40 L, the valve for the exigency tank and the fresh solvent mixing tank will be closed.



Shutdown

On the last day (7th day) of the operation, the operator must check all the levels of the day tanks. Once the certain level of fluid is achieved, as specified below, the pumps are then turned off.

Weak phosphoric acid day tank: 0.1 m
Strong phosphoric acid day tank: 0.06 m
Solvent phase mixing tank: .06 m

This is followed by draining the pipes and tanks into their respective waste storage and holding tanks.

Clean-up

After draining the excess phosphoric acid in both weak and strong phosphoric acid day tanks, and the product tank for the weak barren phosphoric acid, all tanks are rinsed with water. Then 30 L of tap water is pumped into both day tanks. The mixer-settler will be scrubbed and cleaned to remove any solids, contaminants that may adhere onto the walls. The mixing tank of the solvent phase is also drained, then 24 L kerosene is pumped into the mixing tank. The 1st cycle extraction and stripping will be operated for 5 hours using the water and kerosene as aqueous and solvent phase.

2nd cycle extraction and Stripping

Start-up

The 2nd cycle extraction and stripping will commence once the total volume of the strip acid in the product tank is approximately 480 L.

The next step consists of batchwise preparation of the service process streams namely: 0.3 M DEHPA-0.075 M TOPO in kerosene (solvent phase for 2nd cycle extraction), oxidized dilute phosphoric acid (aqueous phase for 2nd cycle extraction), 1M sulfuric acid (scrubbing solution for 2nd cycle scrubbing), and the 30% (m/v) sodium carbonate (aqueous phase for 2nd cycle stripping). The service process streams are then pumped into their respective day tanks. Operator should inspect the volume of each liquid in the day tanks. Volume of solvent phase should be at the 200-L mark at the start of the operation. The volume of the diluted phosphoric acid is at 300 L mark, the 1M sulfuric acid and process water should be at 120 L mark, and the volume of 30% (m/v) sodium carbonate should be at 20-L mark. Once the feed solutions are available, inspection of pipes is done to insure proper connections and tight fittings. Flow rates are set according to the following:

Phosphoric acid to mixer-settler: 5 L/hr
30% (m/v) sodium carbonate to mixer-settler: 0.27 L/hr
Solvent phase to extraction and stripping mixer-settler: 4 L/hr
1M sulfuric acid to 1st scrubbing mixer-settler: 4 L/hr
Process water to 2nd scrubbing mixer-settler: 4 L/hr

The valve for the solvent phase is then opened and the process is then commenced. Once solvent and aqueous phase are completely flowing in the extraction, scrubbing, and stripping stages,



the operator should check the flow meters to verify the flowrates of the aqueous phase, scrubbing phase, and the solvent phase. Depending on the reading of the flow meters the pumps are increased or decreased accordingly to achieve the desired flow rates. The setup is allowed to run for 1 hour before opening the solvent exigency tank. Once the solvent exigency tank fills to 40 L, the valve will be closed.

Shutdown

On the last day (7th day) of the operation, the operator must check all the levels of the day tanks. Once the certain level of fluid is achieved, as specified below, the pumps are then turned off.

Phosphoric acid day tank: 0.1 m
1M sulfuric acid day tank: 0.09 m
Process water day tank: 0.09 m
Solvent phase mixing tank: 0.06 m

This is followed by draining the pipes and tanks into their respective waste storage and holding tanks.

The following are the conditions that may require emergency shut-down:

1. Pipe rupture
2. Pump cavitation
3. Pipe, valve, or fitting leaks
4. Power interruptions.
5. Tank overflow.
6. Fire hazard.
7. Natural calamity hazards.

Below are the procedures for emergency shutdown:

1. Flip the breaker switch.
2. Close all valves.
3. Call for a service engineer to fix pipe rupture, cavitation, and leaks.
4. In case of fire, prepare the fire extinguisher and execute SOP for fire safety.
5. In case of chemical spill, evacuate the area, and call the chemical safety officer.
6. In case of earthquake, flip the breaker switch, and follow standard protocols from earthquake drills.
7. In case of power interruptions, connect the process to the power generator.

Special Operational Needs

Crud formation

If operating at high optical density (beyond required OD) a stable emulsion or crud tends to form at the interphase of the aqueous and organic extractant. This makes it difficult to separate the two phases and could lead to loss of expensive solvent as well as significant reduction of extraction efficiency. A transparent slit that goes along the height of the mixer-settlers can be



useful in visually monitoring the height of the crud. The crud can then be manually pumped out of the tank and place into a centrifuge to separate and recover the solvent.

Precipitate buildup in scrubbing section

During scrubbing of loaded solvent phase some of sulfate precipitates at the bottom of the settlers, periodic removal of buildup is required.