The design and operation of a continuous ion-exchange demonstration plant for the recovery of uranium

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SYNOPSIS
A description is given of the design of the continuous ion-exchange demonstration plant at Blyvooruitzicht Gold Mine, including details of the process design, the column construction, and the control system. The operating and process results gathered over a period of seventeen months are summarized, and development work and modifications to the process are discussed. It is concluded that the system comprising continuous loading and continuous elution is technically feasible and can be scaled up with confidence.

SAMEVATTING
Die ontwerp van die demonstrasieaangeleg vir deurlopende ionontriefteling by die Blyvooruitzicht-goudmyn word beskryf met vermelding van besonderhede van die prosesontwerp, die kolumkonstruksie en die kontrolesetel. Die bedryfs- en prosessresultate wat oor 'n tydperk van sewentien maande versamel is, word opgesom en ontwikkelingswerk en wyeigings aan die proses word bespreek. Die gevolgskrywings word gemaak dat die stelsel wat uit deurlopende lading en deurlopende uitwassing bestaan, tegnieks uitvoerbaar is en met vertroue volgens skaal vergroot kan word.

Introduction
During the course of the feasibility study undertaken by Blyvooruitzicht Gold Mining Company Limited on the retreatment of certain slimes dams, it became clear that the newly developed circuit consisting of countercurrent decantation and continuous ion exchange, which was undergoing pilot-plant tests at the time, offered many advantages over the conventional system of rotary-drum vacuum filtration and solvent extraction. Countercurrent decantation itself is a well-known operation, and no problems were expected in the scaling up of the data obtained on the small pilot plant.

However, the system of continuous ion exchange was new and, although extensive pilot-plant test work had been carried out, the envisaged scale of the plant made an intermediate unit essential. It was therefore decided to design and install a demonstration plant so that the various scale-up factors could be assessed and the previously developed design procedures could be verified. The work described in this paper was undertaken as a collaborative project between the Extraction Metallurgy Division of the Atomic Energy Board (AEB), the National Institute for Metallurgy (NIM), Blyvooruitzicht Gold Mining Company Limited, and the seven mining groups that are members of the Nuclear Fuels Corporation of South Africa Limited (NUFCOR).

Plant Design and Engineering
In view of the availability of a solvent-extraction plant at Blyvooruitzicht with sufficient uranium capacity to cope with material resulting from the residue dams, it was decided to base the design of the new recovery plant on the Bufflex (Eluex) system. This is the well-known process in which ion exchange and solvent extraction are combined; sulphuric acid is used to elute the resin, and the uranium is re-extracted from the eluate and purified by conventional solvent extraction. The use of this route conveniently allowed for the demonstration plant to be matched to the existing Blyvooruitzicht plant without any real disruption of the flow of uranium, and it was thus possible to test not only the ion-exchange process itself but the total integrated system on a full commercial scale. The flow-sheet is shown in Fig. 1, and the demonstration plant in Fig. 2.

Design Parameters
The following are the parameters on which the plant was based:

- Flow-rate of pregnant solution 1450 l/min
- Recovery of uranium 99 per cent
- \( U_2O_8 \) concentration in eluate 5 g/l.

The Loading Column
Experience suggested that a specific flow-rate of 300 l/min/m² should be used, which would require a column 2.5 m in diameter. Under this condition, the average bed expansion for IRA-400 (the resin used initially in the column) was 100 per cent, although it varied from top to bottom as a result of the loading of uranium onto the resin. It was estimated that six active stages would give the required recovery of 99 per cent based on a resin loading of 30 g of \( U_2O_8 \) per litre, the top stage being twice as high as the others to accommodate the slug of resin in transfer. The resin inventory was 14.8 m³.

Except for the bottom stage, the stages were separated by simple perforated trays, the perforations being 12 mm
in diameter. The bottom tray was fitted with caps that were designed to control the resin flow and to ensure uniform distribution of the resin over the stages.

The column was constructed from 316L stainless steel of 6.3 mm thickness, and comprised seven flanged sections (each 1 m) fitted with Flexitallic gaskets, an upper section flared to a diameter of 3.75 m, an overflow launder, and a conical bottom. The trays were also made of 316L stainless steel 6.3 mm thick. Windows were fitted to each stage to facilitate observation of the movement of resin.

The Elution Column

The elution column was similar to the loading column, having a diameter of 1.2 m and 7 stages (each 1 m in height) to give the desired residence time. The resin inventory was about 6.2 m³ of wet-settled resin in the sulphate form. The internal design of the column was similar to that of the loading column.

The column was constructed of 316L stainless steel of 6.35 mm thickness, and consisted of 8 flanged sections (each 1 m) fitted with Flexitallic gaskets (each section being a single stage), a flared top section (2.4 m), an overflow launder, and a conical section at the bottom. The trays were made of 316L stainless steel of 6.3 mm thickness.

The tops of both columns were flared to accommodate surges that might cause the solution to overflow during the addition of resin.

The Resin-transfer Vessels

Each of the columns required a resin-transfer vessel to receive and isolate the resin that was transferred from vessel to column under hydraulic pressure. The vessels were constructed from 316L stainless steel of 6.3 mm thickness and consisted of a standard flanged dome (1.5 m in diameter) at the top and a flanged cylindrical section (1.5 m in height) onto which was welded a 45° cone bottom.

A division tray of 6.3 mm thickness and fitted with resin strainers was fitted between the dome and the vertical section to prevent loss of resin during reverse flow. The size of these vessels was based on the transfer volume of all the resin that had settled on a tray in one stage of the loading column.

Control Equipment

The plant was automated so that the operator would be required to spend a minimum amount of time on plant operation. Electromechanical timers were used to control the cycle times on the loading and elution columns. Valve sequencing was achieved by an electrical circuit incorporating the electromechanical timers, which activated solenoid valves. The solenoid valves activated air-operated valves by means of an air signal.

Reverse Flow of Resin

The control of reverse flow was achieved by a floatless switch and electrodes fitted at the top of each column.

Fig. 1—Flowsheet of the demonstration plant
The electrodes were immersed in the liquid and positioned in such a way to control the volume of solution and resin that was extracted from each column on reverse flow. They achieved this control by activating and deactivating the air valve at the bottom of the column.

**Flow**

Differential-pressure cells and orifice plates controlled the flow by activating an air-controlled valve. The controllers were coupled to a chart recorder, and were installed on the pregnant-solution and eluant feed lines.

**Make-up of Acid**

A conductivity unit was used in conjunction with a current-to-air transducer and a two-term controller to record and control the strength of acid in the eluant make-up tank.

**Resin Level**

An ultrasonic level detector was used in conjunction with two electromechanical timers to control the resin interface in the elution column, and thus a balance of resin was maintained automatically between the loading and elution columns.

**Alarms**

A low-level alarm fitted in the eluant make-up tank consisted of a floatless switch, the electrodes being positioned so that the conductivity probe was always immersed in acid. This arrangement prevented incorrect make-up of acid and gave warning when there was too little acid in the circuit.

A further alarm gave warning of the failure of the flow of concentrated acid due to empty storage tanks or freezing of the acid-supply line. This was again accomplished by means of a floatless switch, the electrodes being two lengths of acid-supply pipes connected by a polyvinylchloride (PVC) union. The acid flowing through the pipe gave a resistance of 450Ω, as opposed to a resistance greater than 20 kΩ during a no-flow period. As a safety feature, a Mercoid pressure switch was fitted in the air-line to the valve to prevent a warning signal when the valve was closed. A warning could therefore be given only when the valve was open and no acid flowed.

A pressure switch was fitted to the main air-line to the plant so that, if the pressure fell below a predetermined level, an alarm would be activated.

**Ancillary Equipment**

Various items of ancillary equipment were required.

**Gyratory Screens.** The system required dewatered, washed resin to be sent into the top of each column. This requirement was achieved by gyratory screens of 1,22 m diameter fitted with 50-mesh 316L stainless-steel screening and a washing unit employing a circular spray of water.

**Pumps.** The pumps used on the plant were acid resistant, and were mainly of a plastic, glandless type. These gave many problems and would not normally be recommended.

**Piping.** Polypropylene piping of suitable size was used throughout the plant.

**Valves.** Both automatic and manual diaphragm valves lined with rubber or PVC were used on the plant.

**Engineering Performance of the Plant**

The five objects of the engineering and development programme are set out in another paper1. The demonstration plant provided a major test of the five aims, and some interesting points emerged that are worth discussing.

The design of the distribution tray, which was based on work carried out on the 1,8 m hydraulic rig and the 0,6 m-diameter pilot plant, proved to be adequate. This was determined by visual observations and tracer tests. The demonstration plant showed what is believed to be an advantage of the column approach over the system of open tanks. The solution flowing up the column is redistributed at each stage so that, by the time it reaches the top stage, it is perfectly distributed and the fluidization of the top bed is absolutely uniform. This leads to almost no surging in the top stage and thus minimizes the entrainment of resin into the barren solution. The least-uniform stage is the bottom, where the solution flowing at relatively high velocity out of the feed pipe has to be distributed. With a system of horizontal tank contacting, this problem arises at every stage.

The resin-control caps worked perfectly, and at no time were any stages observed to be depleted.

No changes needed to be made to the resin-transport cycle. The cycle provided stable movement of the resin, and there were no unforeseen problems in moving the large slugs of resin.

The flaring of the top of the column is considered to be essential if it is to cope with surges during the addition of resin to the top of the column. Both columns operate fairly close to the terminal settling velocity of the resin beads, and hence any sudden increase in velocity can give rise to elutriation of the smaller beads. It should be remembered that the problem is not with the bulk flow but with eddies generated during the dumping of the resin into the top.

**Metallurgical Performance**

The demonstration plant operated for a period of seventeen months, nine months of which was on Amberlite IRA-400 and the balance on Duolite A101 DU. The former batch of resin comprised 50 per cent new resin, 25 per cent unused resin that had been stored for 7 years and had partially dried out, and 25 per cent used resin. This batch was therefore unrepresentative of IRA-400, and will not be discussed here except to indicate that, apart from slower kinetic reactions and possibly poorer attrition resistance, it was similar in performance to the A101 DU. The specification for the Duolite A101 DU is given in Table I, which compares that resin with new IRA-400.

Table II gives a summary of the metallurgical results for the run with A101 DU based on 24-hour drip samples. All the down-time in this period can be attributed to general maintenance, weekly planned plant stoppages,
regeneration, and generally to lack of feed solution from the main plant because of various problems.

A feature of the run was that, because the plant was built as a demonstration plant and not a production unit, no regeneration facilities were provided. Silica was merely allowed to accumulate until such time as the efficiency of loading and elution became completely unacceptable. The plant was then stopped, and the complete resin inventory was regenerated in the loading column. This mode of operation influenced the consistency of the metallurgical results.

Thus, the initial stage of each phase gave barren solutions containing 0.001 g of U₃O₈ per litre, and, as the silica loading approached 4.5 per cent, the usual decrease in loading rate was observed, resulting in an increase of U₃O₈ in the barren solution.

In the first phase the loading column gave an average of 0.005 g of U₃O₈ per litre in the barren solution, although the U₃O₈ in the barren solution gradually increased from 0.001 to 0.005 g/l owing to the accumulated silica. The average loading was 29.5 g of U₃O₈ per litre, resulting in a loading efficiency of 98.5 per cent.

The elution column averaged 6.28 g of U₃O₈ per litre in the concentrated eluate, with an average eluted-resin value of 1.6 g of U₃O₈ per litre, resulting in an elution efficiency of 94.0 per cent.

The only down-time during this period was for the normal weekly stoppages of the plant.

In the second phase the loading column gave an average barren solution of 0.009 g of U₃O₈ per litre with an average loading of 30 g of U₃O₈ per litre, resulting in a loading efficiency of 98.5 per cent.

The elution column gave an average concentrated eluate of 6.5 g of U₃O₈ per litre, with an average eluted-resin value of 1.23 g of U₃O₈ per litre, resulting in an elution efficiency of 96.0 per cent.

The main down-time was again due to normal weekly stoppages of the main plant. Some forty-eight hours of down-time was needed for the replacement of two sight windows that had cracked.

Phase 3 lasted from 29th October, 1976, to 30th January, 1977. This run was conducted on clarified pregnant solution, the results being similar to those in the first two phases.

Tables III and IV give typical concentration values for both the loading column and the elution column.

### Analysis of the Metallurgical Results

During the 17 months of operation, a large amount of metallurgical data was amassed. So that these data could be analysed, multilinear regression techniques were used to establish the effect of the various operating variables on the important outputs of the two columns, namely the barren solution from the loading column and the eluted resin from the elution column. The following operating variables were considered:

**Loading column**
- Flow-rate of pregnant solution
- U₃O₈ in pregnant solution
- H₂SO₄ in pregnant solution
- pH of pregnant solution
- Fe⁺⁺ in pregnant solution
- SiO₂ on eluted resin
- U₃O₈ on loaded resin

**Elution column**
- Flow-rate of eluant
- U₃O₈ in eluant
- H₂SO₄ in eluant
- Temperature of eluant
- U₃O₈ on eluted resin
- SiO₂ on eluted resin

### Tables

#### TABLE I

**SPECIFICATION OF THE RESINS**

<table>
<thead>
<tr>
<th>Mesh Tyler</th>
<th>Sieve opening</th>
<th>Distn of</th>
<th>Data of</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>IRA-400</td>
<td>Duolite</td>
</tr>
<tr>
<td></td>
<td></td>
<td>%</td>
<td>A101 DU</td>
</tr>
<tr>
<td>mm</td>
<td>in</td>
<td>%</td>
<td>%</td>
</tr>
<tr>
<td>&gt; 20</td>
<td>0.84</td>
<td>0.0331</td>
<td>23.00</td>
</tr>
<tr>
<td>&lt; 20 &gt; 48</td>
<td>0.295</td>
<td>0.0116</td>
<td>15.95</td>
</tr>
<tr>
<td>&lt; 48 &gt; 100</td>
<td>0.147</td>
<td>0.0058</td>
<td>0.12</td>
</tr>
</tbody>
</table>

Basis: air-dried sulphate form

#### TABLE II

**SUMMARY OF METALLURGICAL RESULTS WITH A101 DU RESIN**

<table>
<thead>
<tr>
<th>Period</th>
<th>Running time</th>
<th>Date of measurement</th>
<th>Flow-rate, l/min</th>
<th>Efficiency, %</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>h</td>
<td></td>
<td>Loading</td>
<td>Elution</td>
<td>Loading</td>
</tr>
<tr>
<td>15.6 to 9.8</td>
<td>1280</td>
<td>15.6.76</td>
<td>1450</td>
<td>42</td>
<td>98.5</td>
</tr>
<tr>
<td>10.8 to 17.10</td>
<td>1100</td>
<td>25.8.76</td>
<td>1450</td>
<td>45</td>
<td>98.5</td>
</tr>
<tr>
<td>18.10.76 to 30.1.77</td>
<td>1800</td>
<td>29.10.76</td>
<td>1450</td>
<td>50</td>
<td>97.7</td>
</tr>
</tbody>
</table>
The parameters of resin flow were not included because these remained constant.

The agreement between the curves was not very good owing to the usual problems in the analysis of plant data, and quantitative relationships are not worth quoting. However, the fit obtained was considered adequate for the selection of those variables that should be watched carefully in normal plant operation.

The following variables were found to have the most effect (in order of significance) on the \( \text{UO}_2 \text{O}_4 \) concentration of the barren solution:

- the silica content of the eluted resin,
- the \( \text{Fe}^{3+} \) content of the pregnant solution, and
- the \( \text{UO}_2 \text{O}_4 \) content of the eluted resin.

The influence of the first and third variables was expected, but the effect of the \( \text{Fe}^{3+} \) in the pregnant solution seems rather surprising.

It is well known that the sulphate complexes of ferric iron compete with uranium for ion-exchange sites, but it would be expected in normal circumstances that the uranium would displace the ferric complex. An interesting feature of the A101 DU resin was that it was observed to turn brown almost as soon as it was introduced at the top of the loading column, indicating that the iron was adsorbed very rapidly. It has been reported that iron adsorption slows down the rate of uranium adsorption, and this possibly explains the strong influence of the iron on the barren solution.

The concentration of the eluted resin was influenced most significantly by the silica content of the resin and the temperature of the eluant. Again, the influence of the silica is very significant.

The temperature of the eluant was not controlled and was allowed to fluctuate with the atmospheric temperature. It also changed during acid make-up. The significance of the influence of temperature emphasizes the advantages of hot elution.

### Silica Fouling

The rate of loading of silica onto the A101 DU is shown in Fig. 3, which indicates that little change in rate occurred after successive regenerations. It appeared that the rate of uranium loading was really significantly affected only by a silica content of more than 4.5 per cent.

### Loss of Resin Due to Attrition

During the first six months of this test, a determined effort was made to obtain a reliable figure for resin losses, efficient screens being mounted at all the solution exits from the plant in an attempt to control the physical loss of resin. It was estimated that losses during this period were 0.25 per cent per month. So that the amount of resin in the plant could be determined, all the resin was placed in the loading column and the settled height was measured. The loss was at the limit for accurate measurement; a more accurate figure could be obtained only by running the plant for much longer periods. The value was nevertheless very much lower than those previously estimated.

Regular microscopic examination of the A101 DU

<table>
<thead>
<tr>
<th>TABLE III</th>
<th>URANIUM LOADING ON A10I DU RESIN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Forward flow-rate of pregnant soln</td>
<td>1450 l/min</td>
</tr>
<tr>
<td>Period of forward flow</td>
<td>155 min</td>
</tr>
<tr>
<td>Stages</td>
<td>( \text{UO}_2 \text{O}_4 ) in solution g/l</td>
</tr>
<tr>
<td>Overflow</td>
<td>&lt; 0.001</td>
</tr>
<tr>
<td>Top 1</td>
<td>0.001</td>
</tr>
<tr>
<td>2</td>
<td>0.001</td>
</tr>
<tr>
<td>3</td>
<td>0.001</td>
</tr>
<tr>
<td>4</td>
<td>0.012</td>
</tr>
<tr>
<td>5</td>
<td>0.045</td>
</tr>
<tr>
<td>6</td>
<td>0.094</td>
</tr>
<tr>
<td>7</td>
<td>0.145</td>
</tr>
<tr>
<td>Pregnant soln in</td>
<td>0.200</td>
</tr>
</tbody>
</table>

Note: These are maximum values. The samples were taken 10 minutes before a resin transfer.

<table>
<thead>
<tr>
<th>TABLE IV</th>
<th>ELUTION OF URANIUM FROM A10I DU RESIN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow-rate of eluant</td>
<td>45 l/min</td>
</tr>
<tr>
<td>Period of forward flow</td>
<td>45 min</td>
</tr>
<tr>
<td>Stages</td>
<td>( \text{UO}_2 \text{O}_4 ) in solution g/l</td>
</tr>
<tr>
<td>Overflow</td>
<td>7.08</td>
</tr>
<tr>
<td>Top 1</td>
<td>9.15</td>
</tr>
<tr>
<td>2</td>
<td>5.53</td>
</tr>
<tr>
<td>3</td>
<td>4.58</td>
</tr>
<tr>
<td>4</td>
<td>2.55</td>
</tr>
<tr>
<td>5</td>
<td>2.65</td>
</tr>
<tr>
<td>6</td>
<td>1.50</td>
</tr>
<tr>
<td>7</td>
<td>0.66</td>
</tr>
<tr>
<td>Eluant</td>
<td>0.028</td>
</tr>
</tbody>
</table>
RESULTS OF MICROSCOPIC EXAMINATION OF THE RESINS

<table>
<thead>
<tr>
<th>Resin</th>
<th>Uncracked spheres</th>
<th>Cracked spheres</th>
<th>Non-spherical particles</th>
</tr>
</thead>
<tbody>
<tr>
<td>New Amberlite IRA-400 (CL) ex factory</td>
<td>95</td>
<td>5</td>
<td>0</td>
</tr>
<tr>
<td>New Amberlite IRA-400 (SO₄) ex factory</td>
<td>95</td>
<td>5</td>
<td>0</td>
</tr>
<tr>
<td>New Duolite A101 DU (CL) ex factory</td>
<td>100</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>New Duolite A101 DU (SO₄)</td>
<td>100</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Duolite A101 DU (SO₄) 29.9.76 after 1st regeneration</td>
<td>100</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Duolite A101 DU (SO₄) 27.11.76 after 2nd regeneration</td>
<td>95</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Duolite A101 DU (SO₄) 22.2.77 as in plant at present</td>
<td>95</td>
<td>5</td>
<td>0</td>
</tr>
</tbody>
</table>

Uranium Balance

The uranium balance for the plant taken from averaged assay values is given in Fig. 4. The column cycles are shown in Fig. 5.

Power Consumption

The power consumption of the plant was estimated from a knowledge of the current drawn by the pumps and of the duration of the pumping periods. The power drawn by the control circuit was negligible. This gave a specific value of 0.105 kW.h per cubic metre of pregnant solution.

Conclusions

The following conclusions can be drawn from the operation of the demonstration plant.

(a) The ion-exchange contacting system comprising a loading column and an elution column is mechanically and metallurgically sound and can be scaled up with confidence.
(b) Unclarified pregnant solution having a suspended-solids content of 200 to 300 p.p.m. can be handled without any difficulty.
(c) Continuous elution with 10 per cent (by mass) sulphuric acid is feasible.
(d) The rate of silica fouling is no greater than with
LEGEND
L = Solution (m³/d)
C = U₂O₅ in solution, kg/m³
R = Wet-settled resin (m³/d)
r = U₃O₈ on resin, kg/m³
U₅ = U₃O₅ in solution, kg/d
U₇ = U₃O₅ on resin, kg/d
V = Volume of solution plus resin (m³/d)

Fig. 4—Uranium balance on the demonstration plant using AL101 DU resin
conventional ion exchange, but the continuous system, being sensitive to kinetic factors, requires that the silica content of the resin should be kept below 4.5 per cent for the Duolite A101 DU resin.

(e) Attrition losses amount to 0.25 per cent per month and are considerably less than was originally expected.

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References