

## **Uranium-REE Keynote**

## PLANNING FOR A SUCCESSFUL URANIUM PILOT PLANT PROGRAM

Bу

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## ABSTRACT

Many there are in Project Development and Engineering that consider Pilot Plant work before understanding all they should about their uranium containing feed ore(s). Premature commitment to piloting can, inter alia, result in the adoption of an inappropriate flowsheet in piloting. The consequence of this outcome can be far reaching; from project closure on the one hand because of cost and or schedule blow out, through to adopting a flowsheet yielding a sub-optimal return on the other.

This paper addresses the key aspects in the preparation and planning a pilot plant for a uranium project.

Keywords: Pilot Plant, Uranium, Sample Selection, Batch Testing, Variability Testing, Data Interpretation



#### INTRODUCTION

Many there are in Project Development and Engineering that consider Pilot Plant work before understanding all they need to about their uranium containing feed ore(s). Premature commitment to piloting can, inter alia, result in the adoption of an inappropriate flowsheet. The consequence of this outcome can be far reaching; from project closure on the one hand through to adopting a flowsheet yielding a sub-optimal return on the other.

The procedure advocated for planning for a uranium pilot plant is no different to that employed for most metals apart from some of the unique features associated with radiation.

Pre-pilot preparatory work can take from several months to several years and its duration is normally related to the complexity and variability of the ores to be processed.

This paper will address the importance of:

- Sample selection, representivity and mineralogy,
- Understanding project and process constraints, for example, environmental factors, impurity deportment, gangue reagent consumers, water quality etc.,
- Upgrade opportunities,
- Batch and batch variability testing,
- Early mass balances,
- Flowsheeting and interim project costing,
- Scoping and managing the pilot plant campaign.

#### SAMPLE SELECTION AND REPRESENTIVITY

Sample selection and representivity are important to ensure that the ore submitted to testwork is relevant to the mine plan and the resource. Selecting unsuitable samples or non-representative samples may result in an:

- incorrect or a sub-optimal process flowsheet being selected,
- incorrect economic analysis on the viability of the project as testwork may show lower or higher than expected recovery and reagent consumption.

The importance of understanding the orebody cannot be understated. A recent uranium acid leach project in South Africa is an example where there was insufficient knowledge of the orebody. This resulted in a lower than expected uranium head grade to the plant and with that the plant has been shut down.

There have been similar cases in Africa where alkaline leach processes have failed because of inter alia insufficient knowledge of the orebody before committing to the project.

Ore sampling and committing the samples to a rigorous testing regiment has no substitute in the development of uranium projects.

Sampling can be undertaken using several different methods depending on the type and location of the deposits. Methods such as trench sampling shown in Figure 1 and Figure 2 may be suitable for surficial deposits whereas diamond drill core sampling (shown in Figure 3) may be more suitable for deeper deposits.





Figure 1: Radiometry mapping of trench wall (Courtesy of JUMCO)



Figure 2: Channel sample cut in the trench wall by a diamond saw (Courtesy of JUMCO)



Figure 3: Diamond drill core sample<sup>(1)</sup> (Courtesy of A-Cap Resources)

Metallurgical samples should be split from material that represents the orebody:

- Spatially,
- At depth,
- By lithology, and
- In its variability.

## **PROJECT AND PROCESS CONSTRAINTS**

Two key project constraints will be discussed, namely;

• effluent, and



water.

#### **Environmental and Effluent**

The selection of a process flowsheet will need to take into consideration environmental constraints and effluent management. Tailings disposal in open dams which have been common practice to current time is unlikely to be condoned in the future as issues such as tailings dam failures, seepage and acid mine drainage become apparent after mine closure.

An example of a uranium legacy mine site is Rum Jungle which was closed in 1971 and subsequently there was significant environmental damage from acid and metalliferous drainage polluting the Finniss River<sup>(2)</sup>. Remediation work on the Rum Jungle site was first undertaken in the 1980s and is still ongoing today to improve water quality, vegetation and aesthetics.



Figure 4: Rum Jungle Legacy Mine Remediation Site<sup>(2)</sup>

The best practices today as recommended by the Nuclear Regulatory Commission for the management of uranium tailings is to store dry tailings in below grade disposal sites<sup>(3)</sup>. This will require the tailings to be filtered rather than disposed of into a tailings dam as slurry. An example of a site which has adopted below grade dry tailings disposal is the Moab Tailings Relocation project. The task at hand involves excavating 14.5 million tonnes of inactive tailings and relocating it to a below-grade disposal site approximately 50km away at Crescent Junction in Colorado<sup>(4)</sup>. At the disposal site, the tailings are stacked to a depth of 7.5m below grade and 7.5m above grade and capped with 2.5m thick multi-layer cover as shown in Figure 7. The ground water at the MOAB tailings site is also being remediated to remove <sup>226</sup>Ra from seepage fluids.





Figure 5: Moab Tailings Site<sup>(4)</sup> (Courtesy of US DOE)



#### Figure 6: Below Grade Tailings Disposal Site at Crescent Junction<sup>(4)</sup> (Courtesy of US DOE)



The upliftment of uranium tailings and relocation to another site followed by remediation of the first and capping at the second site is costly and would unlikely have been envisaged in the initial project economics.

Hitherto, governments and tax payers have often been called on to fund these remediation activities, a practice that is unlikely to be acceptable going forward.

Operators should therefore only consider a flowsheet that embraces a residue or tailings concept that adequately contains the waste components and in the case of uranium, its daughters as well.



Flowsheets that are tested at bench and pilot scale should therefore give consideration to incorporate a final residue form that meets these requirements. This will invariably include receiving return fluids and leachates into the process from the tailings facility, which in turn may result in elevated concentrations of conservative elements within the circulating process liquors.

#### Water

Water and water quality are critical to the hydrometallurgical recovery of uranium from ores and concentrates.

Some factors that need to be considered in the metallurgical testing are:

- regional water salinity and the expected water quality available to the leach process,
- regional water availability and possible restrictions on use,
- if water de-ionisation is required for the process, and the constraints that will likely be placed on the disposal of the concentrate (brine).

There are limits on the concentration of chlorides in water, not only from materials of construction perspective but primarily because chloride is a competing ion in the transfer processes of solvent extraction and ion exchange.

#### **MINERALOGY**

#### **Gangue Reagent Consumers**

The method for processing an ore for the recovery of uranium is determined by the ore's mineralogy. The mineralogy of the gangue minerals rather than that of the uranium minerals often determine the leach regime employed.

In leach processes employing sulfuric acid, only a relatively small quantity of acid is gainfully employed in extracting uranium from the host ore. The remainder of the acid is consumed by the gangue constituent minerals. There are new projects that are being considered today have a gangue acid consumption in excess of 95% of the total applied fresh acid.

Many of the gangue elements that are solubilised by sulfuric acid report to the leachate where they build up in concentration within a closed flowsheet. In some cases, the impact of this may be deleterious to the extraction of uranium, whilst in others there have been some benefits identified when leaching in high solute concentration solutions.

Similarly, in alkaline leaching, where the unit reagent cost is approximately 3 times that of sulfuric acid, the ore that is leached must also have economically low levels of reagent consumption.

In the Namibia, West Africa, Jordan and other desert locations where the uranium is carbonate hosted for example, the upper ores are often not treated because they contain elevated levels of labile sulfate e.g. strontium and calcium sulfate which increase the reagent consumption.

#### **Quantitative Mineralogy**

It is important to quantify the reagent consuming gangue minerals both in the leach feed and the leach residue in order to construct the mass balance.

Biotite, for example, is common in many ores and reacts with sulfuric acid.



Figure 8: Biotite Minerals<sup>(5)</sup> (KMg<sub>1.5</sub>Fe<sub>1.5</sub>(AlSi<sub>3</sub>O<sub>10</sub>)(OH)<sub>2</sub>)

 $2KMg_{1.5}Fe_{1.5}(AISi_3O_{10})(OH)_2(s) + 6H_2SO_4(aq) \rightarrow 2KAISi_3O_8(s) + 3MgSO_4(aq) + 3FeSO_4(aq) + 8H_2O(1) + 2KAISi_3O_8(s) + 3MgSO_4(aq) + 3FeSO_4(aq) + 2KAISi_3O_8(s) + 3KAISi_3O_8(s) + 3KA$ 

Carbonates of the type ankerite, dolomite etc, consumes acid in a n acid heap leach operation. Invariably at the toe, gypsum crystallization is evident as the leach front moves through heap.



Figure 9: Gypsum precipitate at the toe of an Acid Leach Pad

 $\begin{array}{l} Ca(Fe,Mg)(CO_3)_2(s) + 2H_2SO_4(aq) \rightarrow CaSO_4(aq) + 0.5 \ MgSO_4(aq) + 0.5 \ FeSO_4(aq) + 2H_2CO_3(aq) \\ CaSO_4(aq) \xrightarrow{H_2 0} CaSO_4 \cdot 2H_2O(s) \end{array}$ 

Simple elemental chemistry is both insufficient and inadequate to model the uranium and related gangue minerals in the leaching process. Quantitative mineralogy will provide a clear indication of the mineral alterations in the leach and a basis and foundation for the mass balance.

## **Uranium Minerals Particle Size and Liberation**

The required ore particle size can impact the flowsheet selection as it can impact the comminution step required to liberate the uranium minerals.

Figure 10 shows some QEMSCAN particle images in which the uranium is either locked or liberated in copper-iron-sulfides and oxides. Accessing the uranium that is locked may be facilitated by further particle size reduction or similar means.





#### Figure 10: QEMSCAN Particle Image of Uranium Bearing Minerals in Copper Concentrate

## **UPGRADE PROCESSES**

Some uranium ores are amenable to upgrade processes. Upgrading has several benefits:

- Converting lower grade ores into an economic feed grade,
- Reducing the size of processing plant lower capital cost,
- Reducing reagent consumption as less reagent will be wasted in gangue ore leaching lower operating cost,
- Reducing the amount of processed tailings and hence the environmental footprint of a project.

The following processes have been examined for upgrading uranium ores:

**Radiometric Ore Sorting (ROS)** - the uranium distribution in the ore must be heterogeneous (e.g. Vein) and uranium and radium must be in equilibrium<sup>(6)</sup> for it to be a possible candidate for ROS.

**Flotation** – flotation has been used to float sulfides to reject gangue minerals or to recover secondary product. Olympic Dam employs this process<sup>(6)</sup>.

**Scrub and Screen** – Scrubbing and screening upgrading has been employed on surficial ore to recover a uranium concentrate. Langer Heinrich currently employs this process<sup>(8)</sup>.

**U-PGrade<sup>™</sup> Process** – This has been patented by Marenica Energy and employs scrubbing, size separation by screening elutriation and cyclone and standard carbonate removal. The first commercial application is reported likely to be the Reptile Uranium Namibia Tumas Project<sup>(9)</sup>.





Figure 11: Schematic of Radiometric Ore Sorter<sup>(7)</sup>

## PLANNING BATCH TESTWORK

## **Testwork Quality**

The quality of testwork has been found to have a direct impact on plant start-up and more importantly on ramp-up and hence project economics.

The duration of a plant's ramp up period and whether at the end of ramp-up the plant achieves name plate capacity can invariably be linked to the diligence of the project team to include appropriate and sufficient test work.

Delays in plant startup can be costly and in some cases, it can result in the plant's premature shutdown. Based on the startup pattern of 41 greenfields projects, McNulty<sup>(10)</sup> found that there was a link between the extent of testing conducted during the process development and the plant ramp up time. This effect is shown in Figure 12. The characteristics for each series can be summarised as follows with respect to the testwork component:

- Series 1 the plant flowsheet was based on mature technology. Thorough pilot plant testing was conducted on potentially risky unit operations,
- Series 2 the plant flowsheet was based on a new process with incomplete pilot plant testing or pilot testing that was conducted on non-representative samples,
- Series 3 Very limited pilot plant testing conducted on the plant flowsheet with some important process steps ignored. Feed characteristics were poorly understood,
- Series 4 No pilot plant testing conducted on the plant flowsheet or process chemistry was poorly understood.





Figure 12: Design Feed Rate Achieved and Ramp- Up Time (McNulty Series 1 - 4<sup>(10)</sup>)

## **Batch Testwork**

When planning a batch test work program, there are some well tested guiding principles:

- Batch tests for uranium recovery can be conducted either in stirred tank or percolation columns.
- Batch tests are employed to optimize the process conditions of;
  - Temperature,
  - o Reagent concentration,
  - Grind size,
  - o Eh,
  - SG (Percent solids in case of tank leach or application rate for columns).
- Batch tests (and not pilot) are employed to establish the kinetics in dynamic leach processes. Pilot continuous with tracer testing is employed to corroborate the batch test outcomes.
- Batch tests initially focus on the Leach Step but are often extended to other building blocks such as thickening, filtration, solvent extraction, ion exchange etc.
- The common lixiviants in the case of uranium ores and concentrates are sulfuric acid and a sodium carbonate / sodium bicarbonate blend.
- Acid lixiviants are employed for both primary and secondary uranium ores where gangue acid consumption is acceptable.
- Alkaline lixiviants are employed on secondary ores and then only when gangue reagent consumers are at acceptably low levels.
- In some cases and where uranium mineral liberation permits a coarse grind in the leach step can be economically attractive particularly where gangue reagent consumption may be prohibitive at normal leach grind size.





Figure 13: Leach Tank for Coarse Grind Ore P80 = 1.5mm at 68% solids SG <sup>(11)</sup>

Critical to all batch leach testwork are the following objectives:

- maximise economic overall uranium recovery,
- minimise reagent input,
- consider only a sustainable process flowsheet and employ a process that delivers minimal harm to the environment (consider dry solid waste disposal).

Give consideration that the above objectives may only be achievable by processing an "accept" fraction from an upgrade process in order to minimize harm to the environment.

#### **Open and Closed Circuit Testing**

Batch leach testwork normally begins in open circuit with local fresh water employed in the make-up fluid.

Uranium recovery flowsheets are rarely one-pass open circuit and at some stage, it will be prudent to migrate to closed circuit testing.

In column leaches, it is customary after the basic leach parameters have been determined, to reconfirm the process conditions in a closed circuit with solvent extraction or ion exchange being employed to "close" the flowsheet. Refer to Figure 14.



Figure 14: Example of a Closed Circuit Column Leach Flowsheet with Solvent Extraction



As the solute levels in the lixiviant build up in a closed circuit, recoveries may decline.

In the Lethlakane project, A-Cap Resources Limited discovered that high solute levels of 350 g/L did not materially impact the recovery of uranium in column leaches (refer to Figure 15).



# Figure 15: Column Leach Uranium Extraction and Acid Consumption Comparisons Between Lixiviant Makeup in Water and Mature Solution (Courtesy of A-Cap Resources Limited) <sup>(11)</sup>

## Standard Batch Leach Tests

A Standard Batch Leach Procedure (SBLP) coupled to an economic model is invaluable for ranking competing ores.

The SBLP is normally developed from extensive leach trials and the geomet model.

Additionally, the SBLP would normally employs a synthetic lixiviant the composition of which is determined by the closed circuit mass balance.

## **Batch Lock - Cycle Testing**

Batch lock-cycle leaches are sometimes employed in the form of intermittent bottle rolls and batch leach tests to establish the impact of recycles.

## PLANNING BATCH VARIABILITY TESTWORK

Bulk ores are normally subjected to the standard batch leach procedures and the outcomes of that work applied to the geomet model.

However, where variability is significant and where the mine plan suggests that the variability cannot be "blended out", it may then be prudent to consider these variability ores not only in batch leach testing but also for inclusion into a pilot plant campaign.

Table 1 shows that the Letlhakane project has at least two significant ores in addition to the dominant primary ores. The variability ores have varying levels of organic carbon compared to the primary ores. These two variability ore types, namely mixed oxide and mudstone, have been tested on a batch basis in open and closed circuit columns.



Analysis	Unit	Kraken Primary	Gorgon Primary	Mixed Oxide	Shallow Mudstone
AI	%	9.22	10.01	8.38	13.3
Fe	%	0.68	1.15	2.72	0.99
K	%	0.50	0.41	0.00	0.54
Mg	%	0.11	0.12	0.10	0.42
S	%	0.13	0.68	0.2	0.05
Si	%	25.8	23.3	-	25.4
U	ppm	202	198	182	136
V	ppm	329	522	234	138
Total C	%	5.48	11.0	2.54	0.77
C Org	%	4.34	9.59	2.43	0.38
CO <sub>3</sub> _C	%	1.14	1.41	0.11	0.39
Acid Neut, Capacity	ka H <sub>2</sub> SO <sub>4</sub> /t	17	7	NA	NA

## Table 1 : LetIhakane Uranium Project Primary and Variability Ores<sup>(12)</sup> (Courtesy of A-Cap Resources)

The uranium in, for example, copper iron sulfide as derived from the Gawler Craton region in South Australia, reports to both the primary and secondary sulfides.

As can be seen from Figure 10, for the secondary sulfide, the uranium is locked in the iron oxides hosting the ore whilst some is surficial on the copper-iron-sulfide.

For the primary chalcopyrite rich concentrates, the uranium mineralization is both surficial and locked in the sulfides.

Because the uranium deportment was so different in the primary and secondary concentrates, batch testing was not considered to be adequate for the secondary concentrates and so piloting was embarked on to confirm sustainable removal of uranium<sup>(13).</sup>

The process employed a blend of metathesis and hydrothermal mechanisms in which the concentrate was subjected to a pressure leach in the presence of low concentrations of aqueous copper.

The reason for employing a copper leach was to alter the copper-iron-sulfides concentrate sufficiently to release uranium (and its associated daughters) whilst producing a super-concentrate with low levels (< 0.2 Bq/g) of uranium and enriched levels of copper.

Figure 16 shows the fractured mineral structure of the dominant concentrate created when iron from chalcopyrite (CuFeS<sub>2</sub>) leaves the mineral particles.

It is this fracturing that is thought to create pathways for uranium to escape and allow the upgrade process to take place.







**Figure 16: Chalcopyrite Alteration under Metathetic and Hydrothermal Conditions at > 200°C** The kinetic relation for this process is given in Figure 17.



Figure 17: Concentrate Leach Kinetics

The mineral alteration is shown in Figure 18.





Figure 18: Dominant Concentrate Leach Feed and Product<sup>(13)</sup>

In the case of the Secondary Concentrate, the removal of uranium was less complete and kinetically impaired probably an account of the slow transformation of the iron oxides under weak acid conditions in the autoclave (see Figure 19).



Figure 19: Variability Concentrate Leach Feed and Product<sup>(13)</sup>

## PRE-PILOT INTERIM PROJECT COSTING

Well scoped closed circuit batch testwork generally provides sufficient data from which to develop interim project economics for the process plant. Interim Project Costing (IPC) is extremely valuable for guiding process flowsheet finalisation and is recommended as a pre-pilot decision making tool.

IPC is possibly best undertaken at a Scoping Study Level when the batch testwork has achieved an acceptable degree of completion.

The refinery circuit of a uranium flowsheet is reasonably well understood as far as risks and uncertainties are concerned. The important process costs are in:

• ore preparation,



- leaching,
- the leach "closing step" of solvent extraction and ion exchange, and most importantly,
- the tailings facility.

The outcomes of the study, both Capex and Opex, as well as projected revenue flows, can identify whether the project is ready to commit to a pilot campaign.

## STUDY COMPONENTS AND SKILLS

#### Mass and Energy Balance

The important inputs to the mass balance are:

- Mine plan the dominant and variability ore types,
- Quantitative mineralogy of these ore types,
- Quantitative mineralogy of the leach residues,
- Solid-liquid separation data relevant to the residues derived from the ore types,
- Inputs to the important process areas of ore preparation, leaching, leach closure and tailings disposal.

#### Preliminary Engineering

Preliminary engineering normally comprises:

- Process Flow Diagrams (these are also imported into the model),
- Process Design Basis,
- Mechanical Equipment listing with electrical power determined, and
- Simple plot plan of the plant.

#### **Capital and Operating Cost Estimate**

The study will provide an estimate of the process plant capital and operating costs to a level of accuracy as determined by the engineers.

Excluded from the estimate will ordinarily be:

- Pre-project expenses,
- Infrastructure,
- Mining,
- Closure.

The operating costs draw on the usual inputs of:

- Labour,
- Energy,



- Maintenance spares,
- Reagents and consumables,
- Residue disposal,
- Cost of sales.

## **Third Party Review**

A third party review of the batch testwork and process economics is recommended prior to committing to a pilot campaign.

All the key building blocks of the flowsheet should be scrutinised using the IPC outcomes and opportunities to minimise costs should be interrogated. This may lead to a further iteration in the Batch Testwork program before the project is ready for continuous pilot plant testing.

## SCOPING THE PILOT PLANT

The purpose of a pilot plant is to deliver data required for engineering the project's next phase. The production of uranium oxide concentrates (UOC) maybe a secondary objective.

## **Pilot Plant Service Provider (Laboratory)**

It is important for the Client's team/ Consulting Engineer to provide a scope of work for the pilot plant. The scope of work is then issued to competent Laboratories to tender for the work.

In most cases, the Pilot Plant Laboratories do not know the test flowsheet as well as the Client's team does. As a result, the Laboratory needs to be provided with a scope of work that adequately address the pilot flowsheet so that it can assemble the pilot plant and also understand clearly what data is required. Laboratories are unable to guess the unwritten needs that the Client's team requires in an under-scoped program of work. This could lead to conflict and cost overruns at the expense of good data delivery.

Laboratories will also need to assemble a team of process, analytical and mineralogy specialist to deliver the pilot plant.

Process Engineering has moved on from the days when Laboratories were asked to provide a proposal without a written comprehensive work scope.

#### Scope of Work - General

The scope of work should address at least the following:

- Flowsheet to be tested
  - Process flow diagrams for all building blocks,
  - Process conditions for each building block,
  - De-scoped mass balance to pilot plant nominated flows to allow for equipment sizing and costing of equipment set up.

Figure 20 provides a sample flowsheet for a column leach campaign as part of an integrated pilot campaign.





Figure 20: Example of Testwork Flowsheet

• Environmental Health and Safety

Uranium is invariably accompanied by some of its daughters (refer to Figure 21 for the <sup>238</sup>U decay chain).



Figure 21: Uranium 238 Decay Chain<sup>(14)</sup>

In the case of primary uranium, there is normally a secular equilibrium between <sup>238</sup>U and its daughters. This equilibrium can be disturbed in the case of flotation concentrates where the activities of <sup>210</sup>Pb and <sup>210</sup>Po can often exceed that of the parent (refer to Table 2)

#### Table 2 : Radionuclide Assay of a High Uranium Copper Concentrate<sup>(15)</sup>



	D	NA		Gamma	Radiochemistry		
	U-238		Th-230	Ra-226	Pb-210	Po-210	
	ppm	Bq/g	Bq/g	Bq/g	Bq/g	Bq/g	
Drum 5 Composite	1179	14.6	14.8	14.1	17.9	18.6	

Secondary uranium ores are generally derived from uranium that has leached from exposed granites for example and has moved as an aqueous front until it was attenuated in a location remote from its source. Consequently the equilibrium has been disturbed and the long lived daughter products are sometimes not present to any significant extent.

Laboratories need to be made aware of these alterations so that they can establish their Environmental Health and Safety plan for operating the pilot plant.

• Feed to Pilot Plant

The Client is responsible for providing the dominant (and any variability) ores to the pilot plant. These are delivered to the Laboratory under a chain-of-custody. The ores tested normally represent the first 5 to 8 years of the project life.

• Pilot Periods to be Negotiated

The pilot plant periods need to be agreed with the Laboratory. Included in this are:

- Commissioning and ramp up a reliability determining period,
- Operating Periods these could span one or more weeks. The duration will be dependent on the time to establish steady state. Any testing of variability ores could extend the operating period,
- Breaks for rest and relaxation.
- Pilot Plant exclusions

The Laboratory should be requested to provide any exclusions whether called up or otherwise in the Scope of Work.

This assists the Client's team to understand what contingency may be needed in the budget. Exclusion could include:

- o Return of ripios and waste to country of origin,
- o Radio-assay etc.

#### **Pilot Plant Deliverables**

These include:

- Batch confirmatory testwork (small column or batch tank leach tests) may be required to confirm the leach parameters. This is particularly the case if the pilot feed materials do not derive from an early batch testwork campaign,
- Synthetic lixiviants and water (site and process). Water may need to be calcium saturated to simulate plant process water,
- Vendor Tests:
  - Comminution testing,
  - In-line assay amenability,
  - Thickening,
  - Rheology,
  - Filtration,
  - Geochemistry tests,



- Geotechnical tests.
- Analytical:
  - Sample preparation methods,
  - Analytical precision and tolerances,
  - Matrix matched standards and frequency of standards testing,
  - External laboratory cross checks.
- Nominated deliverables:
  - Control assay including priority control assays,
  - Profiles and how they are to be presented,
  - Mass balance,
  - Daily metallurgical report,
  - Particle size determination,
  - Absolute density of solids,
  - Bulk density,
  - Transportable moisture levels,
  - Corrosion coupon tests for (see example in Table 3 below):
    - Alloys,
    - Coatings,
    - Glass (in cases where fluorides are present).
  - Scale mapping,
  - Mineralogy,

- Retention time testing,
- Gas analysis,
- Radio assays,
- Geochemical tests (e.g. Toxicity characteristic leaching procedure (TCLP) tests),
- Geotechnical test,
- ICP multi-element scans.
- Daily meeting and reports,
- o Decommissioning and waste disposal for the laboratory to include in their pricing,

• Pilot Plant Testwork Report – normally, with an agreed table of contents, Table 3 : List of Corrosion Coupon Specified for Testing in a Pilot Campaign

			Materials of Construction											
Location	Position	Ti Gr 2	Ti Gr 17	SAF 2507	SAF 2205	D411-350	D470-300	D441- 400	VE-8300	VE-8730	VE-8360	UHMWPE	HDPE	GI ASS
	Vapour			Х										
NO. I Leach Feed	Slurry	Х				Х	Х	Х	Х	Х	Х	Х	Х	
No. 2 Leach Feed	Slurry					Х	Х	Х	Х	Х	Х	Х	Х	
Reagent Trim Tank	Aqueous					Х	Х	Х	Х	Х	Х	Х	Х	
Leach Tank 2	Vapour	Х	Х											
	Slurry	Х	Х											
Leach Tank 5	Vapour													Х
No. 2 Barren Liquor Neutralisation Tank	Slurry			Х	х	х	Х	х	х	х	х	х	х	

## **Adjudication of Proposals**



Proposals are often requested from multiple Laboratories.

Alignment meetings with the Laboratories are recommended to ensure expectations on both sides are understood.

Consider adjudicating conforming proposals and then examine non-conforming bids on their merits.

The team selected for the adjudication process could comprise Consultants, Clients and Engineers. Generally allow 2 to 4 weeks for the proposal and alignment period.

As the scope of pilot plant may change during the testing period, a contingency commensurate with any envisaged scope growth is recommended.

## MANAGING THE PILOT PLANT

The Pilot Plant activities need to be carefully managed to ensure:

- the objectives are achieved, and
- quality data produced is suitable for the next level of engineering.

#### **Client Project Manager**

The Client's Project Manager provides:

- technical oversight,
- reviews data and reports to the Laboratory Project Manager in a timely fashion,
- requests trouble shooting and reviews outcomes of investigations,
- ensures that data stipulated in the Scope of Work is being attended to.

#### Laboratory Project Manager

The Laboratory Project manager:

- provides operational management,
- manages the data flow according to Figure 22,
- leads the daily meetings at which the following data is presented and reviewed;
  - o Daily Metallurgical Report and Mass Balance,
  - Control Assays,
  - Profile Assays,
  - Water Balance,
  - o Trouble Shooting Outcomes,
  - Go-Forward Plans,
  - Any Vendor Activities and Special Samples.





#### Figure 22: Data Delivery Structure

#### Water Balance

The water balance of a Pilot Plant can be delicate and is influenced by:

- Evaporation losses from elevated temperature steps,
- Sample abstraction,
- Sample return net of that required by sample preparation,
- Vendor samples,
- Spills and losses.

The expected sample load can be factored into the starting aqueous inventory so that only water top-up for evaporation is considered during the pilot campaign. The downside of this is the higher overall circuit retention time and the longer time to achieve steady state. See



Table 4 as a simple example of how a Pilot Plant retention time is determined.



Equipment	No. of Tanks	Retention time per Tank (h)	Total Retention Time (h)	
Leach Feed Makeup Tanks	2 (alternates)	3	3	
Leach Tanks	5	3	15	
Filter Feed Tank	1	2	4	
PLS Tanks	1	4	4	
Ion Exchange Circuits			4	
Loaded Eluate Tank	1	2	2	
Solvent Extraction Feed Tank	1	2	2	
Solvent Extraction Mixer -Settlers	4E + 1 A/S, 3 Sc, 3S, 1W	0.5	6	
Solvent Extraction Loaded Strip Tank	1	2	2	
Sodium Diuranate Precipitation Tanks	4	1.25	15*	
Sodium Diuranate Precipitation Thickener	1	5	5	
SUBTOTAL (Leach, IX, SX, SDU)			62	
SDU Resolution Tanks	2	2	4	
Uranium Oxide Precipitation Tanks	4	1	12*	
Uranium Oxide Precipitation Thickener	1	5	5	
TOTAL			83	

#### Table 4 : Example of Pilot Plant Retention Time (refer Figure 23)

\* Seed Recycle allowed for

Alternatively, a lower solution starting inventory could be considered; however, in this option, a solute and solvent (water) makeup will be required during the pilot campaign.



Figure 23: Example of Acid Tank Leach Flowsheet

## **Metal Accounting**

Metal accounting across a pilot plant is vital to demonstrate the expected value metal recoveries. Generally, any uranium unaccounted cannot be assessed to be recoverable.

A typical metal accounting log across the flowsheet in Figure 23 is highlighted in



Table 5.



## Table 5 : Typical Met Accounting Proforma

Parameters	Wet Mass (kg)	Dry Mass (kg)	Volume (L)	U Assay (ppm)	U Mass (g)
INPUTS					
Ore / Concentrate					
First Fill					
TOTAL INPUT					
OUTPUTS					
MEASURED:					
UOC / SDU					
Samples - Assay					
- Vendor					
- Stored Off					
I alls - Residue					
Spills					
Measured Out					
CALCULATED:					
Open Stock (Tanks, Ponds, IX, SX)					
Closing Stock (Tanks, Ponds, IX, SX)					
Calculated Change					
GRAND TOTAL OUTPUT					
UNACCOUNTED					

## WHEN TO CONSIDER DEMONSTRATION SCALE

Pilot plant data generated in a steady state conditions should provide;

- confirmation of the metallurgy and mineralogy of the process,
- the solid-liquid separation fluxes and related rheology,
- an early understanding of the deportment of scale, and
- the adequacy of any materials of construction that may have been tested.

For novel processes without an adequate operating reference plant or where there is uncertainty in the long term suitability of materials and/or equipment selection, there could be merit to consider an extended Demonstration Plant campaign.



Table 6 provides a high level comparison between Pilot and Demonstration Plants.



Pilot Plant	Demonstration Plant				
<ul> <li>Small equipment – laboratory bench top,</li> </ul>	<ul> <li>Longer running time (4 to 5 months minimum),</li> </ul>				
• Provides retention time data,	• Requires larger quantities of feed,				
Provides steady state assay data,	<ul> <li>Permits longer term testing of materials of construction,</li> </ul>				
<ul> <li>Provides steady state solid-liquid separation information,</li> </ul>	• Employs real mechanical equipment,				
• Preliminary data on materials suitability,	<ul> <li>Long term understanding on build-up of minor elements in closed circuit</li> </ul>				
• Short running time (2 – 6 weeks),	flowsheet,				
<ul> <li>Limited or no data on adequacy of mechanical equipment,</li> </ul>	<ul> <li>Generally considered for novel or established processes with one or more novel steps,</li> </ul>				
• Cost range A\$1 - 6 M (rental basis).	<ul> <li>Costs can be A\$20-50 M or more (rental basis).</li> </ul>				

#### Table 6 : Comparison between Pilot and Demonstration Plants

#### **Risk Mitigation**

Pilot and demonstration plant testing provides and additional level of risk mitigation over batch testing that will guide flowsheet development. Some are however prepared to design the commercial plant from Batch testwork. However, as shown by McNulty, inadequate testing may lead to long ramp uptime and the risk of not achieving the design throughput.

Demonstration scale testing provides additional risk reduction over what can be expected in a well scoped and managed pilot plant. Figure 24 and Figure 25 show typical pilot and demonstration plant filters. The filter presses employed in a uranium removal demonstration campaign (Figure 25) involved the use of inflatable membrane chambers. This filter press was fitted with commercial cloths and the cakes were washed with process fluids and finally squeezed and air blown.



Figure 24: Pilot Plant Pot Filters

Figure 25: Demonstration Plant Filter

In the extended running time of the demonstration plant, the long term adequacy of filter fabrics and the need for a cloth wash program was identified – thus extending the expected filter cycle time.

The longer running time of a demonstration plant permits additional data gathering. Figure 26 shows a Class 600 Titanium Grade 2 ball valve that failed two months into a demonstration plant campaign. A Scanning Electron Microscopy (SEM) study on the corroded ball confirmed crevice corrosion as responsible for the loss of metal.





Ball Valve Body Leakage

Valve Ball displaying Crevice Corrosion

## Figure 26: Ball Valve Leakage and Ball Valve Crevice Corrosion

Figure 27 shows scale development on a Demonstration Plant leach reactor shaft. Mapping the scale growth rate assists in identifying the duration of an operating cycle in the commercial plant.



Figure 27: Metallurgical Scale on a Leach Vessel Agitator Shaft and Impeller

## CONCLUSIONS

Pre-Pilot:

- Relevant ores / concentrates to be tested.
- Comprehensive Batch Dominant ore and Variability ore tests to be conducted.
- Testwork to actively embrace the final residue disposal method and any recycles therewith.
- Mineralogy of Feeds and Residues are critical to modelling the flowsheet.



• Consider upgrade and therewith minimise environmental footprint (be good stewards of what has been entrusted to us).

• Use Interim Project Costing as a tool to optimise the flowsheet. Pilot Plant:

- Comprehensive pilot plant scope of work document aligns the Laboratory with Client's expectations and requirements.
- Pre-pilot batch and Pilot plant testwork thoroughness has been shown to impact the Commercial plant ramp-up and NPV.
- Pilot plants have a limited suite of deliverable restricted by short running times and frequent use of non-mechanical equipment.
- Pilot plant may not offer sufficient risk mitigation. A demonstration plant may be appropriate where the process is novel.
- A demonstration plant could cost at least 10 times more than a pilot plant and run for several months.

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