



Cobalt and nickel removal from Zincor impure electrolyte by Molecular Recognition Technology (MRT)—pilot plant demonstration

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Synopsis

The use of Molecular Recognition Technology (MRT), a highly selective separation technology using specifically designed ligands (such as macrocycles), has been demonstrated to be an effective treatment process for removal of several ions that are commonly found in many hydrometallurgical circuits as well as in environmental treatment operations. Ions removed by MRT include base, heavy and transition metals, precious metals, halides, and alkali/alkaline earths.

A MRT pilot plant run was conducted at Zincor to demonstrate the cobalt/nickel purification of zinc electrolyte, and to recover saleable pure cobalt/nickel sulphate solution. The MRT process holds various advantages, which include the elimination of a catalyst such as arsenic trioxide, conventionally used in cobalt and nickel cementation.

SuperLig® 138 can be used to remove cobalt and nickel from 120–170 g/l Zn-solutions at pH 1–2. The Zincor MRT application demonstrated its capability of concentrating large volumes of copper barren impure solution at low cobalt and nickel concentration levels in the feed, to small solution volumes at high cobalt and nickel concentrations. Around 99.5% of the cobalt was removed from solution with cobalt impurity levels in feed varying between 10 mg/l and 26 mg/l.

Introduction

Molecular recognition technology

Reed M. Izatt, Jerald S. Bradshaw, and James J. Christensen, professors of chemistry and chemical engineering at Brigham Young University in Utah were early pioneers in the development of Molecular Recognition Technology (MRT). The company, IBC Advanced Technologies, Inc., was formed as a spin-off from the university. MRT provides rapid, selective extraction and recovery of cations and anions from process or waste streams and enables commercial metallurgical separations to enhance processes that were previously not technically and/or economically feasible.

MRT represents a significant advance over ion exchange systems in that it is a highly selective system using specially designed ligands, such as macrocycles and others^{1,2,3,5}.

These ligands are chemically bonded to solid supports such as silica gel or polymers or used free in solution to complex with selected ions. This is often accomplished without an exchange of ions¹. The solid phase system consists of the bound ligand material, trade-named SuperLig®, packed into fixed bed columns. The columns can be built in a skid mounted modular form, and can be fully automated for continuous operation (e.g., commercial MRT units at Impala Platinum Ltd. and Britannia Zinc Ltd). The ligand exhibits a long life and can often be used through thousands of process cycles. Compared to ion exchange, the MRT ligands have much faster extraction cycles with much greater selectivity and are often more easily eluted. Also, no regeneration of the resins is required, and the resins do not foul readily. These advantages translate to lower capital and operating costs in comparison to alternative separations technologies^{1–3,5,11,13}.

MRT possesses a number of unique features, which overcome the limitations of present separation technologies^{4,9,10,12,14–17,20}.

- MRT utilizes 'host-guest' chemistry to selectively bind with ions based on multiple parameters such as size, coordination chemistry, and geometry. By contrast, conventional chelation chemistry (such as ion exchange) often distinguishes among ions based only on a single parameter (i.e., charge, solubility, or size). Molecular recognition

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Cobalt and nickel removal from Zincor impure electrolyte by MRT

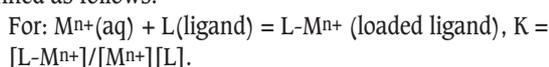
enables ions to be bound even if the target species is present at the ppm-ppb level in the presence of high concentrations (such as 0.1–100 g/l) of other species in large volumes of solution. The system remains effective in difficult matrices such as strong acid and in the presence of other complexing agents.

- ▶ Large numbers of MRT ligands are available which can bind with most of the cations and anions under a variety of process conditions. Various commercial MRT installations are in operation (e.g., Cu electrolyte purification; PGM processing and others).
- ▶ Loading of the target ion is rapid with a sharp loading curve permitting high feed solution flow rates. Removal of the target ion to the desired level can be achieved in a single pass through the system.
- ▶ The stripping of the target species is accomplished in relatively few eluent solution bed volumes, minimizing elution chemical requirements and solution volumes as well as time off line. The high concentration of target species in the eluant solution provides high efficiency and flexibility in the choice of process for product recovery and sale or for disposal. A large choice of elution agents is available to ensure compatibility with plant chemistry and environmental requirements.

Cobalt removal chemistry

The product, SuperLig® 138 binds both Co(II) and Co(III). However, SuperLig® 138 binds Co(III) stronger and with greater selectivity over other elements/ions which have trace affinity for SuperLig® 138. Experimental tests with highly concentrated zinc solutions have shown that Co is only polished to trace or below detection levels from such solutions, bound as Co(III).

This is in agreement with the estimated stability constants of complexation (K-values) of SuperLig® 138 with different metal ions, as given in Table I. The K-value is defined as follows:



Considering the relative K-values of Zn and Co(II) and Co(III), the removal of Co(II) at ppm levels would not be possible at high zinc concentrations (e.g. 150 g/l), whereas Co(III) would bind preferentially even at extremely low concentrations (< 0.1 ppm).

However, Co(III) does not exist to any significant level in the normal ORP-ranges of solutions without a strong Co(III) chelant present. Hence, the only way to bind Co to SuperLig® 138 as Co(III) is to first bind Co(II) to the chelating ligand on SuperLig® 138 and then have the Co oxidized to Co(III). This is possible since Co(II) does bind to the resin (even to a significant but not polished degree in high Zn solutions) and there is only 20–30 ppm Co to be bound to the resin and oxidized in the Zincor solution (which contains sufficient O₂ to perform the oxidation). However, this oxidation can be very slow kinetically if a more rapid oxidizer is not present to speed the process up. Fe(III) is an excellent choice for the task. The Fe(III) is both a rapid oxidizer and the Fe(II) produced is readily reoxidized by O₂ back to Fe(III) if needed. Hence, the Fe(III) almost acts like a catalyst in that it speeds the reaction up and is eventually returned to its starting state.

The Zincor circuit

Zincor uses a roast-leach-electrowinning process to recover zinc from zinc sulphide concentrate. The impure leach solution contains small quantities of copper, cobalt and nickel, which have to be removed before zinc can be electrowon to the desired specification. Zincor currently uses a two-stage zinc precipitation process (Figure 1). In the first stage copper, nickel and cobalt is precipitated together using zinc dust. Arsenic trioxide is added to catalyse the precipitation of nickel and cobalt in this stage. In the second stage cadmium is precipitated together with several minor impurities.

In the envisaged flowsheet, the proposed MRT process will replace the cobalt and nickel precipitation step, and will follow after copper precipitation.

The MRT process holds various advantages for copper and cobalt separations^{6–8,18,19}. For cobalt removal at Zincor, advantages would include:

- ▶ The elimination of arsenic trioxide (which is a potential environmental and health hazard)
- ▶ Reduction of zinc recycle
- ▶ No generation of an arsenic containing by-product
- ▶ The generation of a high-quality cobalt and nickel product which can be sold for toll refining or cobalt electrowinning
- ▶ Simplification of the zinc circuit
- ▶ Improvement in cobalt recovery
- ▶ Reduction in the solution cobalt content to extremely low levels.

Based on these considerations, and subsequent laboratory test work results, Zincor decided to evaluate the MRT process on pilot scale in order to:

- ▶ Demonstrate the ability of the process to remove cobalt from the leachate stream to low or below detection levels
- ▶ Confirm the loading capacities of SuperLig® materials

Table I
Stability constants of complexation—SuperLig® 138

Specie	Log(K) Value
Cu ²⁺	20.0–21.5
Co ²⁺	18.0
Co ³⁺	30.0
Ni ²⁺	19.0
Zn ²⁺	15.2
Mn ²⁺ and Mg ²⁺	None

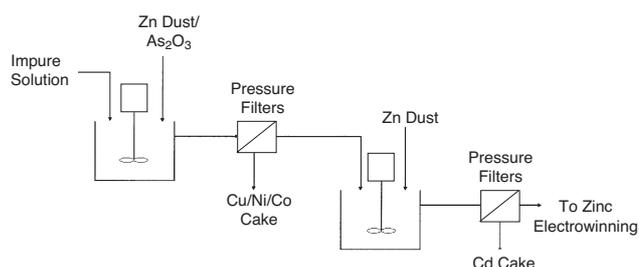


Figure 1—Schematic of the present Zincor cobalt and nickel removal Circuit

Cobalt and nickel removal from Zincor impure electrolyte by MRT

- Determine the requirements for continuous plant operation.
The following methodology was used.
- IBC's bench scale results were used to guide the pilot evaluation, i.e. changes in operating parameters were first tested by IBC and—after consultation with Zincor and MRT Africa—implemented on the pilot plant. (IBC Advanced Technologies is represented by MRT Africa in Africa; Bateman provided engineering support to the pilot campaign.)
- During the pilot plant runs, critical operating and performance parameters were monitored and recorded.

Process description and pilot plant

Process description

The MRT circuit is subsequently described with reference to the Process Flowsheet (Figure 2).

The cobalt-containing leachate solution is fed to tank TK-01 (Table II). The pH is adjusted to between 1 and 2 with sulphuric acid, and a stoichiometric amount of ferric iron added to match the cobalt content. A fixed volume of this solution is passed through a single MRT column (in bulk removal mode, or two columns in series if operated in lead/trail mode). The volume of this solution is based on the cobalt concentration and the ligand capacity.

The raffinate (cobalt-free solution) is discarded to tank TK-07.

Once cobalt breakthrough occurs, the column is washed (from Tank TK-02) with a fixed volume of water to displace any feed liquor. This wash liquor is combined with the raffinate.

Following the water wash cycle, the column undergoes a three-stage elution using 2 M sulphuric acid and 0.5 M SO₂ at 95°C and 250 kPa. The eluate is recycled twice to build up the tenors of cobalt. The cobalt-rich eluate solution is collected as *product* (TK-08).

The eluted column is then washed with a fixed volume of water (from TK-06) for removal of entrained acid from the resin bed, before being brought back on line for the next loading cycle.

Experimental procedure

Various feed solution batches were pumped from the copper precipitation tank to the pilot plant feed tank. Table III shows the compositions of the feed batches.

Wash and eluant solutions were prepared from industrial grade sulphuric acid (supplied by Zincor) and potable water. Samples of these reagents were also analysed for metal impurities. High purity SO₂ was dosed from a gas cylinder during elutions.

Table II
Pilot plant equipment list

Item No.	Description	Size	Qty
TK-01	PLS Surge Tank	2000 litre	1
PP-01	Feed pump: variable speed	300 litre/h	1
SF-01/02	Filters	360 litre/h	1
MT-01/02	MRT Columns	26 litre each	2
TK-02	Pre-elution Wash Tank	500 litre	1
TK-03	Elution 1 Tank (recycle #2)	100 litre	1
TK-04	Elution 2 Tank (recycle #1)	100 litre	1
TK-05	Elution 3 Tank (make-up)	100 litre	1
TK-06	Post-Elution Wash Tank	400 litre	1
TK-07	Raffinate Surge Tank	3000 litre	1
TK-08	Product Surge Tank	100 litre	1

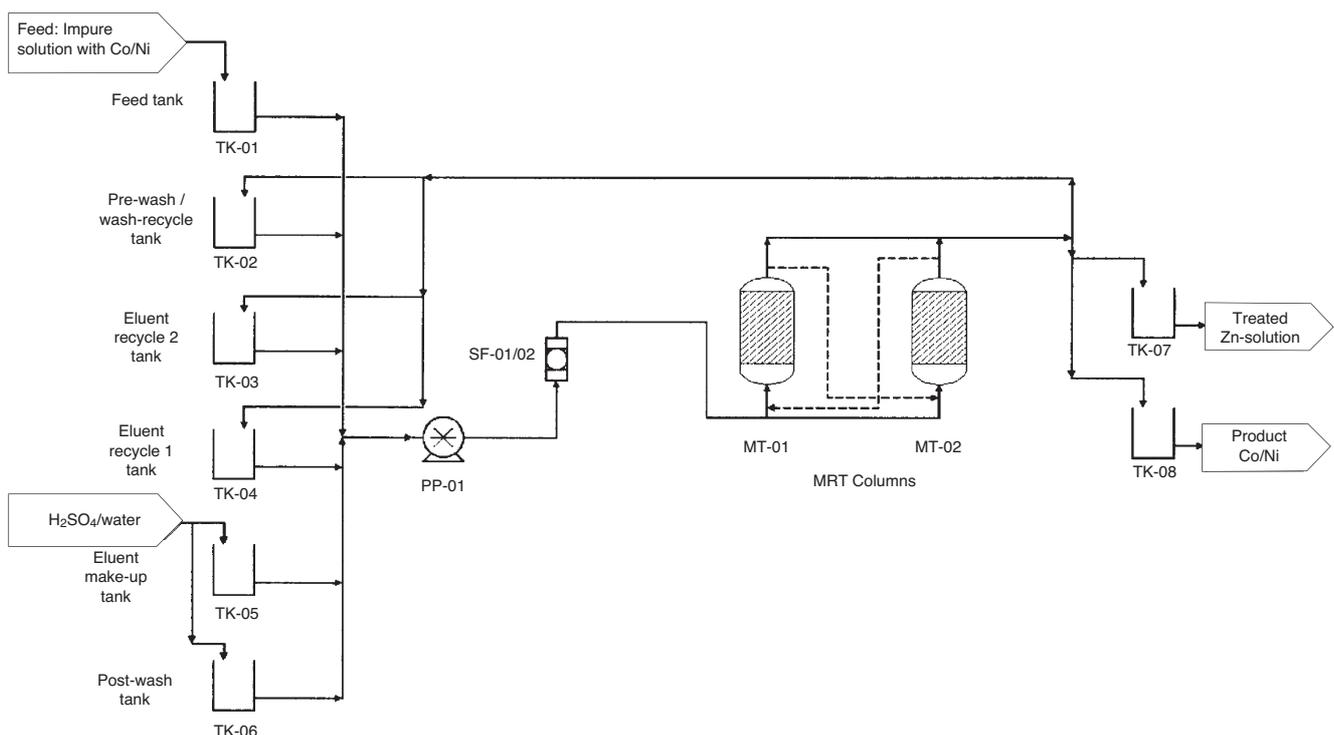


Figure 2—Cobalt removal MRT process flowsheet

Cobalt and nickel removal from Zincor impure electrolyte by MRT

Table III

Zincor leachate solution-batches used as feed in the pilot MRT evaluation after pH and iron adjustment

No.	Chemical composition						
	Co ppm	Cu ppm	Ni ppm	Cd g/l	Zn g/l	Fe ppm	Fe ²⁺ ppm
Feed A	22.6	0.7	2.9	1.4	151	32	4
Feed B ¹	26.1		3.9		183	66	5
Feed C ²	21	0.6	2.5		140	71	4
Feed D	18	0.96	2.2	1.5	164	30	2
Feed E	13	3.6	2.9	1.4	153	35	4
Feed F ³	11	16	2.0	0.1	135	80	63
Feed G	11	16	2.0	0.1	135	80	7

Notes:

1. This feed solution was concentrated due to evaporation as a result of heating.
2. Feed C = Feed B solution corrected by water addition.
3. After ferric sulphate had been supposedly added to the newly prepared feed batch F, poor cobalt removal resulted. Upon investigation the iron-analyses showed that ferrous sulphate must have been added. This was corrected by adding ferric sulphate and peroxide to give feed batch G. Also, note the high copper content of this batch.

Nickel and iron were analysed by atomic absorption spectrophotometry. Cobalt was determined using a colourimetric method using the colour of the cobalt complexed with a nitroso-R-salt. The cobalt analyses were confirmed by ICP.

The operating conditions for the cobalt SuperLig® 138 column are given in Tables IV and V. The test solution input sequence and volumes used for a typical cobalt cycle are shown in Table VI. The solution flow configuration is given in Figure 3.

During the absorption phase of the cycle samples were collected from the cobalt-column outlet at 15 litre intervals, i.e. after every bed-volume of throughput. Both a composite sample of each 15 litre raffinate and a grab sample at the end of the 15 litres were taken.

After the pre-wash solution was displaced from the column by the eluent solution (about 1 BV), the column outlet was sampled at 3 to 5 litre intervals and the temperature profile monitored. The eluate was collected in the product tank, and a composite sample taken.

Results and discussion

Viscosity effect

The initial pilot testing of the cobalt removal SuperLig® 138, at ambient temperature, revealed that the zinc-solution viscosity impairs the cobalt removal performance. Laboratory tests showed that this viscosity effect can be eliminated by operating at an elevated temperature of 60 to 70°C. The pilot plant feed batches were subsequently heated to 70°C. The cobalt-removal performance of SuperLig® 138 in a single column system, at elevated temperature, is shown in Table VII. The pilot plant was operated intermittently and not continuously during this run, and various feed batches were treated.

The associated removal curve for this run is given in Figure 4.

During the initial 10 bed-volumes of this run cobalt breakthrough occurred, because of the extremely high zinc concentration in the feed of 183 g/l (Feed B). Once the feed

Table IV

Loading conditions—SuperLig® 138

Parameter	Value
Flow rate limit	0.2 l/(min.kg SuperLig®)
Resin capacity	0.8 mole Co/kg SuperLig®
Linear velocity	1.2 m/h
Temperature	60 to 70°C for concentrated ZnSO ₄ stream (>120 g/l Zn)
Operating feed pH	1 to 2

Table V

Elution conditions—SuperLig® 138

Parameter	Value
Flow rate limit	0.1 l/(min.kg SuperLig®)
Linear velocity	0.6 m/h
Temperature	95°C
Operating acid content	2 M H ₂ SO ₄

Table VI

Cobalt MRT pilot solution input sequence and operational information (based on 25 ppm Co feed concentration and 3.15 kg SuperLig® per column)

Solution input	Solution volume (l)	Duration (min)	Solution Flow Rate (l/min)
Feed	2655*	4282	0.63
Pre-wash	15.5	25	0.63
2M H ₂ SO ₄ Elution Recycle 2	4.65	15	0.32
2M H ₂ SO ₄ Elution Recycle 1	4.65	15	0.32
Fresh 2M H ₂ SO ₄ Elution	4.65	15	0.32
Post-wash	15.5	50	0.32

Note:

* This was the theoretically predicted load—the actual load was, however, determined from the break-through curves during the pilot evaluation.

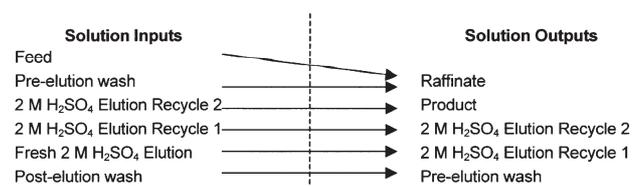


Figure 3—Solution flows for cobalt removal MRT

Table VII

Cobalt removal run 2—single column 2 (SuperLig® 138)

Feed (refer to Table III)	Volume loaded Bed volumes (BVs)	Temperature °C	Flow rate l/min	Avg. cobalt concentration in Raffinate, mg/l	Average removal
Feed B	1 to 10	65	0.63	1.5	94.3%
Feed C	10 to 25	65	0.63	<0.1	99.9%
Feed C	25 to 52	65	0.63	<0.1	99.7%
Feed C exhausted—new batch, Feed D					
Feed D	52 to 60	65	0.63	> 0.1	-

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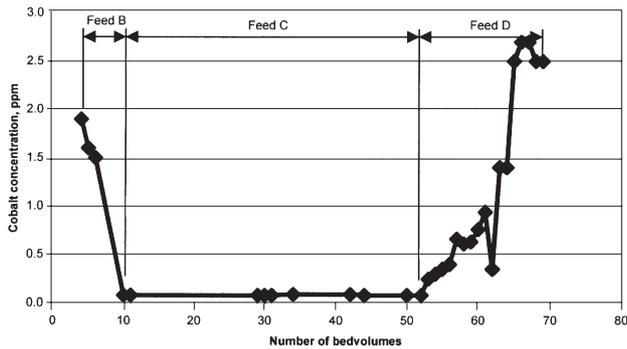


Figure 4—Cobalt removal curve in a single column for run 2 at 65°C and loading rate of 0.2 l/(min.kg resin)

was diluted by addition of water to lower the zinc concentration to 140 g/l, which is Feed C, effective cobalt removal resulted.

This run showed that cobalt can consistently be removed to levels below 0.1 mg/l by operating the system at 65°C. Breakthrough occurred between 50 and 60 bed-volumes (i.e. 750 and 900 litres) of load, which represents a loading of about 0.1 mole Co/kg resin. The consistent removal of cobalt was confirmed by a number of repeat load cycles.

Mass balance

After breakthrough occurred, the column was eluted. The elution was performed at a temperature of above 85°C and absolute pressure of 270 kPa, which ensured that the SO₂ remained in solution. A cobalt mass balance was drawn up, based the amounts of cobalt loaded and eluted. The mass balance given in Table VIII shows similar amounts of cobalt loaded and eluted, which confirms that the elution conditions were effective.

Two columns in series

In a single column system (i.e. bulk removal set-up), the column performs both loading and polishing functions—the resin can therefore not be loaded to capacity. However, when operating two columns in series (in a lead-trail configuration), the load on the system can be increased. Figure 5 shows that with two columns in series (Column 1 lead/Column 2 trail) the system's polishing efficiency improved significantly with the effluent cobalt levels remaining below 0.1 mg/l even up to 72 bed volumes. By extrapolation of the results an estimated 200 bed volumes can be loaded before the effluent cobalt concentration will exceed 0.1 mg/l. This would constitute a loading of about 0.47 mole metal/kg resin (which includes the copper load, as the Feed Batch G treated contained 16 mg/l Cu).

Table VIII			
Cobalt mass balance: elution of column 2			
Composite			
Volume, litre	Co, mg/l		Co, mg
1528	16.7	Loaded	25,541
182	151	Elution	27,539
Per cent eluted			108%

Elution

After the two columns were loaded in a lead-trail configuration (Figure 5), they were separately eluted. The elution curve for Column 1 (lead column) is shown in Figure 6.

High zinc levels in the eluate are indicated in Figure 6. The zinc concentrations had, however, been reduced compared to previous elutions—this was done by eliminating a zinc plug in the inlet line and increasing the pre-wash acid strength. The required pre-wash/elution conditions to eliminate zinc from going to the eluate will be determined by laboratory tests.

Resin colour from columns 1 and 2

Both columns were eluted at a temperature of above 85°C and absolute pressure of 270 kPa (2 M H₂SO₄; 0.5 M SO₂), before washing and unpacking the columns.

The following observations were made when the columns were unpacked (note: the columns were operated up-flow):

- ▶ Column 1 (lead column for runs performed with feed solution containing some copper): Most (>90%) of the resin had a dark blue-green colour; At the top of the column there was a small patch of yellow-brown resin. The blue colour intensified to the bottom of the column and at the bottom some traces of blue-pink patches could be observed.
- ▶ Column 2 (operated in trail position): There was not

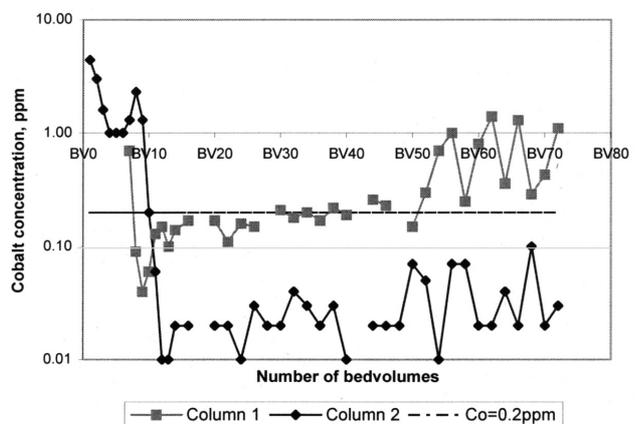


Figure 5—Cobalt removal curve for columns 1 and 2 in series at 70°C and loading rate of 0.2 l/(min.kg resin) from feed G

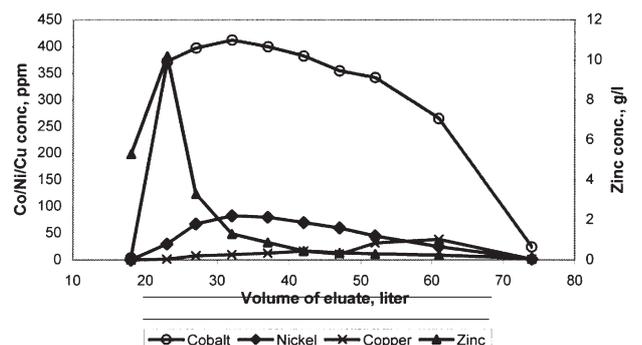


Figure 6—Metal elution curves for column 1 at 270 kPa absolute pressure, flow rate of 1.0 l/min, and at temperatures higher than 85°C

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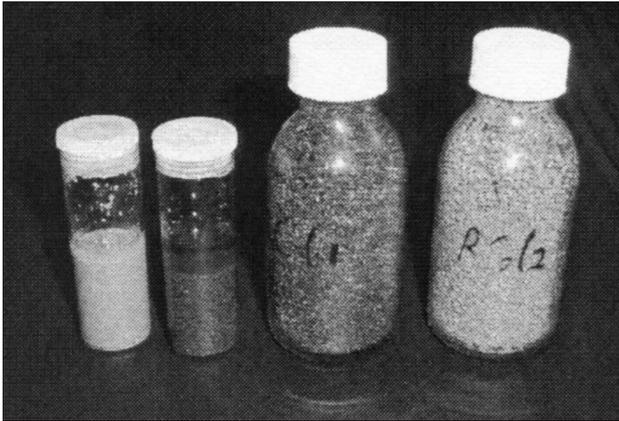


Figure 7—Colour of resin from columns—from left to right: copper-loaded SuperLig® 132 (reference sample), cobalt-loaded SuperLig® 138 (reference sample), copper-loaded eluted SuperLig® 138 (Zincor column 1), and eluted SuperLig® 138 (Zincor column 2)

any trace of blue anywhere to be found throughout the resin-column. The bulk of the resin had a yellow-brown colour. At the bottom of the column some slight traces of a pinkish-tint could be observed.

Figure 7 shows the colours of the resins taken from the columns. To allow comparison, two additional loaded resin samples are shown on the left, namely copper-loaded copper-SuperLig®132, and cobalt loaded cobalt-SuperLig®138. The samples shown are from left to right: (1) light blue loaded copper resin (SL132); (2) maroon loaded cobalt resin (SL138); (3) dark blue 'eluted' copper-loaded cobalt resin (SL138) from Zincor Column 1; (4) yellow-brown eluted cobalt resin (SL138) from Zincor Column 2.

Analysis of the SuperLig® 138 from Column 1 showed that 10.2% of the capacity was loaded with copper. This was done by contacting a known amount of resin with a specified volume of 4 M H₂SO₄ for an extended period of time in a stirred beaker. The solution was then analysed for eluted metals.

Comparing the state of the resin in the two columns, therefore confirms the need to effectively remove copper prior to cobalt MRT. Copper removal can reliably be done by using a SuperLig® 132 MRT-system. Alternatively, to compensate for possible copper slippage, the cobalt SuperLig® 138 column(s) can be periodically eluted with 4M H₂SO₄. Significant amounts of copper in the feed will, however, reduce the available resin capacity for cobalt removal.

Conclusions and recommendations

MRT pilot plant evaluation of cobalt removal from Zincor impure solution showed that:

Cobalt loading

- ▶ Ferric-iron was demonstrated to be an effective oxidant to facilitate the loading of the cobalt on the resin. The possibility of alternative oxidants is currently also being investigated.
- ▶ The viscosity of the concentrated zinc solution (>120 g/l Zn) impaired cobalt removal by MRT at ambient temperature. By increasing the temperature to between

60 and 80°C, the viscosity was lowered, and effective cobalt removal was demonstrated over 40 to 50 bed-volumes in a single column system (bulk removal). The loading rate was 0.2 l/(min.kg resin). The consistent removal of cobalt was confirmed by a number of repeat load cycles.

- ▶ When using two columns in series, the improved polishing effect was demonstrated. Extrapolation of the results indicates that cobalt would be removed for up to 200 bed volumes to levels below 0.1 mg/l in such a system (at a feed cobalt concentration of 11 mg/l).

Cobalt elution

- ▶ The cobalt can effectively be eluted from the resin by performing the elution with 2 M H₂SO₄ and 0.5 M SO₂ at a temperature of above 85°C, and absolute pressure of 250 kPa. Complete elution of the loaded cobalt was confirmed by cobalt mass balance.
- ▶ To eliminate the possibility of copper build-up on the resin, periodic 4M H₂SO₄ elutions (e.g. every 4th cycle) are recommended, or applying MRT for prior copper removal.

This pilot plant evaluation demonstrated the outstanding selectivity of the SuperLig® materials, which provides the benefit of removing cobalt at low levels from the concentrated zinc leachate stream. An MRT-system will simplify the purification circuit and provide increased high-grade zinc yields.

The use of MRT, a highly selective separation technology using specifically designed ligands, such as macrocycles, has been demonstrated to be an effective treatment process for removal of several ions that are commonly found in many hydrometallurgical circuits as well as in environmental treatment operations. Ions removed by MRT include base, heavy and transition metals, precious metals, halides, and alkali/alkaline earths^{4,9,10,12,14-17, 20}.

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Mintek appoints General Manager*

Mintek has appointed Professor Francis Petersen, currently Head of Chemical Engineering (and Acting Dean), at the Cape Technikon as General Manager of its Metallurgical Services.

Francis Petersen, M. Eng, Ph. D (Chemical Engineering), Pr. Eng, graduated from the University of Stellenbosch, and joined the Cape Technikon in 1992. He was promoted to Associate Director and Head of Chemical Engineering in 1995. He has produced 31 papers in international journals, 113 conference presentations, and supervised 20 post-graduate students.

In 1993 he was the recipient of the Ernest Oppenheimer Memorial Trust Award for research excellence. In 1998 he was funded by the National Research Foundation (NRF) on a study visit on technology transfer to Singapore. He also spent six months as visiting professor at Mintek in 1998. He

is an established researcher who enjoys international recognition in the fields of hydrometallurgy and environmental processing, with a focus on precious metal recovery using novel technologies such as ion-exchange, bacterial leaching, and agglomeration flotation.

Professor Petersen, who received the Researcher of the Year awards from the Cape Technikon in 1999, is an active member of various professional societies and committees, and he currently serves on a South African government commission investigating the treatment and disposal of mercury waste. ♦

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