URANIUM RECOVERY FROM ACID LEACH LIQUORS: THE OPTIMISATION OF RIP/SX BASED FLOWSHEETS

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ABSTRACT

Comparison of various technologies for uranium recovery from sulphuric acid leach solutions shows that Resin-in-Pulp (RIP) and/or Solvent extraction (SX) based processing routes are usually cost effective (11). A combined RIP-SX recovery-purification route and a CCD-SX recovery-purification route were investigated and optimised. The design basis used for the investigation was a production rate of 200 kg/h U\textsubscript{3}O\textsubscript{8} over a solution concentration range of 40 to 1500 mg/L U\textsubscript{3}O\textsubscript{8}.

The OPEX for the RIP-SX processing route was found to be sensitive to the variation in uranium concentration in the leach liquor. One of the main drivers for this trend is the leach acid concentration. Conversely, the CAPEX of the CCD-SX processing route was sensitive to the uranium concentration in the leach liquor. The leach acid concentration only affected the OPEX of the CCD-SX processing route modestly, as the bulk of the acid is recovered in a pre-leach circuit.

Contrary to expectation, the effect of extractant loss assumptions on the overall processing route economics was found to be modest to insignificant. It should be noted that the CCD-SX processing route OPEX was more sensitive to extractant loss assumptions than the RIP-SX processing route OPEX.

Economic optimum wash ratios were determined for the CCD-SX processing route and increased with leach concentration – the increase in wash ratio was less than proportional. A 5-stage CCD configuration was found optimal over the concentration range investigated.

1. INTRODUCTION

Historically, uranium recovery and purification has been linked to the development of new technologies to simplify the flowsheets and improve associated process economics. Examples of this include the commercial use of solvent extraction and the development of continuous counter-current ion exchange contactors (such as for the NIMCIX technology). The development of better extractants (i.e. resins and solvents) occurred in parallel with the development and improvement of contacting equipment. The most promising processing routes, based on state of the art technology, were investigated recently and compared on economical grounds (11). This study indicated that a processing route comprising Resin-in-Pulp for uranium recovery, followed by Solvent Extraction (SX) for purification, would be the processing route of choice up to a uranium
concentration of 900 mg/L \( \text{U}_3\text{O}_8 \) in the leach liquor. However, at higher uranium concentrations a processing route comprising Counter-current Decantation (CCD) followed by Solvent Extraction (SX) for purification, would be economically preferable. These findings were based on generalised assumptions. This paper explores these assumptions affecting these two processing routes.

2. LITERATURE REVIEW

The various flowsheets for the recovery of uranium from milled ore, by both sulphuric acid and carbonate leaching, have been comprehensively described by Merritt(1). Earlier literature on the comparison of flowsheet options for the post-leach part of the plant is discussed in this section.

Important factors influencing flowsheet or technology selection include (2):

- Uranium concentration in the leach liquor.
- Leach liquor acid concentration / pH.
- Temperature of the leach liquor.
- Volumetric flow rate.
- Feed slurry solids content and ore mineralogy.
- Environmental constraints and fire hazards.
- Flexibility and control systems – dealing with variation in the volumetric flow, uranium concentration and solids content of the leach liquor.
- Product specifications.

These factors are subsequently discussed separately, but it should be kept in mind that the choice of an optimum flowsheet or technology is based on evaluation of the combined effect and interaction between these factors. The weight given to each factor in a technology selection decision will also shift from application to application depending upon the location and external environment.

2.1 Leach Liquor Composition

The leach liquor composition is, intrinsically, one of the main factors affecting process selection, with the uranium tenor playing a key role. IX might be favoured over SX when large volumes of leach liquors with low uranium concentrations are to be treated, because solvent losses are primarily related to the volume of solution handled (2). Brown and Hayden concluded that, for concentrations greater than 0.9 g \( \text{U}_3\text{O}_8/L \), SX is favoured and below 0.35 g \( \text{U}_3\text{O}_8/L \), IX would be more economical (5).

For some operations, SX might be the preferred technology, as it can treat a greater range of acidic feed solutions (lower pH values) without a major change in behaviour, while an increase in the acid concentration has an adverse affect on the loading capacity of the IX resin, hence the higher cost of an IX circuit. If a variable acid content in the leach liquor
could be handled, it significantly increases the flexibility of the leaching circuit operation (2).

The presence of impurities or by-products will play a further key role in influencing process selection (e.g. calcium sulphate fouling, the presence of molybdenum and vanadium species that poison or co-load onto the IX resins, or chlorides suppressing extraction).

2.2  Leach Liquor Temperature

IX systems can usually handle feed slurries/solutions up to 60°C, while the temperature, in the case of SX, needs to be kept below 50°C to limit solvent losses (7). However, IX systems are more sensitive to temperature shock, which could cause resin breakage.

2.3  Leach Liquor Volumetric Flow Rate

CCIX is particularly suited to the treatment of large flows of near-clarified solution with relatively low uranium concentrations. The direct cost of recovery of uranium, by SX employing mixer-settlers, is proportional to the volume of solution treated, rather than to the amount of uranium produced, whereas the operating costs of IX processes are less dependent on the volume treated (5).

2.4  Leach Liquor/Slurry Solids Content and Characteristics

For FBIX systems, near-completely clarified feed solutions are necessary; otherwise the operating pressure might become excessive and restrict solution flow through the columns (2). The degree of clarification required is a function of the resin bead size and distribution.

Conventional mixer-settler SX systems also require near-completely clarified feed solutions to limit crud formation, associated operational problems and solvent losses. However, SX employing Bateman Pulsed Column technology has proven to be more robust and can handle feed solutions with up to 300 mg/L solids.

In the past, S/L separation has generally been done by either rotary drum filtration, CCD, or horizontal belt filtration. The choice of the S/L separation route was dictated by the characteristics of the solids (i.e. clarification/thickening properties, particle size distribution and filterability) (5).

CCIX techniques were developed for an approach to attain true countercurrent flow of resin and solution. This is achieved by flowing the pregnant solution upwards through a vertical column divided into a number of compartments, each holding a resin charge. The resin is fluidised by the upward flow of pregnant solution, which provides good contact of the resin with the solution for effective adsorption, whilst handling unclarified liquors.
Periodically the flow is interrupted or diverted (downwards) to move the resin charge in each compartment downwards to the compartment below it.

The net effect is that pregnant solution enters the bottom of the column and leaves the top as barren solution, while freshly eluted resin enters at the top of the column and becomes loaded with uranium before discharge from the bottom (2). CCIX systems have a major advantage over other uranium recovery and purification technologies in their ability to handle unclarified solution (e.g. from 300 to 1000 mg/L) and even thin pulp streams up to 10% solids. Higher solids contents could not be achieved because of the limitation imposed by the density of commercially available resins (3, 5). Although the literature refers to a solids content of up to 10%, operating plants often indicate that a solids content of only about 500 mg/L could be handled without difficulty.

S/L separation constitutes around 25% of the capital cost of a uranium plant (4, 5) and 15% of the operating cost (5). Hence, there is a strong incentive to eliminate this step. RIP technology was previously developed based on the obvious economic advantage it would offer. RIP systems are designed to treat slime pulps, i.e. slurry mixtures of fine solids and pregnant solutions. However, there are some practical issues around the implementation of RIP which reduce its applicability (5):

- The viscosity of leached pulps is often high and is not conducive to easy resin/pulp separation.
- The abrasiveness of coarse particles in the pulp will cause attrition of the IX resin beads, which could result in unacceptably high resin losses from the circuit.
- The requirement of high efficiency in the extraction of soluble uranium necessitates some means of countercurrent contact of the IX resin and pulp streams. Countercurrent contact of resin and pulp is most readily done by a multistage process with resin/pulp separation and subsequent downstream transfer of the pulp, whilst the resin is transferred upstream. This complicates the technology somewhat, and could lead to a further mechanism for resin loss.

Conversely, RIP and Resin-in-Leach (RIL) could offer various advantages, namely (6):

- The possibility of eliminating costly S/L separation and clarification circuits.
- Improved uranium recovery, especially where the presence of materials, such as shales, clays and zeolites, can lead to a ‘preg-robbing’ phenomenon, by absorption of uranium from the pregnant solution;
- Minimising the water, and, thus, bleed stream requirements.
2.5 Environmental Constraints and Fire Hazard

The increased fire hazard associated with solvent extraction circuits should also be considered in process selection (3). The reality of this hazard has been demonstrated over the past few years by a number of destructive fires in operating SX plants, which caused major processing delays and layout of significant capital.

The IX route is preferable for in-situ leach (ISL) operations due to the risk of contamination of the well-field by solvent carryover (6).

Water management considerations could also dictate flowsheet selection – in arid or semi-arid regions, processes with lower water usage might be preferred (6).

Should land disturbance and conservation be a critical consideration, flowsheets with a smaller footprint would be preferred. Footprint is linked to the complexity of the flowsheet and the types of technology/equipment employed (e.g. footprint for RIP << CCD + SX).

2.6 Flexibility and Control

The following comments highlight some of the flexibility and control differences between the various IX and SX technologies:

(i) CCIX and RIP can handle unclarified leach liquors and dilute slurries, whereas essentially complete clarification of the feed liquor is required for SX and FBIX. This can be particularly important and cause operational problems when treating ores that contain clay-like materials that are difficult to settle, filter and clarify (2).

(ii) SX can handle acidity fluctuations better than IX (2).

(iii) A disadvantage common to all continuous ion-exchange systems (CCIX, RIP) is the requirement to have transfer mechanisms between the various stages for the transport of liquid and resin. This could cause inventory imbalances within the circuit and hence variation around the performance of the system with regards to uranium recovery and impurity loading. The difficulty of measuring resin concentrations in a pulp previously precluded the use of resin inventory control in an automatic feedback loop (2).

2.7 Product Specifications

SX is generally more selective than IX and, therefore, high yellowcake purities are often easier to achieve. For this reason IX or RIP is usually followed by a small SX circuit (7).
2.8 Process Equipment

The comparison of various IX technologies and associated equipment has been dealt with in depth by other authors (4, 5). With regard to SX, the emergence of Pulsed Columns has introduced various benefits when compared to mixer-settlers, including (9):

- Higher extraction efficiencies.
- Lower tendency to crud formation.
- Capability to handle unclarified solutions (up to 500 mg/L solids).
- Reduced energy input for mixing and phase transfer.
- Flexibility – can handle range of volumetric flow rates and feed concentrations.
- Closed operation – no dust ingress and solvent vapour emissions, i.e. reduced fire risk. Pulsed columns can therefore operate at higher temperatures than conventional mixer-settlers.
- Smaller footprint.
- Better dispersion control.

The Bateman Pulsed Column has been in operation on full scale for uranium extraction for a number of years. Additional development work is currently being done to expand the use of Bateman Pulsed Columns to include the scrubbing and stripping duties (8).

Without ignoring the contribution of ‘soft issues’ and external factors to the complexity of selecting the most appropriate flowsheet, process economics remains a key driver in process selection and optimisation, as discussed in more detail in this paper.

3. INVESTIGATION OF PROCESSING ROUTES AND ECONOMICS

From the literature review it is apparent that a large number of factors affect the selection of a particular recovery, upgrading and purification processing route for a new uranium venture. In an attempt to make this decision process more objective, an economic model has been developed by Bateman Engineering based on our current database and the stated process assumptions. Whilst we believe that this is adequate for the purposes of this comparative study, it should be noted that the actual costs could vary significantly based on the nature of the ore body, plant location and a number of other relevant factors. The information should not be used to predict the financial return of any specific project without detail metallurgical test work and full scale financial modeling.

The economic model rates various possible processing routes on the basis of CAPEX, OPEX and uranium recovery. Two of these processing routes were investigated and some of the underlying parameters are discussed in more detail.
3.1 Processing Routes

The processing routes comprise various “best practice” unit operations. The individual unit operations include:

- Leach, which could be either an atmospheric or pressure leach circuit.
- CCD circuit comprising five high rate thickeners in series, with countercurrent flow of slurry and wash liquor. It was assumed that the thickeners operate at 55% (m/m) solids in the underflow and a wash ratio of 2.7 m$^3$ wash/m$^3$ underflow solution was used.
- Pre-leach (PL) comprising a number of mixing tanks, followed by a thickener for S/L separation. In PL, the fresh solids are contacted with the partially clarified leach liquor (i.e. PLS) recycled from the CCD circuit. PL provides both acid and alkali saving and energy benefits. Furthermore, the PLS is “cooled” prior to introduction into the SX circuit, and early leach reactions are initialized.
- Pinned-bed clarifier acting as final barrier for the removal of solids from the PLS going to SX or FBIX. The Bateman Pinned-bed clarifier is very flexible and can handle variable feed flow rates and solids concentrations of up to 5000 mg/L.
- Resin-in-Pulp (RIP) comprising countercurrent contact of slurry and resin in a series of agitated tanks. Four or more stages were used, based on the resin extraction isotherm. The circuit equipment configuration, sizing and costing was based on the Mintek-Bateman developed MetRIX$^\text{TM}$ technology (10).
- Bateman Pulsed Column SX circuit including extraction, scrubbing, stripping and regeneration. In this study Bateman Pulsed Columns are used for extraction only, while Bateman Settlers$^\text{TM}$ are used for the other three duties.
- Ammonium di-uranate (ADU) precipitation, employing aqueous ammonia, carried out on the loaded strip liquor from all the SX circuits.

A block flow diagram of the CCD-SX processing route is shown in Figure 1. Fresh slurry is contacted with the partially clarified overflow from the CCD circuit in Pre-leach. The slurry is separated in the Pre-Leach thickener and fed to the leach circuit. The Pre-Leach thickener overflow reports to the Pinned-bed Clarifier circuit, and the clarified PLS is then fed to the SX circuit. The SX raffinate is recycled to CCD circuit as wash liquor. The CCD underflow solids are neutralized and report to tails. The SX strip section operates in closed loop with ADU precipitation, where yellowcake is produced.
The combined RIP-SX processing route is shown in Figure 2. As no S/L separation step is required for this processing route, Pre-leach contact is not an option. The uranium is extracted from the leached slurry in the RIP circuit, with the associated elution circuit running in closed loop with SX extraction. Again, the RIP circuit performs the bulk extraction, polishing, partial purification, and upgrade of uranium, with SX on the eluate providing final purification and potential upgrading of uranium.

Figure 1. Block Flow Diagram of the CCD-SX Uranium Recovery Circuit.
3.2 Design Basis, CAPEX and OPEX Estimations

The main assumptions used in the model are summarized in Table 2.

<table>
<thead>
<tr>
<th>Design Basis</th>
<th>Financial Basis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Throughput</td>
<td>200 kg U₂O₅/h</td>
</tr>
<tr>
<td>Leached slurry solids</td>
<td>50% (m/m)</td>
</tr>
<tr>
<td>Leach slurry acid</td>
<td>20 g/L as H₂SO₄</td>
</tr>
<tr>
<td></td>
<td>Uranium Price US$100/lb U₂O₅</td>
</tr>
<tr>
<td></td>
<td>Exchange rates US$1.00 = R7.50</td>
</tr>
<tr>
<td></td>
<td>Euro1.00 = R9.50</td>
</tr>
<tr>
<td>Capital discount rate</td>
<td>15% over 20 years</td>
</tr>
</tbody>
</table>

The CAPEX for each unit operation was derived by performing a rough equipment sizing and costing and then factorizing this major equipment cost value to estimate civils, piping, instrumentation, electrical, installation, construction and commissioning costs. This approach was followed for each unit operation. The individual CAPEX costs arising for the various unit operations for a specific processing route, were then summed to arrive at a total CAPEX cost for that processing route. Different factors were used for the various unit operations based on experience and the complexity of the respective circuits. A median factor of 3 was used for most unit operations. It was assumed that the upstream mining, ore preparation, leach area, utilities, drying/calcining and packaging
sections for the various circuits would be similar in cost and these were therefore excluded from the analysis.\(^1\)

The OPEX of each unit operation was estimated and the OPEX costs for the various unit operations included in a specific processing route were then summed to obtain the total processing route OPEX. In addition to this OPEX calculation, a separate OPEX analysis was performed to quantify the benefit of Pre-leach in the overall circuit.

The recovery for each flowsheet was estimated based on experience and allowed for in a mass balance.

### 3.3 Scenario Analysis Rationale

A processing route cost was used to perform scenario analyses on the effect of various parameters on the economics – the yearly “cost” of each processing route was determined as:

\[
\text{Overall processing route cost (OPRC) = Discounted CAPEX + OPEX + Value of uranium losses}
\]

The value of uranium losses was calculated from the recovery of the specific processing route scenario and product purity penalty.

The reason for not using the more conventional financial rating parameters such as IRR, NPV or payback period, is that the revenue generated in any of the processing route scenarios is for the overall mine and processing plant and not for the recovery/upgrading/purification section only. The question arises on how the financial benefit might be quantified for the different processing route scenarios to offset these costs and to generate the conventional rating parameters. It would, therefore, require that a subjective revenue number be generated. This would not necessarily add much value to the analysis, but, instead, make interpretation of the results ambiguous. Furthermore, with the present uranium market boom and soaring uranium price, the costs for any of the processing route scenarios are small compared to the overall revenue. Comparison on a revenue basis would make such differences seem negligible. The actual cost differences between these scenarios, however, differ significantly and this would translate to the same difference in profits.

The parameters investigated for the processing routes were:

- Leach solution uranium tenor.
- Leach solution acid content.

\(^1\) In view of the foregoing, the comparative approach used is valid, but the calculated CAPEX figures should not be used in estimating the overall cost of a specific plant as it excludes common sections and will in reality depend on the unique characteristics of each individual plant and the location.
• Resin loss assumptions.

For the CCD-SX purification route:
• Leach solution uranium tenor.
• Leach solution acid content.
• Solvent loss assumptions.
• S/L separation wash-ratio and barren concentration.

3.4 Results and Discussion

The analysis described above was performed for various scenarios on the two processing routes.

3.4.1 Leach Liquor Uranium Tenor

To analyse the effect of leach liquor uranium concentration on the two processing routes, the unit processing costs were calculated and are presented in Figures 3(a) to (d). For the CCD-SX processing route the optimised CCD wash ratio was used at each concentration (refer to paragraph 3.4.4). Figure 3(a) shows the total unit cost, which is broken down into its contributing parts in Figures 3(b) to (d). For the analysis the total throughput of uranium was fixed at 200 kg/h U$_3$O$_8$ and the volumetric flow rate adjusted according to the concentration in the leach liquor. Hence, for Figure 5, the volumetric flow of the leach slurry changes in inverse proportion to the indicated concentrations. The unit processing cost is defined as the OPRC divided by yearly U$_3$O$_8$ throughput.

![Figure 3(a). The Effect of the Uranium Leach Liquor Tenor on the Total Unit Processing Cost.](image-url)
Figure 3(b). The Effect of the Uranium Leach Liquor Tenor on the Unit Discounted Capital Cost.

Figure 3(c). The Effect of the Uranium Leach Liquor Tenor on the Unit Operating Costs (including Pre-Leach benefit).
From Figure 3 the following aspects were noted:

- Figure 3(b) shows that the Capital Cost for the CCD-SX processing route is more sensitive to the PLS volumetric flow than is the case with the RIP-SX processing route. This is understandable as the main capital component associated with RIP is the contact tanks, for which the costs do not increase as dramatically as the capital cost of more complex equipment units (e.g. CCDs). The Capital Cost for the RIP-SX processing route is significantly lower than that for the other processing route at high volumetric flow.

- Figure 3(c) shows that the effective Operating Cost for the RIP-SX processing route is more sensitive to the PLS volumetric flow than is the case with the CCD-SX processing route. The main contributor to this trend is the Pre-leach (PL) unit operation. For the CCD-SX processing route a substantial amount of the acid and heat from the leach circuit is recovered in the PL circuit, whereas without S/L separation this is not possible in a RIP circuit. The associated acid and neutralization costs become more pronounced as the volumetric flow rate increases. The Operating Cost for the RIP-SX processing route is significantly higher than that for the other processing routes at high volumetric flow.

- Figure 3(d) shows that the value of losses is similar for both flowsheets over the concentration range. Similar barren assumptions were used.

- Figure 3(a) combines these costs and shows that the overall unit cost for these processing routes behaves very similarly.
3.4.2 Leach Liquor Acid Content

The required leach acid concentration depends mainly on the ore characteristics and leach recovery considerations. The effect of the leach liquor acid tenor on the economics was therefore investigated for the two processing routes under consideration and the results are presented in Figures 4 and 5.

![Figure 4. The Effect of the Leach Acid Concentration on the Unit Operating Cost of the CCD-SX Processing Route.](image)
The following aspects are noted from these figures:

- The operating cost of the RIP-SX based processing route is comparatively more sensitive to the leach acid concentration assumption. The CCD-SX processing route is “immunised” to the variation in the leach acid concentration by the incorporation of a Pre-Leach section in this flowsheet. The Pre-Leach section acts as an acid exchanger and recovers the bulk of the acid coming out of the leach. In contrast to this, no acid recovery is possible in the RIP-SX circuit, which not only results in higher leach feed acid requirements, but also in higher associated alkali consumption for tails neutralisation.

- The operating cost is more sensitive to the leach acid concentration at low uranium concentrations than at high uranium concentrations. As the investigation was based on a fixed uranium load, the volumetric flow at higher uranium concentrations is proportionately lower, with an associated lower acid load / cost.

### 3.4.3 Resin / Solvent Loss Assumptions

The sensitivity of the technology operating cost to extractant losses was investigated. Normal loss levels were chosen in line with typical solvent losses in existing applications. A normal resin loss was assumed based on demonstration scale durability trials.
This loss assumption was then varied and the resultant unit OPEX calculated. The results are presented in Figures 6 and 7 for the RIP-SX and CCD-SX processing routes. It should be noted that for the RIP-SX processing route, only the resin loss assumption was varied, and not both the resin loss and solvent loss assumption for the associated SX circuit.

From Figure 6 it should be noted that the resin loss assumption has a small relative effect on the unit OPEX. In contrast with this, Figure 7 shows that the solvent loss assumption has a significant relative effect on the OPEX. The annual resin loss constitutes 4.4% of the OPEX for the RIP-SX processing route against the solvent loss constituting 16.2% of the OPEX for the CCD-SX processing route.

Comparison of the absolute increase in Unit OPEX shows that: At 200 mg/L U₃O₈ leach concentration the effect of doubling the extractant loss was to increase the Unit OPEX by 46 cents/lb U₃O₈ for the CCD-SX processing route against 23 cents/lb U₃O₈ for the RIP-SX processing route. This difference can be attributed to the normal extractant loss assumptions used, which resulted in the cost of the solvent losses to be double that of the resin losses. Comparison of extractant cost per unit loading capacity shows that the solvent cost is 21% higher than the resin cost at a leach liquor concentration of 200 mg/L U₃O₈.

This emphasises the importance of performing extractant loss testwork for flowsheet selection and confirmation on representative ore samples. Furthermore, the findings also show the importance of selecting the type of solvent extraction equipment such as pulsed columns, which minimise organic losses.

![Figure 6. Unit Operating Cost for Selected Resin Loss Scenarios.](image-url)
3.4.4 CCD-SX Wash Ratio

To study the economic optimum CCD wash ratio, the wash ratio was varied to minimise the OPRC at each leach concentration. The result is presented in Figure 8. The wash ratio is defined as the volume of wash solution per volume of mother liquor in the CCD underflow slurry.
The economic optimum wash ratio increases with concentration to compensate for the increased losses to tails that would result when the leach concentration increases. Due to the compounding wash-effect in the series-staged CCD’s, the increase in optimum wash ratio tapers off as the concentration increases.

The present investigation used 5 CCD stages. The wash ratio optimisation assessment was repeated at low leach concentration (i.e. 40 mg/L) with 4 CCD stages and at high leach concentration (i.e. 1500 mg/L) with 6 CCD stages. However, even at these extremes the 5 CCD stage configuration had the lowest OPRC. It can therefore be stated that this investigation found that 5 CCDs at the wash ratios presented in Figure 8 is the economic optimum CCD-SX configuration over the concentration range 40 to 1500 mg/L.
4. **CONCLUSIONS**

The OPEX for the RIP-SX processing route was found to be sensitive to the variation in leach uranium concentration. Conversely, the CAPEX of the CCD-SX processing route was sensitive to the leach uranium concentration. The leach acid concentration only affected the OPEX of the CCD-SX processing route modestly, as the bulk of the acid is recovered in a pre-leach circuit.

Contrary to expectation, the effect of extractant loss assumptions on the overall processing route economics was found to be modest to insignificant. It should be noted that the CCD-SX processing route OPEX was more sensitive to loss assumptions than the RIP-SX processing route OPEX.

Economic optimum wash ratios were determined for the CCD-SX processing route and increased with leach concentration – the increase in wash ratio was less than proportional. A 5-stage CCD configuration was found optimal over the concentration range investigated.

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6. **REFERENCES**


