

URANIUM RECOVERY BY CONTINUOUS ION EXCHANGE OF ALKALINE LEACHATE

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ABSTRACT

One of the challenges of the alkaline leach process is to deliver a high concentration eluate of purity to the refinery. Ion exchange often plays a role in this process. This paper describes a sequence of flowsheet improvements to an ion exchange circuit to achieve this objective.

Some testwork data is provided to describe the journey taken in improving the CIX circuit to deliver the required eluate composition. The linkages between the refinery and the ion exchange circuit are described as well as the typical stream purities and composition of final oxide product.

Modelling of a continuous ion exchange process allows the behaviour of the process to be investigated, and the process to be quantified. An example is included to demonstrate some of the key factors that should be included when modelling ion exchange processes.

1. INTRODUCTION

Australia has the largest known recoverable uranium resources in the world. With recent increase in the demand of uranium, there is a growing interest in the processing of more challenging secondary uranium ore such as surficial calcrete deposits. A significant amount of Australia's uranium deposit (4.9%) is surficial (calcrete) uranium deposit (McKay, A.D. and Mieztis Y., 2001), such deposits are commonly extracted via the alkaline leach method.

Surficial uranium deposits usually have secondary cementing minerals such as calcite, gypsum, dolomite, ferric oxide and halite (McKay, A.D. and Mieztis Y., 2001). However, uranium deposits in calcrete are the largest of the surficial deposits. The calcrete deposits are interbedded with tertiary sand and clay, which are usually cemented by calcium and magnesium carbonates (McKay, A.D. and Mieztis Y., 2001).

Yeelirrie Mine in Western Australia is the world's largest surficial deposit with 52,500t of U_3O_8 in resources at an average grade of 0.15% U_3O_8 (McKay, A.D. and Mieztis Y., 2001). Other significant surficial deposits in Western Australia include Lake Way, Centipede, Thatcher Soak and Lake Maitland (McKay, A.D. and Mieztis Y., 2001).

2. TYPICAL ALKALINE URANIUM LEACH FLOWSHEET

There are a number of different methods of leaching uranium depending on the ore type, type and contribution of gangue minerals and impurities. Acid and alkaline uranium leaching are the most common employed today for the recovery of uranium. Generally, the acid leach process has faster kinetics with higher recovery and can be done at lower temperature compared to the alkaline leach processes.

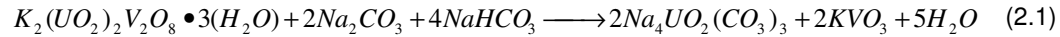
However, with ore bodies containing significant acid consuming gangue materials, in particular calcite, alkaline leach process can be a more cost effective option in this scenario. A general guide has been that if the ore contains more than 12% of carbonates, then alkaline leaching often is more economical than acid leach (Yan D. and Connelly D., 2008). Although, other considerations such as efficiency of uranium extraction, reagents cost, water and energy consumption, product quality and environmental impact must also be considered (Uranium Extraction Technology, 1993).

The alkali leach is generally more selective than acid leach and often reduces the impurity load that has to be removed in the downstream processes. However, the alkali leach has slower kinetics, which requires the process to be carried out at elevated temperature of 75-120°C and at around a pH 10-11. Sodium carbonate solution (30g/L) with small quantity of sodium bicarbonate is generally employed in the alkali leach process.

A typical alkaline uranium leach flowsheet for calcrete hosted uranium is as shown in Figure 1. The uranium in the ore body can be concentrated in the clay. Crushed ore is scrubbed in re-circulated CCD overflow sodium carbonate solution (30g/L Na_2CO_3) to remove the clay from any sand and

some uranium is leached in the scrubber. The scrubber product is then screened, or cycloned, in the stage desliming process and the oversize fraction (scats) is finally washed with clean water before rejected as a barren. The screen underflow, or cyclone overflow, is thickened and the overflow then reports to the continuous ion exchange (CIX) circuit. The concentration of uranium in the overflow solution in parts per million is nominally similar to the uranium concentration in the ore in grams per tonne.

The thickener underflow is leached at approximately 85°C. Heat recovery from the leach discharge is introduced into the advancing leach slurry. Uranium occurs in nature in either hexavalent or tetravalence form (Uranium Extraction Technology,1993). Secondary uranium ores generally contain oxidised uranium, and the alkaline leach reaction is as described in Equation 2.1.



The leached slurry enters a washing stage using a series of counter current decantation (CCD) thickeners to recover the leached uranium. The washing efficiency of uranium can be improved by recovering uranium from a bleed stream from a CCD thickener overflow and recovering the uranium in the ion exchange circuit before returning it as wash liquor lower down in the CCD train.

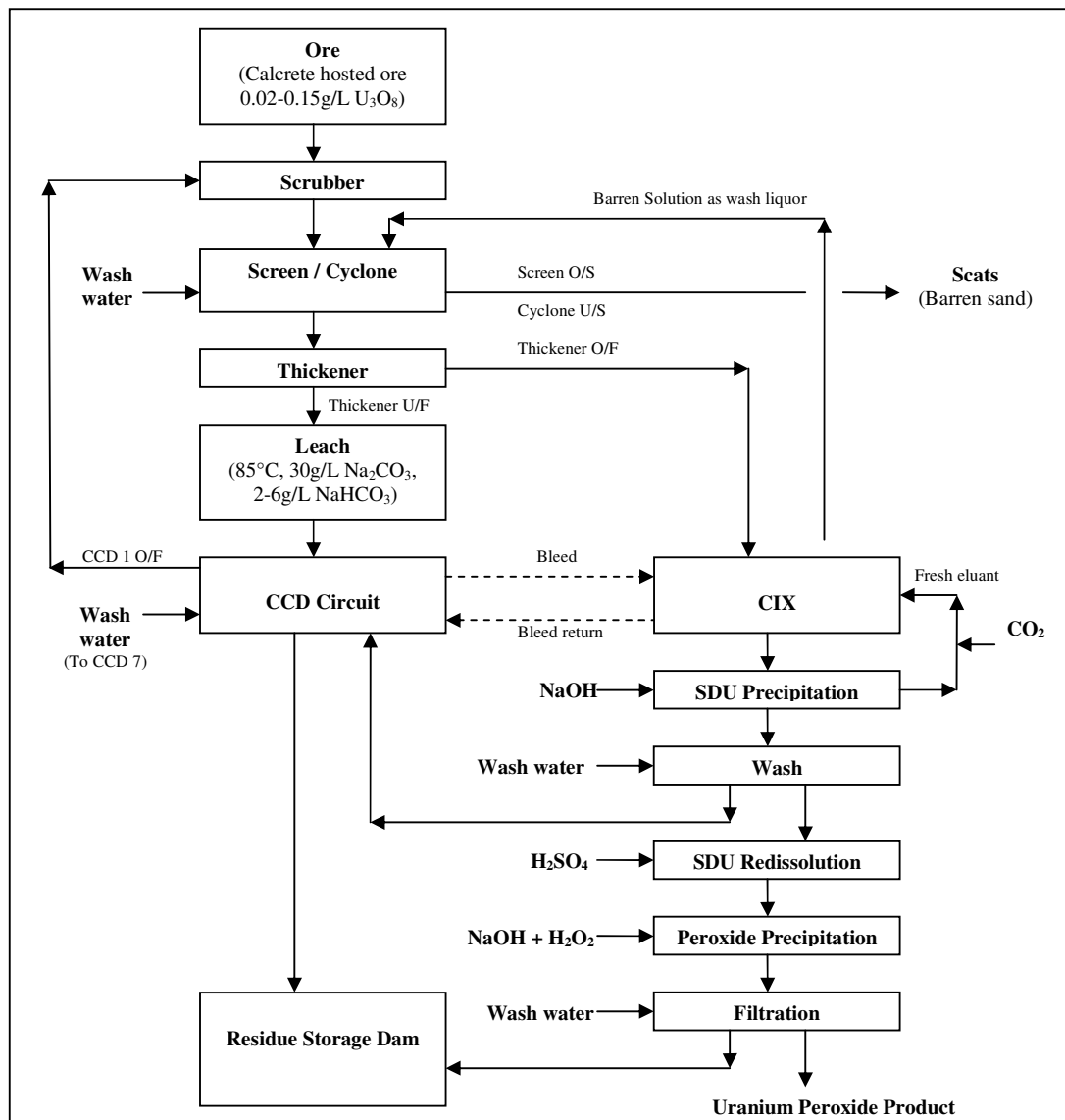
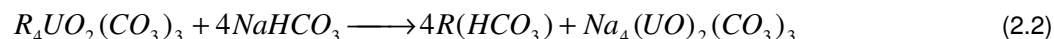


Figure 1: Typical alkaline uranium leach flowsheet

Uranium is loaded on the ion exchange resin and barren liquor is produced containing as little as 2mg/L U. Certain anionic impurities e.g. vanadium, phosphate, sulphate, chloride can also be

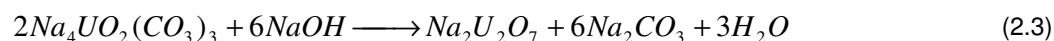
extracted along with uranium. Some of these impurities on the resin can be scrubbed prior to elution.

Sodium bicarbonate is often the reagent of choice in the elution step.

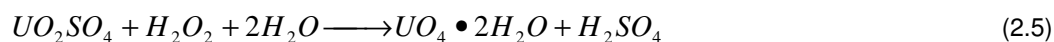
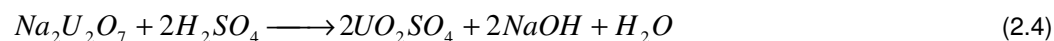


The refining process for uranium normally employs a sodium diuranate precipitation step followed by a sulfation resolution in sulphuric acid, and finally the precipitation of an oxide from the product with sodium hydroxide and hydrogen peroxide.

Sodium hydroxide is used to precipitate the dissolved uranium to form sodium diuranate ($Na_2U_2O_7 \cdot xH_2O$), as shown in Equation 2.3 below. The sodium hydroxide initially reacts with the bicarbonate ion in the alkaline leach solution. Then, with the presence of excess sodium hydroxide, uranium will hydrolyse and precipitate at pH12 and above (Merritt, R.C., 1971).



To increase the purity of the final product, the sodium diuranate is re-dissolved with sulphuric acid as shown in equation 2.4 and then re-precipitated with hydrogen peroxide solution in equation 2.5 as hydrogen peroxide is highly selective in the precipitation of uranium.



3. ION EXCHANGE

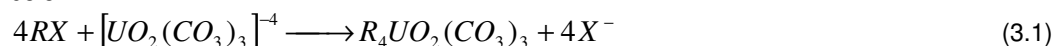
Solvent extraction or ion exchange is normally employed to concentrate and purify the uranium leachate and remove impurities. Solvent extraction and ion exchange both involve the interchange of ions between the aqueous solution and either a liquid organic solution or a solid resin.

As the pregnant liquor solution (PLS) in carbonate leaching normally has a relatively low concentration of uranium (approx 150 to 1000ppm U), an upgrading step is normally required prior to oxide recovery. In most cases, ion exchange is ideal in this duty.

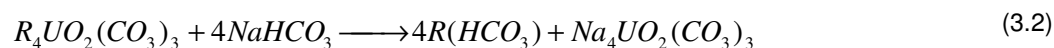
3.1 URANIUM ION EXCHANGE CHEMISTRY

In uranium ion exchange, strong base anion resins are normally used. The strong base anionic resin for uranium ion exchange contains quaternary ammonium functional group. The degree and type of cross linking varies between different manufacturers. This influences the chemical and mechanical characteristics of the resin (Merritt, R.C., 1971).

The uranium adsorption reaction that occurs in the resin can be described by the equation 3.1 below:



The adsorbed uranium can be released in the elution stage by using a strong sodium bicarbonate solution. The reaction can be described in Equation 3.2 below



3.2 FIXED BED ION EXCHANGE VERSUS CONTINUOUS ION EXCHANGE (CIX)

Traditionally, fixed bed ion exchange is used in industry. Conventional fixed bed ion exchange typically employs a few large columns with for example, 2 or 3 columns in loading phase, 1 column in scrubbing, and another in the elution phase. As the loading, scrubbing and elution cycle times are different, there is frequently a column idling for a period of time in fixed bed arrangements.

The continuous Ion Exchange (CIX) process utilises a larger number of smaller ion exchange columns (frequently more than 20 columns) which operates in a continuous manner via counter-current contacting of liquid with resin. Although having a larger number of smaller columns adds a degree of complexity in the circuit, it also introduces a flexibility that is not available in a fixed bed ion exchange circuit.

Some of the benefits of continuous ion exchange over conventional fixed bed arrangements are as follows (Rossiter, G.J., 2009):

1. Reduced Resin Charge (50-80%) reduces the capital cost of resin inventory in column and equipment size.
2. Higher concentration eluate product (by a factor of 3-4) which translates into lower volumetric flow of concentrated eluate to the refinery and oxide recovery circuits and hence lower capital cost. Having a higher purity product also helps to maintain higher recovery in the precipitation step.
3. Higher first pass uranium recovery.
4. Reduced reagents consumption (eluants), reducing plant operating cost.
5. Reduced water consumption (40-60%).
6. Maximises new water addition to the CCD washing circuit.
7. Permits the use of a pre-elution stage and /or a post adsorption scrubbing stage to remove co-loaded impurities and produce a purified eluate, which allows simplification in the downstream product recovery process.

In CIX, having a larger number of smaller columns (e.g. 5 to 10 columns in series) in the loading step allows the ion exchange resin in the leading column to be loaded to near saturation without compromising on the overall recovery. To illustrate this, an example from ion exchange batch testwork with 3 columns in series is shown in Figure 2. As demonstrated, uranium was able to be loaded to saturation in column 1, whereby the concentration of uranium into and out of the column were approximately the same. There was a significant amount of breakthrough uranium in column 2. By having an additional column 3, there is negligible uranium breakthrough when the first column is at or near saturation.

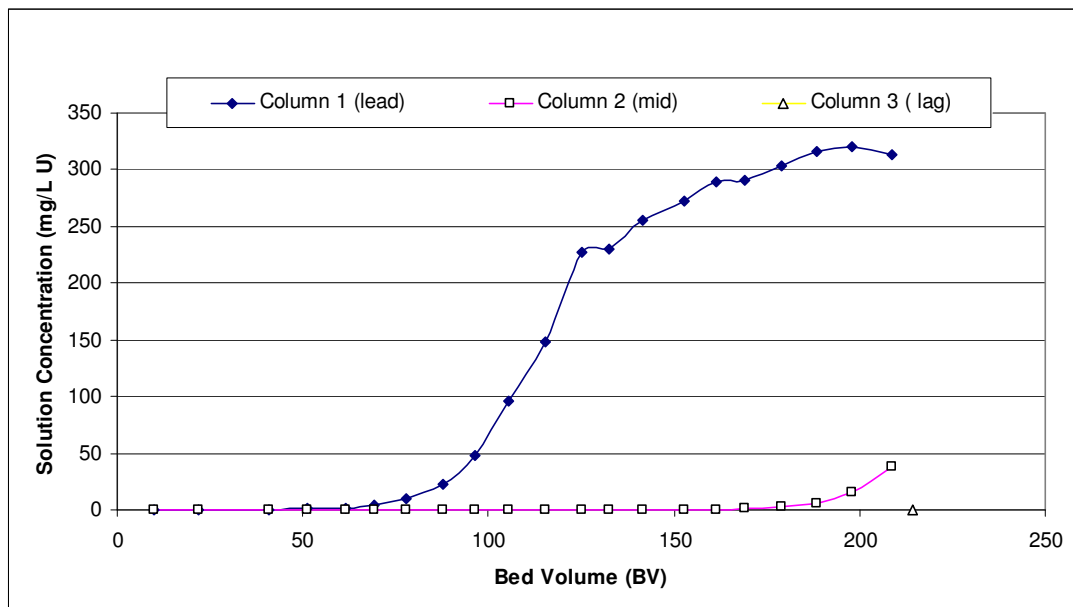


Figure 2: Typical breakthrough curve for uranium ion exchange

3.3 CONTINUOUS ION EXCHANGE CIRCUIT

Due to the flexibility of the CIX circuit, intermediate steps such as feed displacement, pre-elution and conditioning stages are able to be introduced to remove impurities and improve on the grade of the eluate. Figure 3 shows the configuration of a CIX pilot plant.

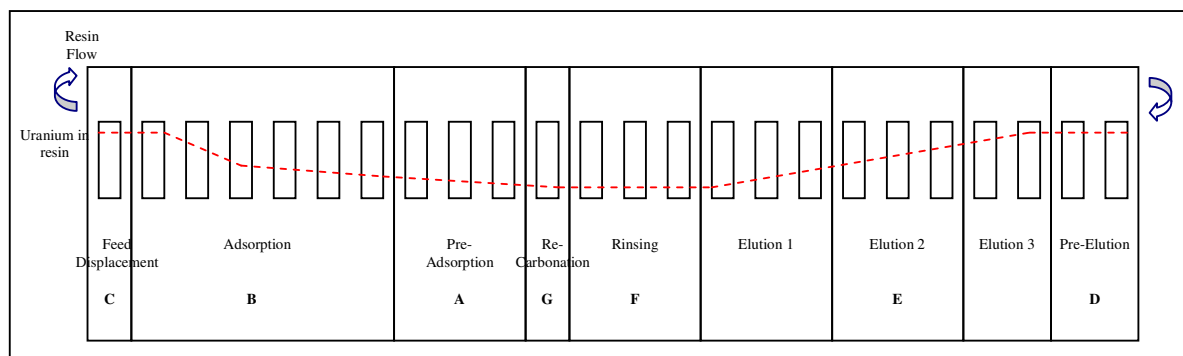


Figure 3 Continuous Ion Exchange circuit configuration

The CIX circuit configuration for alkaline uranium can for example consist of:

- A. **Pre-Adsorption Zone:** Bleed solution from the CCD circuit overflow is contacted with the freshly regenerated and rinsed resin. The uranium fed to this zone is about 10 to 15% of the total uranium feed to the CIX.
- B. **Adsorption Zone:** Solution from thickener overflow (PLS solution) is fed counter current to the resin to optimize on the resin loading.
- C. **Feed Displacement Zone:** Resin leaving the adsorption zone passes through the feed displacement zone whereby the entrained liquor is displaced from the columns with rinse solution and returned to the adsorption zone.
- D. **Pre-Elution Zone:** In the pre-elution zone, concentrated eluate is sacrificed to displace the low concentration and purity interstitial fluid in the column. The displaced solution is returned to the PLS feed tank.
- E. **The Elution Zone:** The fresh eluant flows in counter current direction to the ion exchange resin in this zone to strip the uranium from the resin. Warm sodium bicarbonate solution is used in this process. The elution zone can be divided into 3 stages.
- F. **Eluant Displacement Zone:** The column is rinsed with CIX barren solution from the adsorption stage to displace the eluant and any stripped uranium in the resin.
- G. **Re-Carbonation Zone:** The column is recirculated with barren solution to condition the column and displaces bicarbonate from the resin.

4. DEVELOPMENT OF THE CIX CIRCUIT

The findings from some batch and continuous testing will be presented showing the optimisation developments in producing uranium high grade eluate.

4.1 BATCH TESTWORK

Early batch testwork was conducted using three ion exchange columns connected in series. Strong base anion resin (Amberjet™ 4400HCO₃) was tested and used in the ion exchange columns. These columns were loaded with uranium PLS solution and then subjected to the subsequent steps of washing, scrubbing, elution and conditioning. The results from the batch testwork are shown in Figure 4. The typical PLS solution composition in batch testwork is as shown in Table 1.

Components	Typical Concentration (g/L)
U	0.2-0.4
P ₂ O ₅	0.2-0.3
SO ₄ ²⁻	0.8-1.2
Cl ⁻	0.1-0.3
Mo	0.001-0.005
Si	0.005-0.03
V ₂ O ₅	0.001-0.003

Table 1: Typical Uranium alkaline leach PLS solution

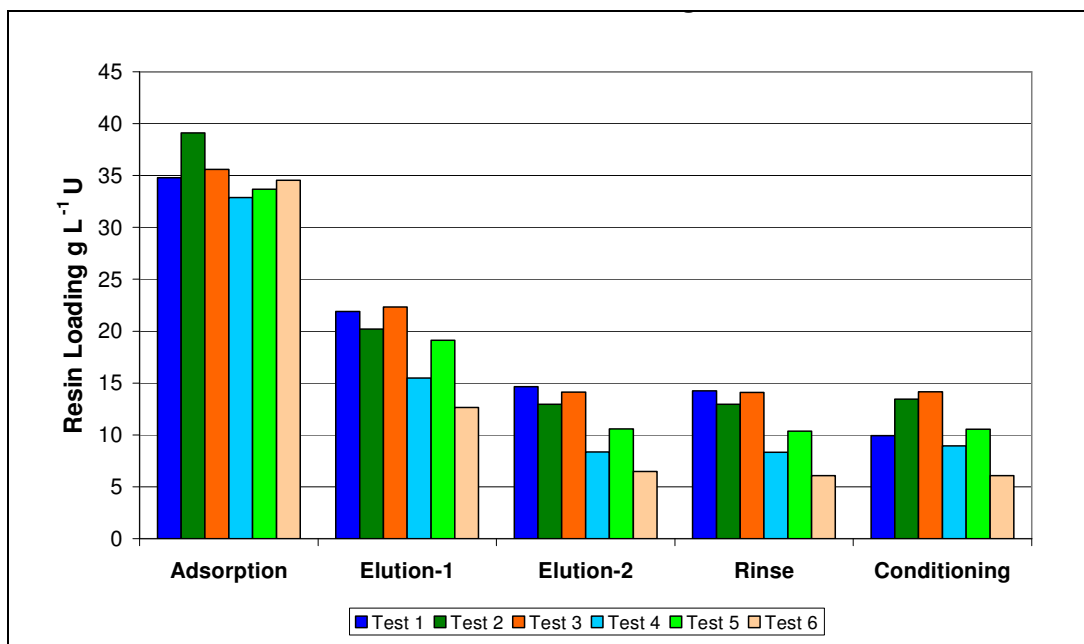


Figure 4: Batch testwork results of uranium loading and stripping

The uranium loading on the resin after adsorption was approximately 35g/L U. The early testwork showed that the uranium was not fully eluted with the stripped resin uranium concentration of 8-14g/L U. As a result of incomplete stripping, the net uranium loading capacity of the resin was between 21 to 27 g/L U.

The eluant concentration was 1 Molar sodium bicarbonate. It is customary to employ elevated temperatures (typically 40-60°C) in the elution of ion exchange resins employed in uranium duty as this was the case in this work. The earlier tests (Test 1 to 3) reflect data from a low temperature elution and the latter tests (Test 4 to 5) are for an elution performed at 40-50°C. This temperature elevation improved the uranium elution somewhat (reducing the uranium on resin by approximately 5g/L U).

4.2 PILOT TESTWORK SETUP

An IONEX (Ionex Separations, USA) multiport pilot valve of the type shown in Figure 5 was employed in the continuous testwork. The optimised batch testwork outcomes provided the foundation for the continuous circuit flowsheet. A three stage split elution circuit operated at elevated temperature of 40-60°C was employed. The eluant comprised of a blend of 1 Molar sodium bicarbonate with 0.15 Molar of sodium carbonate. Carbon dioxide was gassed through the eluant and the interstage eluates to convert eluted carbonate to bicarbonate.

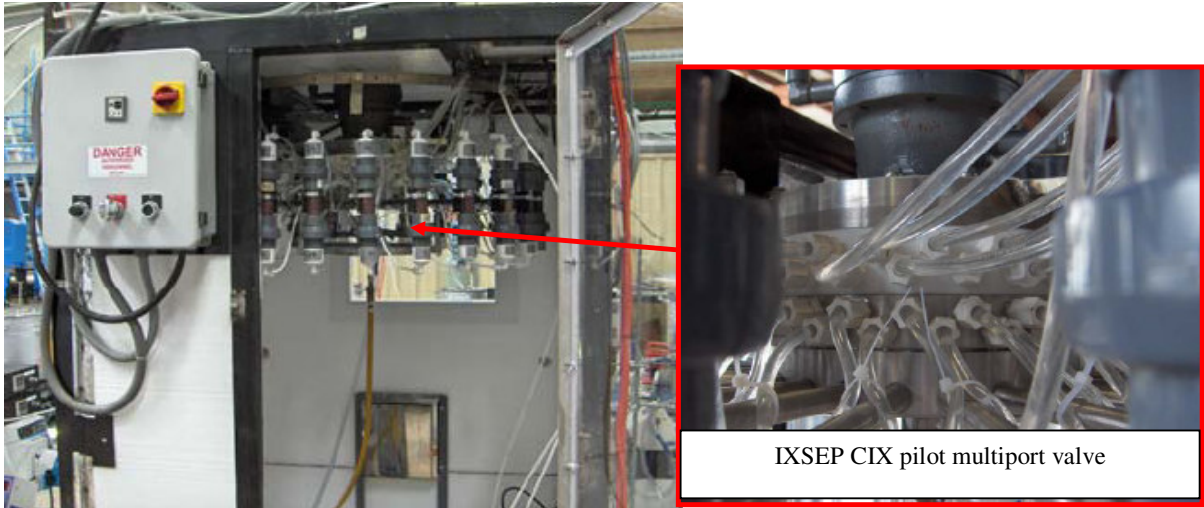


Figure 5: Pilot plant setup for the Continuous Ion Exchange (CIX) unit housing the ion exchange columns and valve head

4.2.1 Enhancing Overall Uranium Recovery – CCD Intervention

In an alkaline leach circuit, two efficiency issues present themselves to the process engineer tasked with the design of the circuit. These are:

- Uranium recovery from the leachate, and
- Minimisation of reagent loss from the leach residue solid-liquid separation step.

These have to be balanced with the bleed of unwanted solute rich in vanadate, sulphide, chloride and other impurities. Supporting the optimisation of these objectives is the minimisation of the introduction of extraneous water to the circuit other than in the residue washing stage.

A further enhancement that was explored was the introduction of a CCD uranium wash. This incorporated CCD #2 thickener overflow uranium PLS that was contacted with freshly conditioned resin and the barren from which was then returned to CCD #6. This flowsheet is shown in Figure 6. This served to improve the overall circuit uranium recovery.

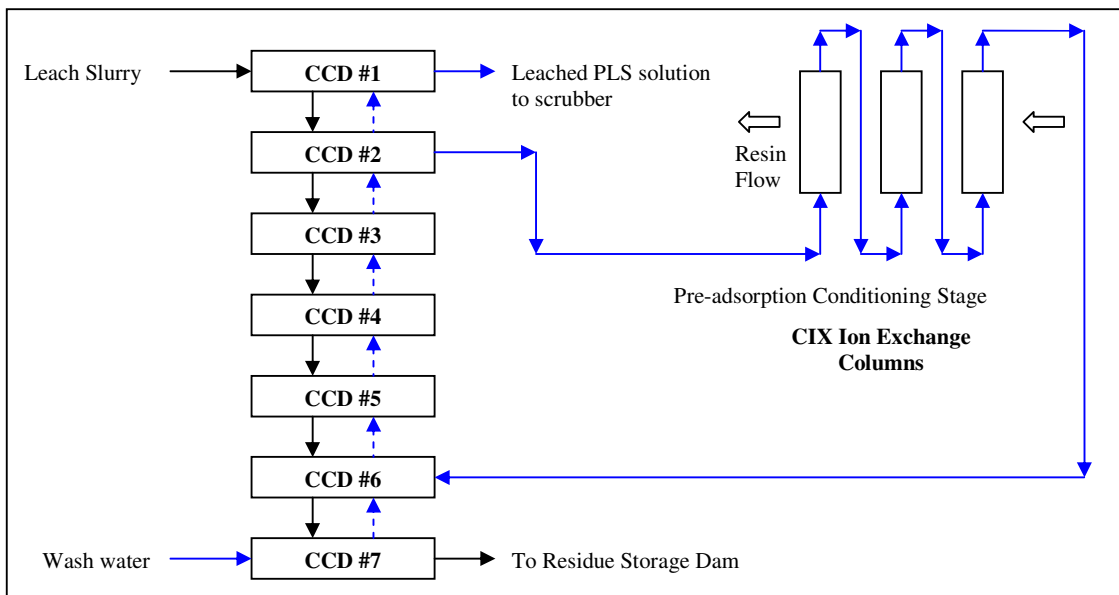


Figure 6: CCD#2 PLS solution to Ion Exchange

4.2.2 Enhancing Overall Uranium Recovery – Concentrated Eluate

In both acid and alkaline leach circuits that front up to direct precipitation of uranium from the eluate, higher concentrations of uranium in the eluate favour both first pass and overall uranium recovery. The pre and post elution components of the flowsheet in Figure 7 support this objective. The initial attempt at achieving higher uranium tenors and recoveries in the elution circuit adopted an eluant entrainment rejection step termed “rinsing” in Figure 7. The rinsate was then employed in leach PLS entrainment step termed “pre-elution” in Figure 7. The eluant (shown in equation 4.1) comprised 1 molar sodium bicarbonate solution. Interstage carbon dioxide gassing was employed to convert eluted carbonate to bicarbonate thereby restoring the interstage elution efficacy in the ensuing split elution stage.

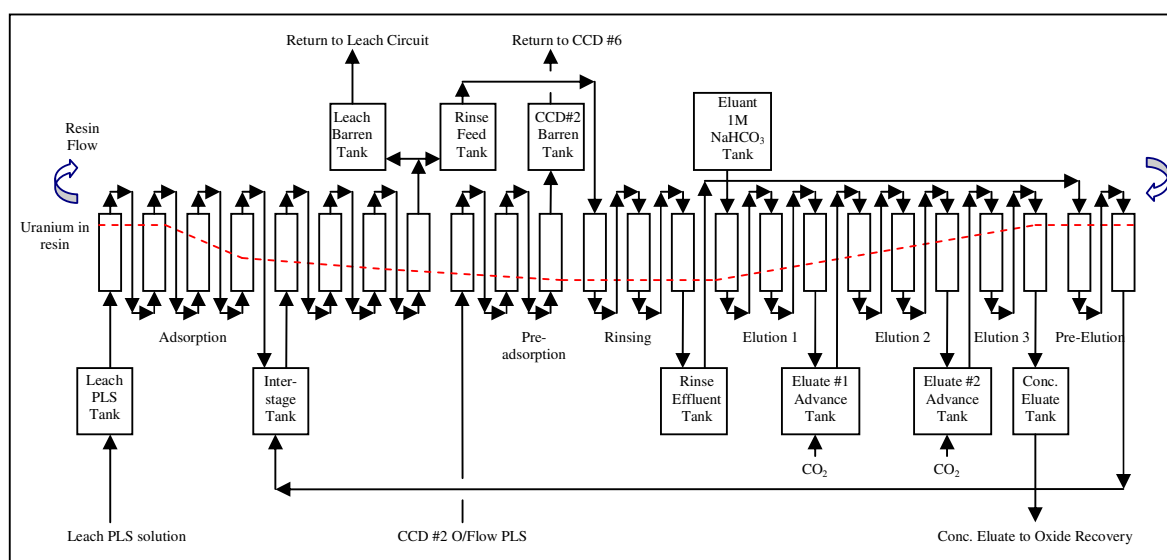
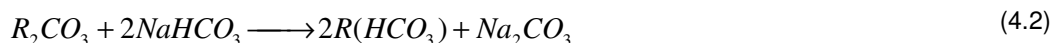
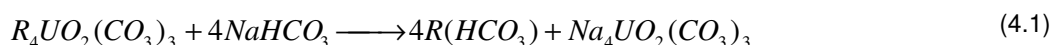


Figure 7: Initial Flowsheet

The enhancements from this initial phase of leaching are provided in Table 2 feature of which were:

1. The overall uranium recovery was low at approximately 94% from the leach PLS and approximately 90% from the CCD #2 overflow PLS
2. Net resin loading of 22g/L uranium was achieved and often exceeded.
3. Final concentrated eluate of 8.5g/L U was considerably lower than the target of 15g/L U.

Solution Assays – g/L								
Assay	U	Na	P	Mo	Si	SO ₄	Cl	pH
Leach PLS	0.234	13.020	0.082	0.000	0.018	1.593	0.027	10.5
CCD #2 PLS	0.095	11.510	0.095	0.000	0.012	1.419	0.020	10.3
Eluant	0.028	22.460	0.072	0.001	0.027	1.494	0.017	9.2
Rinse Feed	0.011	15.560	0.116	0.001	0.014	1.877	0.027	10.6
PLS Barren	0.014	13.330	0.084	0.000	0.017	1.575	0.024	10.6
CCD #2 Barren	0.011	12.120	0.098	0.000	0.012	1.135	0.071	10.1
Eluate 1	2.990	28.790	0.026	0.001	0.007	0.551	0.131	8.7
Eluate 2	13.940	28.170	0.007	0.005	0.001	2.275	0.040	8.7
Concentrated Eluate	8.370	26.420	0.058	0.000	0.004	3.392	0.131	9.4

Table 2: Typical solution assays achieved from the initial flowsheet

4.2.3 Enhancing Eluate Concentration

The rapid turnaround of profile solution assay across the continuously operated pilot plant permitted changes to the flowsheet. Several variations were considered, however, the final circuit that was derived from this cycle of testing was that shown in Figure 8.

This flowsheet incorporated:

- Leach PLS entrainment displacement after extraction employing the eluant entrainment displacement barren.
- A pre-elution step in which some concentrated eluate was employed to displace the interstitial fluid in the loaded resin column before advancing into the elution step. The barren fluid from this pre-elution step contained a small quantity of sacrificed eluate that was released to the leach PLS tank.
- An eluant concentration increased to 1.3 Molar sodium bicarbonate.
- The split interstage eluate carbon dioxide gassing was retained to convert eluted carbonate to bicarbonate, and
- A bicarbonate elution step prior to the pre-adsorption step.

The removal of several adsorption stages was required to incorporate the above changes.

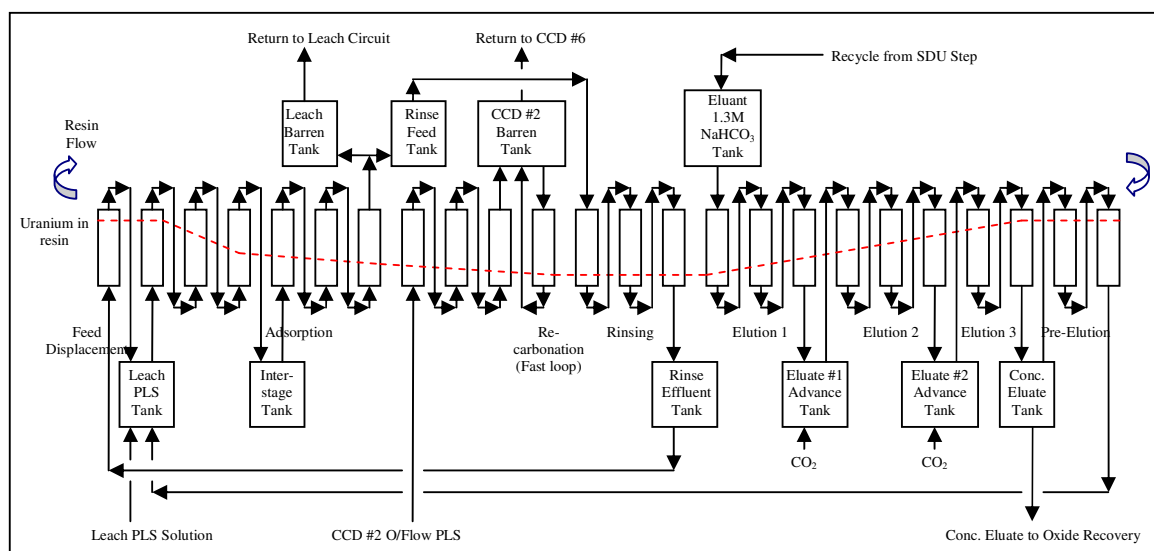


Figure 8: Final CIX Pilot Circuit Configuration

The final stages of the pilot plant yielded the results shown in Table 3. Of importance here was,

- The leach PLS uranium recovery increased a further 4% and that from the CCD#2 PLS by 7-8%.
- Concentrated eluate to oxide production had a uranium tenor of 13.8g/L U (still lower than the target of 15g/L U) but commensurate with the low PLS tenor into adsorption.
- The leach barren liquor uranium levels were reduced below 5mg/L U.

Solution Assays - g/L								
Assay	U	Na	P	Mo	Si	SO ₄	Cl	pH
Leach PLS	0.233	13.320	0.068	0.001	0.013	1.677	0.047	10.3
CCD #2	0.071	12.170	0.065	0.001	0.006	1.401	0.081	10.3
Eluant	0.238	39.900	0.000	0.015	0.000	2.524	0.145	8.7
Rinse Feed	0.002	11.430	0.076	0.001	0.015	1.287	0.061	10.3
PLS Barren	0.002	13.580	0.069	0.001	0.013	1.706	0.084	10.3
CCD #2 Barren	0.002	12.520	0.067	0.001	0.008	1.156	0.088	10.2
Eluate 1	4.281	39.030	0.050	0.006	0.017	2.371	0.229	8.7
Eluate 2	14.490	39.230	0.049	0.013	0.017	2.299	0.229	8.6
Concentrated Eluate	13.770	42.940	0.061	0.015	0.013	4.790	0.229	9.1

Table 3: Typical solution assays achieved from the final flowsheet

5. URANIUM OXIDE PRODUCT

The flowsheet that was employed in the production of the final oxide is shown in Figure 9.

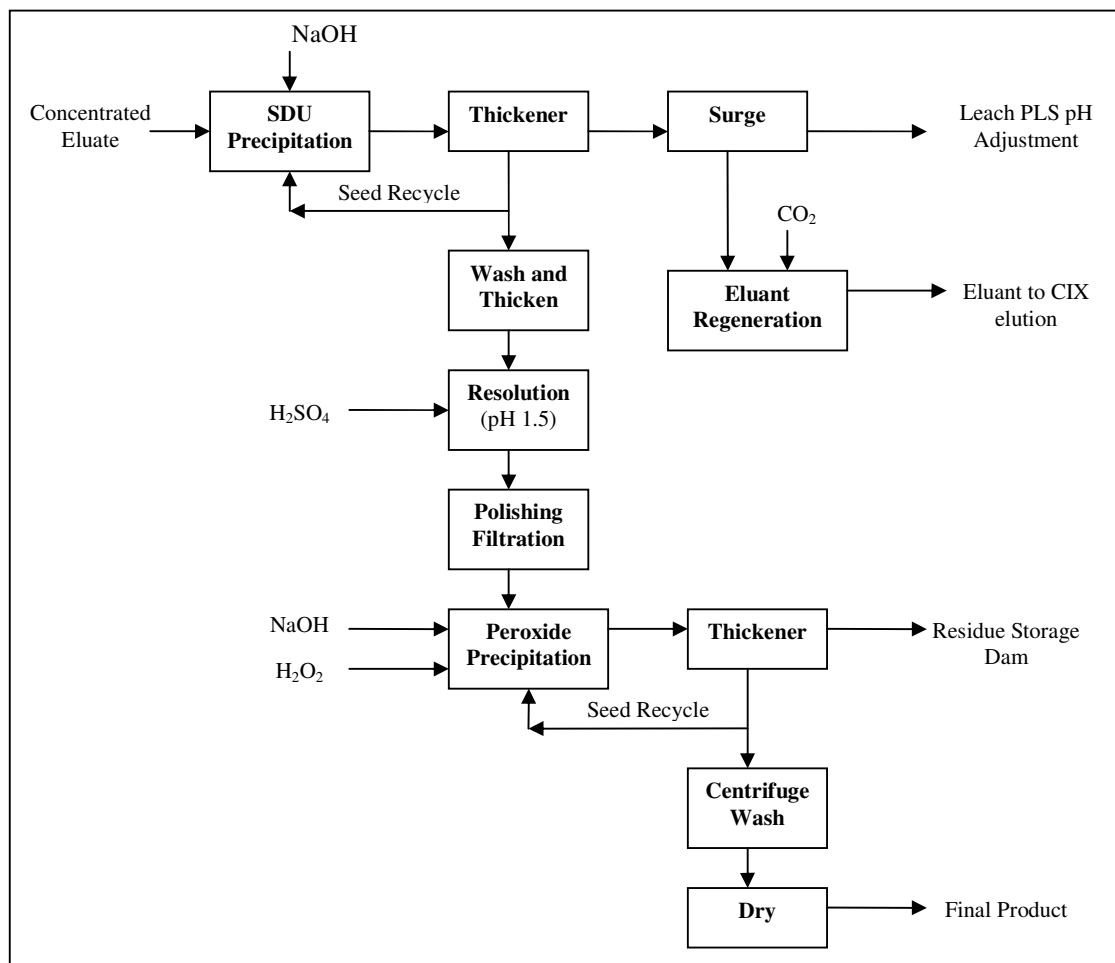


Figure 9: Uranium Peroxide Production Flowsheet

The concentrated eluate from the CIX circuit was converted to acceptable purity uranium peroxide. A sodium diuranate intermediate was produced in order to recover a barren that could readily be converted in part to the eluant for use in the CIX circuit. A bleed of SDU barren was always maintained to the leach PLS to adjust the pH prior to ion exchange. This pH adjusted step is particularly important when carnotite ores are processed and also assists in improving the selectivity of the resin for uranium over vanadium. The final product purity is as shown in Table 4.

Solid Assays - %										
Assay	U	Na	Ca	P	V	Mo	Si	SO ₄	Cl	CO ₃
SDU Precipitate	62-68	6-8	0.001	0.01-0.3	0.07-0.1	<0.001	0.2-0.8	0.03-0.15	0.001-0.03	0.5-3
UO ₄ Precipitate	62-68	0.001-0.1	0.001	0.001-0.1	0.07-0.1	<0.001	0.01	0.03-0.4	0.001-0.03	0.05

Table 4: Typical solid assays for alkaline uranium leach

6. STEADY STATE MODELLING OF CIX SYSTEMS

Modelling of ion exchange circuit at steady state requires consideration of the batch nature the process. The modelling aspect is broken down into two sections,

1. Chemical
2. Physical

The chemical aspects cover the interactions of the resin with the solutions present in the circuit. Based on the solution concentration, flowrate and column configuration, the key variables that need to be determined are

- Resin Bed Volume Requirements
- Resin Chemical Properties
- Resin Chemistry

Physical aspects address the entrainment and impurity department issues that relate to the mechanical design of the unit. The key variables that need to be determined are,

- Resin Physical Properties (size & packing characteristics)
- Resin Inventory
- Column & pipe work Properties

Pilot plant data is used to calibrate the resin behaviour. Relationships can be developed to generate a predictive model of the resin for the PLS solution matrix. In order to gain a greater insight, samples of liquor can be taken from individual columns at any stage through the process. This allows the actual data to be cross checked against testwork data.

When the CIX unit is scaled up to production size, the physical change in the unit, and changes to piping volume can be modified to identify the change in interstitial solution hold up, and the change in mechanical impurity entrainment.

There are two separate ion exchange models. The first is the plug flow, the second is the flow dependent mixing option.

6.1 OPTION 1: PLUG FLOW

Plug flow is a simplified view of flow through the column. The interstitial liquor in the column does not mix with the incoming liquor. The displacement is volume based therefore a solution density can cause mass changes in the column when changing positions.

For high column flow scenarios, typically greater than 2 bed volumes, and low flow scenarios less than 0.4 bed volumes, are accurately represented by the plug flow column.

Shown in Figure 10 is an example of a 3 column displacement wash cycle. The assumption has been made that the station time (the time each column spends in a given position) is long enough to cause complete mixing of the two liquors. Alternatively if the step time was short, the two liquors can be left in their respective positions without mixing. The new flow of 1 BV is introduced to column 3, to displace entrained uranium in stream P105 and prevent it from reporting to stream P108.

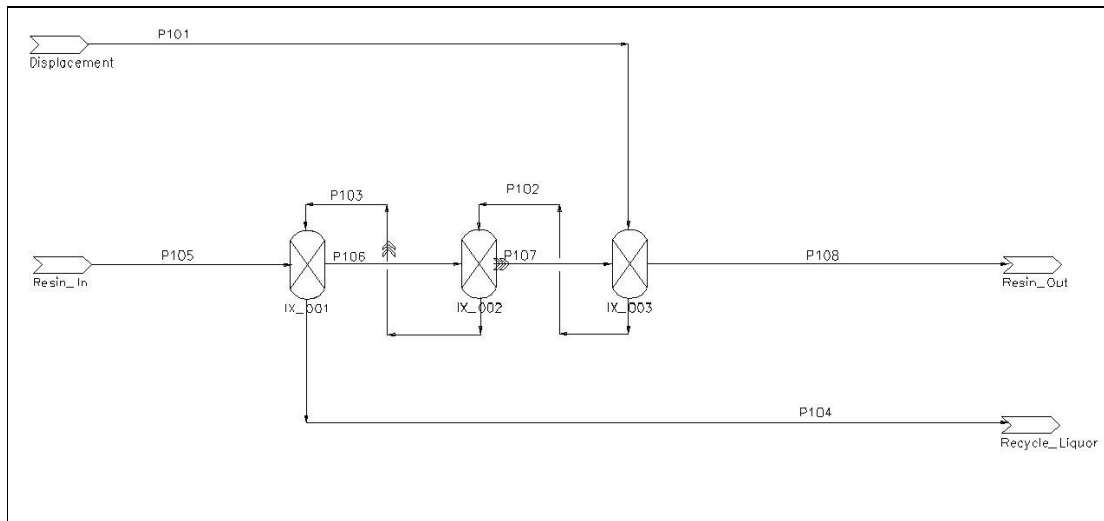


Figure 10: Three Column Displacement Wash Cycle Flowsheet

Stream Number		P101	P102	P103	P104	P105	P106	P107	P108
Stream Name		New Liquor	Column 3 Discharge Liquor	Column 2 Discharge Liquor	Displaced Liquor	Resin In	Resin Transfer C1 to C2	Resin Transfer C2 to C3	Resin Out
Total mass	kg/h	89	89	89	88	175	176	176	176
Solids	kg/h	-	-	-	-	100	100	100	100
Liquids	kg/h	89	89	89	88	75	76	76	76
Total flow	L/h	88.5	88.5	88.5	88.5	163.7	163.7	163.7	163.7
Solids	L/h	-	-	-	-	88.5	88.5	88.5	88.5
Liquids	L/h	88.5	88.5	88.5	88.5	75.2	75.2	75.2	75.2
Liquids Mg	kg/h	0.180	0.178	0.174	0.026	-	0.148	0.151	0.153
Liquids U ₃ O ₈	kg/h	-	0.001	0.002	0.050	0.050	0.002	0.001	-
Liquids Mg	g/L	2.034	2.007	1.963	0.294	-	1.963	2.003	2.034
Liquids U ₃ O ₈	g/L	-	0.009	0.023	0.568	0.664	0.023	0.010	-

Table 5: Example Mass Balance

As demonstrated in the example, all soluble uranium is displaced and prevented from reporting to the next stage.

6.2 OPTION 2: FLOW DEPENDENT MIXING

Flow dependent mixing is based on the flow characteristic of solution through an ion exchange column. The interstitial liquor is no longer directly replaced by incoming liquor. Depending of the number of bed volumes of new liquor introduced to the column, the amount of interstitial liquor that is ejected is a function of the resin hold up. This makes allowances for liquor that is held up in the resin and not subjected to the bulk flow through the column.

A typical curve of this system is show in Figure 11.

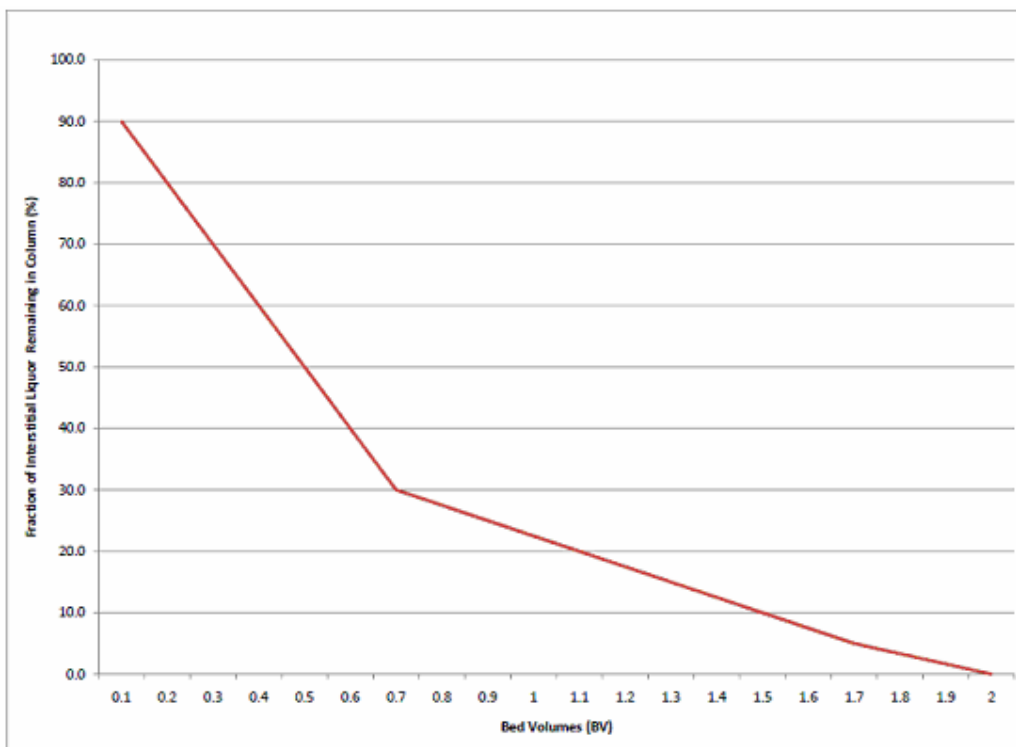


Figure 11: Interstitial Liquor in Column Vs Bed Volumes of New Liquor

7. CONCLUSION

The process to achieve both high first pass and high overall recoveries for uranium coupled with acceptable purity from the alkaline leach liquor has been described. The following steps played an important part in achieving these objectives

- The use of multicolumn ion exchange columns supported by an efficient rotary distribution valve of the type produced by Ionex Separations;
- Incorporating column interstitial liquor entrainment removal intervention;
- High concentration eluant with interstage carbon dioxide conversion of eluted carbonate to bicarbonate;
- Eluted resin reconditioning prior to adsorption;
- Incorporating the CCD#2 overflow into the ion exchange circuit to enhance uranium wash recovery, and
- Three stage split elution circuit.

8. REFERENCES

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