2019 - THE CLEAN TEQ SUNRISE PROJECT – PILOT DEMONSTRATED UPGRADE OF PRESSURE ACID LEACH DISCHARGE THROUGH USE OF CONTINUOUS RESIN IN PULP TECHNOLOGY

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ABSTRACT

With the objective of further validating design data for the Clean TeQ Sunrise Project, during 2018 Clean TeQ Limited conducted an integrated pilot plant operation of the ore leach and continuous Resin in Pulp circuits. These pilot activities were conducted with ALS Metallurgy in their Balcatta facility, Western Australia. Pilot plant feed ore was prepared and composited from 60-tonnes of sample collected from the Clean TeQ Sunrise deposit in central New South Wales.

Pilot plant activities were split into two major programs;

1) ore preparation pilot including low charge attrition ball milling, fine screening for upgrade via quartz rejection, chromite rejection and feed thickening, and
2) integrated feed preparation pre-heating, pressure acid leach (PAL), pre-reduction (PR), partial neutralisation (PN), continuous resin in pulp (cRIP), tailings neutralisation and thickening.

Clean TeQ's cRIP pilot plant incorporated a 10-stage adsorption circuit in which the counter current contact of ion exchange resin with partially neutralised PAL discharge slurry achieved selective extraction of nickel, cobalt and scandium. The loaded resin, collected from adsorption Stage 1, was washed to remove entrained pulp by using a trommel screen coupled with an elutriation wash column. Loaded resin was desorbed using sulfuric acid. After desorption, the barren resin was washed for acid recovery, prior to conversion to a neutralised form to limit proton return to the cRIP adsorption circuit.

The integrated pilot activities were successful in demonstrating the ability of cRIP to achieve near-complete extraction of soluble nickel, cobalt and scandium from the slurry. Residual nickel and cobalt concentrations of less than 10 and 5 mg/L respectively were achieved, reflecting > 99.5% metal extraction in cRIP. The pilot campaign also demonstrated the selectivity of the ion exchange resin in recovering nickel, cobalt and scandium directly from the neutralised PAL discharge slurry, in preference to lower-affinity cations and impurity species including calcium, magnesium and manganese. The desorption process configuration proved particularly effective at scrubbing co-extracted magnesium and manganese from the loaded resin, resulting in a partially purified and enriched nickel, cobalt, and scandium liquor for downstream refining.

This paper outlines the successful integration of cRIP in the overall process for nickel, cobalt and scandium extraction and upgrading directly from acidic PAL leach slurry, along with the main operating parameters and resulting plant performance.

Keywords: Nickel, Cobalt, Scandium, Continuous Resin in Pulp, Ion Exchange, Pressure Acid Leach, Impurity Rejection, Pilot Plant, Sunrise.

INTRODUCTION

The Clean TeQ Sunrise Project is a greenfield development designed to produce battery-grade nickel and cobalt sulfates from the mining and treatment of nickel-cobalt laterite ore. The process also generates by-products scandium oxide and ammonium sulfate.

The project's Measured and Indicated Resources total 163 million tonnes averaging 0.54% Ni and 0.09% Co, with a Proved and Probable Reserve of 147 Mt at 0.56% Ni and 0.09% Co (2018 Ore Reserve). The mineralisation to be processed is goethite dominant, with the two major lithotypes defined as goethite and silicified goethite. The PAL feed grade for years 2 to 10 averages 0.87% Ni and 0.17% Co. The mineralisation contains minor clay content and is characterised by low magnesium head grades (< 1.0%) and correspondingly low overall gross acid consumption (life of mine between 200 to 300 kg/t, with years 2 to 10 averaging 238 kg/t).

The Clean TeQ Sunrise process flowsheet consists of conventional pressure acid leaching of the laterite ore followed by continuous resin-in-pulp technology (cRIP). Incorporating this technology into the hydrometallurgical process route, in place of counter current decantation, is consistent with prior development of RIP for treatment of leach residues and precipitation residues that have poor filterability or settling characteristics (Yahorava and Kotze, 2011).

Further, coupling cRIP with Clean TeQ's proprietary ion exchange desorption process enables Clean TeQ Sunrise to achieve an enhanced recovery of soluble nickel and cobalt, and process intensification through upgrade of the nickel and cobalt liquor concentration and reduction in impurity elemental concentration. This offers a simpler, lower CAPEX alternative to either mixed hydroxide or mixed sulfide precipitation process routes.

This paper demonstrates the impact of this circuit through results achieved during a medium-scale (100 L/h cRIP circuit feed rate) pilot program conducted with ALS Metallurgy in their Balcatta facility, Western Australia, during 2018.

BACKGROUND

Several companies considered processing of nickel laterite PAL slurry using RIP during the 2000s commodities boom, with Zainol (2005) ascribing this motivation to the technology's inherent characteristics:

- It is effective for recovering soluble metals from pulps with poor filterability;
- The selectivity of the resin allows for the extraction and upgrading of nickel and cobalt in the presence of calcium, magnesium and manganese;
- The overall nickel and cobalt recoveries on the plant can be improved; and
- It offers flexibility, allowing operating strategies to efficiently accommodate changes in the nickel and cobalt tenors of the feed pulp.

Yet, for many years, the Western mining industry was wary of resin loss, which can be caused by poor quality ion exchange resin or poorly matched resin mixing and movement equipment. Nowadays, however, significantly improved RIP grade resins are available, and ample guidance is available from gold and uranium recovery applications in the Former Soviet Union (FSU), where RIP has been in commercial application since 1958, to guide engineering and equipment selection.

Turning to the desorption process, it is known that conventional sulfuric acid desorption of base metals loaded onto iminodiacetic acid (IDA) resin in a straight column arrangement suffers from the following drawbacks (Littlejohn and Vaughan, 2014):

- Impurities are eluted together with valuable elements, complicating downstream processing; and
- Generally poor water balance and incomplete acid utilisation.

Therefore, Clean TeQ Sunrise has pursued deployment of the Sorption-Desorption column, also known as the SDK or U-column. The SDK is used extensively for insitu leach (ISL) resin desorption in the Kazakhstan uranium industry. Further background is available in Zontov (2008), Carr (2008) and Carr (2010).

PILOT PLANT OPERATIONS

In total, two Clean TeQ Sunrise Project pilot trials were undertaken at ALS Perth during 2018. The first pilot trial (Ore Preparation) involved PAL feed beneficiation and thickening processes with feed material beneficiated to size fractions of nominally -1 mm, -500 µm and -212 µm. The second pilot trial (Ore Leach) saw -212 µm beneficiated and thickened PAL feed slurry processed through PAL, pre-reduction (PR), partial neutralisation (PN), continuous resin in pulp (cRIP), continuous desorption in an SDK, tailings neutralisation (TN) and neutralised tailings thickening processes. This paper presents outcomes of the Ore Leach pilot trial.

The primary circuits piloted, and their sequence are presented in Figure 1.

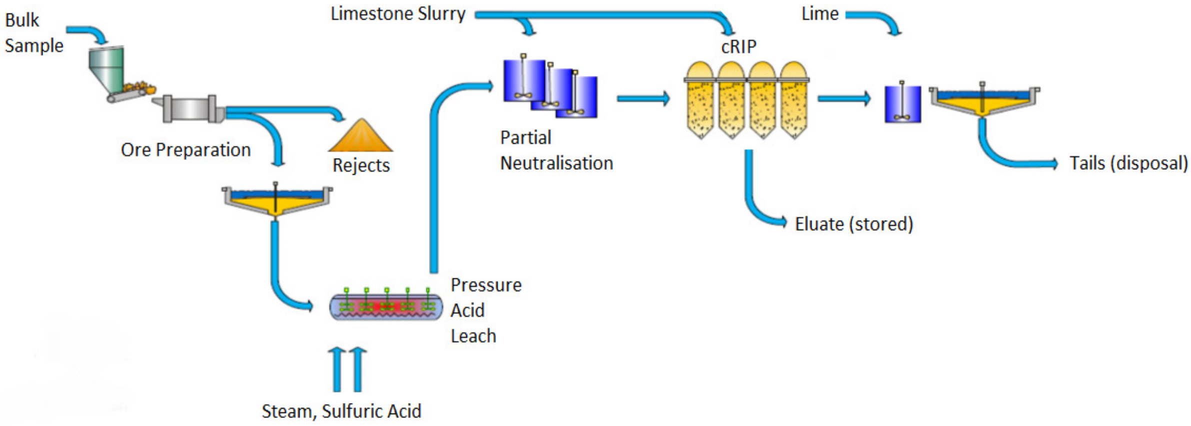

Figure 1: Clean TeQ Sunrise Ore Leach Pilot Plant Flowsheet

ORE PREPARATION PILOT PLANT DESCRIPTION AND RESULTS

The pilot ore blend was crushed and screened to -3.35 mm and homogenised prior to storage in 200 L drums. Drums were randomly selected and campaign-processed through the ore preparation circuit which comprised: a low-charge ball mill; a 500 µm aperture woven-wire vibrating mill discharge screen for rejection of coarse silica-rich particles; a HG10, 7-turn spiral separator for chromite rejection and potential PGM recovery to the spiral concentrate, and; a 212 µm aperture woven-wire vibrating circuit closing screen which received the spiral middlings and tails (see Figure 2).

Figure 2: Ore Preparation Circuit (ball mill, reject screen, closing screen are visible)

The undersize from the closing screen became the PAL feed. A total of 28.9 tonnes (dry) were processed across seven campaigns yielding 25.8 tonnes (dry) of Ore Leach pilot feed, with head grade shown in Table 1.

Table 1: PAL Feed Pilot Blend Head Assay

Ni %	Co %	Sc g/t	Al %	Ca %	Cr %	Cu g/t	Fe %	Mg %	Mn %	Si %	Ti %	Zn g/t
0.74	0.18	94	3.41	1.10	0.61	57	37.9	1.19	1.38	8.50	0.20	390

The Ore Leach pilot feed was thickened to a solids concentration of approximately 40 - 42% w/w and stored in forty-nine 1 m^3 intermediate bulk containers (IBC). Supernatant was returned to the milling circuit.

Perth tap water was used for the program as it closely matched site water. Minor salt additions were used for trace adjustments.

ORE LEACH PILOT PLANT DESCRIPTION AND RESULTS

The Ore Leach pilot trial was conducted as two discrete campaigns, each of approximately 2-weeks' duration. Prior to commencement of these pilot activities, batch testwork was conducted to establish initial operating conditions for the circuit. Setpoints were then refined during the pilot plant operation.

Pressure Acid Leach (PAL)

Leaching of the pilot ore blend was performed in a 6-compartment, horizontal titanium autoclave with an operating volume of 95 L, as shown in Figure 3. The total autoclave operating duration was 594 h, equally split over the two campaigns. This duration provided some stable operating periods of 4 to 6 consecutive days from which full cRIP performance could be quantified.

The autoclave operating temperature was 250 °C, with pressure approximately 4600 kPa g. The PAL feed was blended with elemental sulfur as a reductant at 0.6 kg S^0 / t dry solids, with concentrated sulfuric acid injected into Compartment 1 at 280 kg H_2SO_4 / t dry solids.

Figure 3: ALS Metallurgy's 95 L Titanium Autoclave

Extraction

Typical elemental extractions to PAL discharge liquor are shown in Table 2, with the corresponding Compartment 5 free acid concentration ~ 51 g/L and the calculated retention time being 62 minutes.

Table 2: Typical Pilot Pressure Acid Leach Extractions to Liquor

Element	Ni	Co	Sc	Al	Cr	Cu	Fe	Mg	Mn	Zn
Extraction, %	95.8	96.0	88.9	12.1	1.9	89.7	0.9	92.0	83.2	93.8

PAL Discharge

From a feed slurry at 37 – 38% w/w solids with d_{80} of 183 µm, the post-flash discharge slurry contained 35% w/w solids with d_{80} 11 µm, indicative of the bulk dissolution and subsequent precipitation occurring within the autoclave. The PAL feed slurry was lower than commercial design to achieve target compartment 1 solids target, reflecting the indirect heating within the pilot plant relative to the Clean TeQ Sunrise commercial plant design, which will operate at 45-48% w/w solids with direct heating, i.e. steam dilution. The corresponding average mass loss was calculated as 10 – 12%. Residue assays indicated the presence of 329 g/t Ni, 75 g/t Co and 11 g/t Sc.

Compartment 5 (C5) and post-flash discharge liquor compositions are presented in Table 3. The corresponding discharge liquor ORP was approximately 850 mV (vs Ag/AgCl), with all iron present as Fe(III) and majority of chromium present as Cr(VI).

Table 3: Typical Pilot Pressure Acid Leach Liquor Tenors

Element	Ni	Co	Sc	Al	Cr	Cu	Fe	Mg	Mn	Zn
C5, mg/L	4208	978	51	2742	70	--	1149	6544	6333	--
Post Flash, mg/L	5090	1163	62	3225	82	43	2335	8043	7757	238

-- not measured

For the piloted ore blend, we concluded that the elemental sulfur (S^0) addition could be marginally increased below 0.6 kg/t without Fe(II) formation.

Partial Neutralisation (PN)

The Partial Neutralisation circuit consisted of two steps: pre-reduction to lower the ORP and neutralisation to raise the pH.

Pre-Reduction (PR)

The objective of PR was to reduce the ratio of Cr(VI) to Cr(total) to < 0.15 (nominally < 10 mg/L Cr(VI), without excessive amounts of Fe(III) being converted to Fe(II). The PAL discharge slurry ORP was therefore “trimmed” using a 350g/L SMBS solution, dosed directly to the PAL discharge slurry via an agitated water jacketed tank held at 80°C.

As evident in Table 4 and Figure 4, operating with a PR discharge at 560 – 580 mV at 80°C provided a good reduction of Cr(VI), with minimal formation of Fe(II). Note that some dilution water (Perth tap water) was added to the PR vessel to offset evaporative losses and to ensure the downstream Partial Neutralisation circuit operated with a solid concentration comparable to that forecast at commercial scale.

Table 4: Typical Pilot Pre-Reduction Liquor Parameters

Parameter	PR Feed	PR Discharge
Fe(II) / Fe(tot)	~0.00	0.01
Cr(VI) / Cr(tot)	0.57	0.10
ORP (vs Ag/AgCl), mV at 80°C	780	587

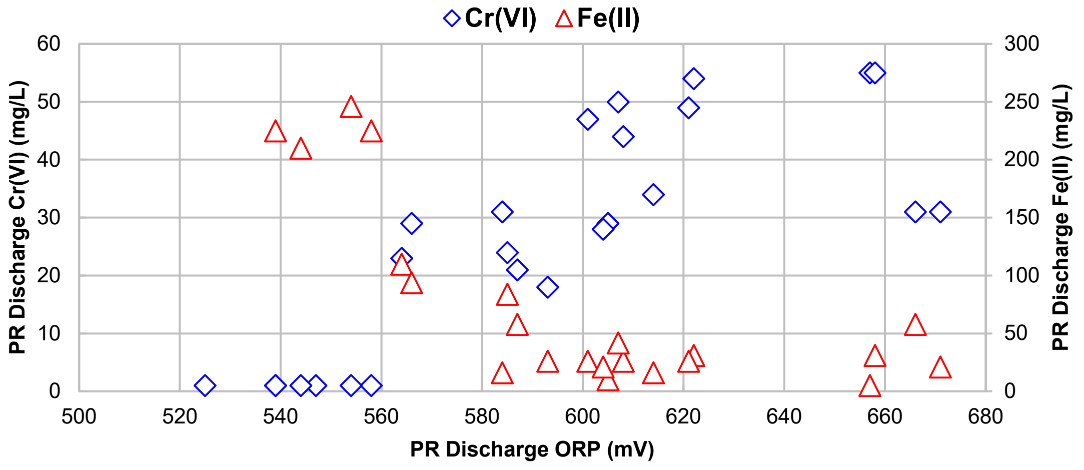

Figure 4: Pre-Reduction Discharge Tenor vs ORP

Partial Neutralisation (PN)

The objective of PN was to increase the pH of the pre-reduced PAL discharge slurry, and, through precipitation of metal hydroxides, achieve a PN discharge liquor low in concentrations of trivalent impurities, Al(III), Cr(III), Fe(III) without precipitating significant Ni or Co. These trivalent elements are adsorbed strongly onto IDA resin and their precipitation from the liquor phase during PN assists optimal performance through cRIP (i.e. prevents excessive competition with nickel and cobalt in cRIP adsorption). However, a trade-off against the potential co-precipitation of nickel and cobalt in the PN circuit exists.

To explore this trade-off, the PN terminal pH was incrementally decreased across the operating period, with results summarised in Table 5. Neutralisation was achieved via the addition of the Sunrise project 30%w/w limestone slurry to three agitated tanks in series at 80°C (see Figure 5). This limestone slurry was prepared using synthetic process water containing magnesium, manganese, and sodium sulfates. The tanks were also sparged with air to promote iron oxidation and to assist in purging of evolved carbon dioxide gas. A fourth equilibration tank provided a buffer between the PN and cRIP circuits.

Results summarised in Table 5 indicate that quantitative Fe(III) precipitation was achieved regardless of conditions tested. Significant precipitation of other trivalent impurities such as aluminium and chromium also occurred. Aluminium precipitation was highly sensitive to pH. Increases in nickel, cobalt and scandium co-precipitation with pH were also seen.

The full pilot data set was used to establish the relationships depicted in Figure 6, with discharge pH of 3.5 at 80°C nominated as a favourable operating point for the flowsheet. The distribution of limestone slurry (neutralant) to the various PN tanks and the resulting slurry pH at this operating point are summarised in Table 6. The corresponding PN discharge liquor composition is shown in Table 7.

Figure 5: Cascade of PN Jacketed, Agitated Tanks (limestone slurry ring main in foreground)

Table 5: Typical Pilot Partial-Neutralisation Liquor Parameters

Parameter	Period 1	Period 2	Period 3
Discharge pH (80°C)	3.75	3.50	3.40
Ni precipitation extent, %	6.6	4.0	2.4
Co precipitation extent, %	3.2	1.4	1.2
Sc precipitation extent, %	88	73	68
Al precipitation extent, %	89	81	74
Fe_{total} precipitation extent, %	99.7	99.8	99.9
Cr_{total} precipitation extent, %	93	92	89
Al, discharge mg/L	318	620	959
Fe_{total}, discharge mg/L	7	25	7
Cr_{total}, discharge mg/L	6	17	25

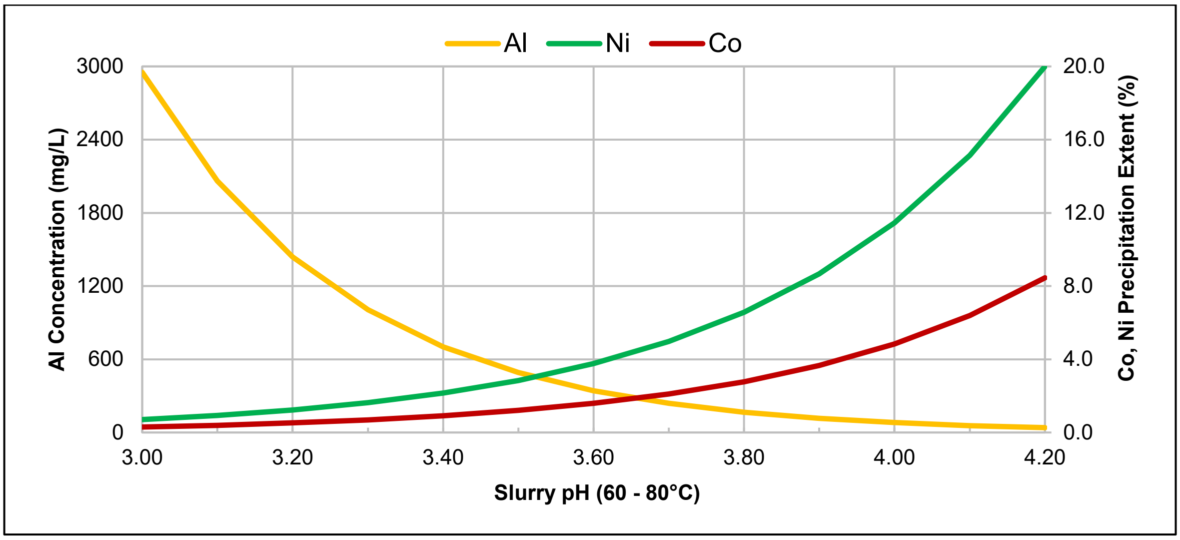

Figure 6: Partial Neutralisation Ni and Co Precipitation Extent vs pH

Table 6: Partial Neutralisation pH Profile

Parameter	Tank 1	Tank 2	Tank 3	Tank 4
Function	Neutralisation	Neutralisation	Neutralisation	Equilibration
Air sparge	Yes	Yes	Yes	No
pH (80°C)	1.9	2.9	3.5	3.5
Limestone addition, % split	60	24	16	0
Residence Time, min	123	120	118	90

Table 7: Typical Pilot Partial Neutralisation Discharge Liquor Tenors

Element	Ni	Co	Sc	Al	Cr	Cu	Fe	Mg	Mn	Zn
PN Discharge, mg/L	4002	970	17	932	25	11	7	7135	7134	178

Continuous Resin-in-Pulp (cRIP)

Adsorption Circuit Overview

The objective of the adsorption circuit was the quantitative recovery of soluble nickel and cobalt from the incoming PN discharge pulp. The total cRIP circuit operating duration was 583 hours, equally split over the two campaigns. Upstream circuit stability was excellent during Campaign 2, which enabled better tuning of the cRIP circuit. Accordingly, results presented in this section are predominately from a consecutive 7-day period (173 hours) during Campaign 2.

The adsorption circuit consisted of 10 air-agitated adsorption contactors (pachucas) through which PN discharge pulp passed counter-currently to ion exchange resin. Corresponding residence times were 60 min for pulp and 78 min for resin. Each contactor had an in-built static separation screen located at the top of the vessel. Contactor contents were continuously air-lifted onto this screen, with:

1) pulp reporting to undersize and advancing down the train sequentially from Contactor 1 to Contactor 10, and

2) resin reporting to oversize and advancing up the train sequentially from Contactor 10 to Contactor 1.

The combination of air-agitation and air-lifting provided intimate mixing of the ion exchange resin and pulp.

The resin concentration in individual contactors was periodically measured but allowed to fluctuate naturally without intervention, being generally 10 – 15% v/v. Regarding the resin itself, Purolite MTS9301 was used during this pilot campaign, being one of several suitable, commercially available IDA chelating resins being assessed for the project.

Figure 7: cRIP Adsorption (single adsorption contactor outlined in red; resin and pulp flow direction arrows are indicated at the top of the figure)

Pulp exiting Contactor 10, containing very low soluble nickel and cobalt concentrations, was passed through a trommel (rotating screen) to recover any stray resin beads. Resin exiting Contactor 1 was heavily loaded with metals, principally nickel and cobalt. This loaded resin passed through a trommel to drain entrained pulp, which was returned to the adsorption circuit feed tank. The loaded resin advanced to the desorption circuit.

Minor additions of limestone slurry allowed pH to be maintained at 3.8 – 4.0 through Contactors 1 – 6, with pH in the range 3.6 – 3.8 targeted in Contactors 7 – 10. Results showed that this pH profile is adequate. However, we believe that refining the pH profile will allow resin selectivity to be better exploited, reducing the impurity loading on the resin advancing to the desorption circuit.

Adsorption Liquor Profile

Slurry discharged from PN was combined with some cRIP recycle streams at the head of the adsorption circuit, namely entrained pulp separated from loaded resin and desorption scrub solution. Additionally, dilution water was used to regulate the solids concentration entering adsorption Contactor 1.

Quantitative extraction from liquor of nickel, cobalt and scandium was achieved across the adsorption circuit, with <10 mg/L Ni and Co residual in the spent pulp liquor. Extended periods of <5 mg/L Co were also achieved. Near-complete extraction of iron, chromium, copper and zinc from the liquor also occurred. The residual aluminium concentration (85 mg/L) was surprisingly high and is likely due to selectivity of the resin at the adsorption operating pH and the existence of the dissolved aluminium as a uni- or di-valent species. This is favourable since aluminium ions occupy a relatively large proportion of loading capacity on IDA resin given that adsorbed aluminium is trivalent and has a low atomic weight. Reflecting the IDA ion exchange resin selectivity, magnesium and manganese were extracted to a much lesser extent, despite being present at elevated levels in the feed pulp liquor.

Table 8: Average Pilot Adsorption Circuit Liquor Tenors

Element	Ni	Co	Sc	Al	Cr	Cu	Fe	Mg	Mn	Zn
PN Discharge, mg/L	4002	970	17	932	25	11	7	7135	7134	178
cRIP Spent Pulp, mg/L	9	8	0	85	<1	<2	<1	4867	5194	<2
Recovery from Liquor, (Eluate)/(Feed), %	99.6	98.7	~100	81.1	~100	~100	~100	0.01	0.80	99.3

The adsorption contactor liquor profile provides further insight into circuit operation. It is best visualised as the ratio of liquor tenor in a given contactor, relative to the liquor tenor in Contactor 1. The cRIP adsorption contactor liquor profile presented in Figure 8 illustrates the extraction of target metals and how the IDA resin selectivity is exploited to reject impurities. Reviewing this profile, it is seen from the low metal concentrations in Contactors 7-10, that the adsorption train was run conservatively to protect the near-complete extraction of nickel, cobalt and scandium from liquor. For the commercial plant operation, neutralised resin recirculation post desorption will be continuous, rather than as operated during pilot operation where routine sequenced batch recirculation (every 30 minutes at pilot scale) was operated. The net result of the continuous recirculation of neutralised resin, coupled with the high nickel cobalt adsorption selectivity of the resin, would allow for further enhancement of 1) the adsorption front, 2) nickel loading on the resin, and 3) potential to reduce the residual nickel and cobalt tenors in Contactors 9 and 10 further. Following are some comments on specific elemental adsorption extraction:

Ni:	Loading rapidly increases across Contactors 1 to 6
Co:	Loading rapidly increases across Contactors 1 to 6, yet extraction of Co “lags” Ni by approximately 1 to 2 contactors, (this is reflected in the overall Ni extraction being slightly above overall Co extraction).
Al:	The effective resin selectivity in this environment suggests that Al is crowded off by other higher affinity elements, presumably, Ni, Co, Sc and Zn, with Al tenor remaining high through Contactors 1 – 5, after which it rapidly decreased as Al was adsorbed onto the resin.

Mn:	Significant loading (30 – 40%) occurs in Contactor 10, however manganese loading on the resin is subsequently crowded off by Ni, Co, Sc and Zn. Mn concentrations in liquor were typically 15 – 20% greater than Contactor 1 for all contactors in which Ni, Co, Zn, Sc are at "tails" concentrations (see adsorption Contactors 6 – 9 in Figure 8).
Mg:	The only appreciable loading of Mg occurs in adsorption Contactor 10, where 10 – 15% is extracted onto the incoming fresh resin.

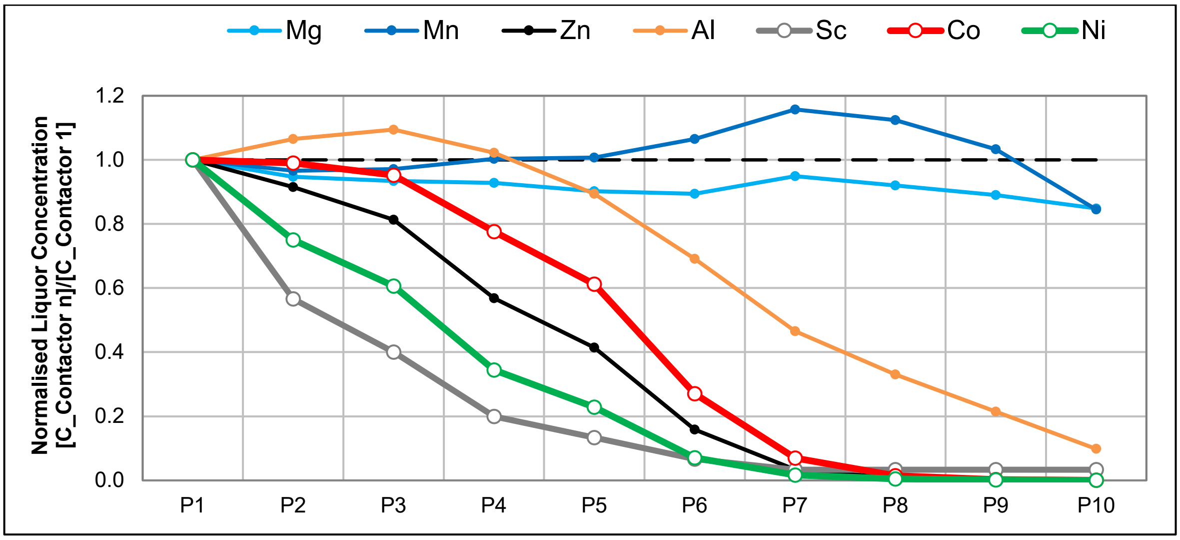

Figure 8: Normalised Adsorption Contactor Liquor Tenor Profile (from PS0023)

Despite nickel and cobalt having comparable overall adsorption extractions, the contactor in which the majority of loading of each element occurred differs considerably. From Figure 9, nickel adsorption primarily occurs in Contactors 1 to 6, whereas cobalt adsorption primarily occurs in Contractors 4 to 7. Also evident in Figure 9 is the importance of the final adsorption stages (Contactors 8 – 10), particularly in terms of achieving quantitative cobalt extraction. Note finally, that the incremental extraction of cobalt across Contactors 2 to 10 exceeds 100% given a net crowding-off of cobalt in Contactor 1 (by nickel).

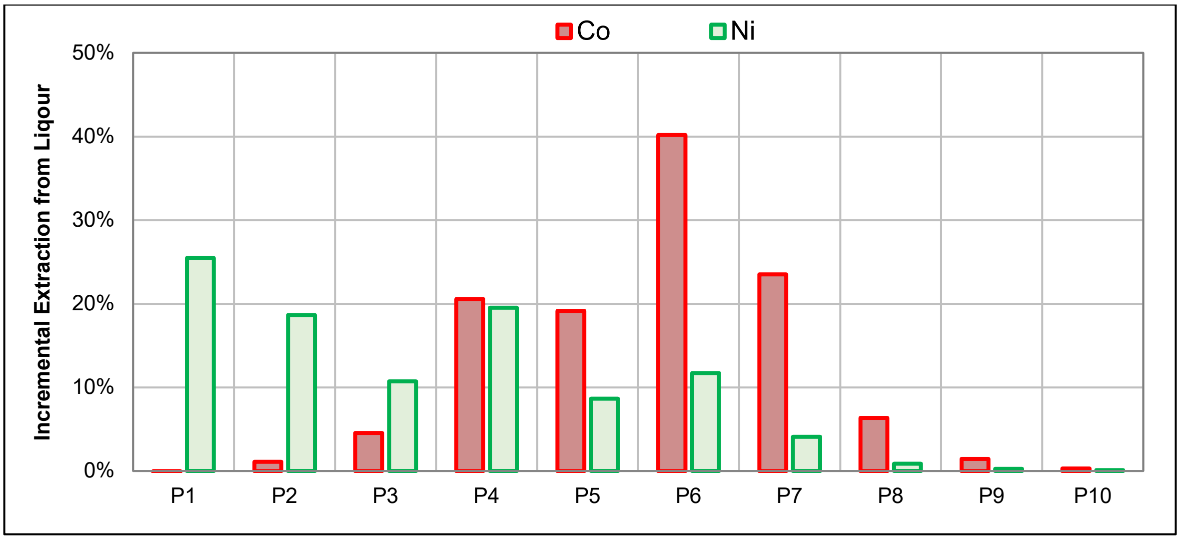

Figure 9: Incremental Extraction by Adsorption Stage (from PS0023)

Loaded Resin Composition

The loaded resin averaged 31.8 g Ni+Co/L wet settled resin in the selected period, with the primary impurities being aluminium and manganese. The broader composition is presented in Table 9, and all loadings are expressed as "g/L wet settled resin metal-form".

Table 9: Loaded Resin Composition (g/L wet settled resin)

Element	Ni	Co	Sc	Al	Cr	Cu	Fe	Mg	Mn	Zn
Loading, g/L	25.0	6.8	0.2	4.2	0.4	0.1	0.4	0.7	3.3	1.2
Loading, eq/L	0.85	0.23	0.01	0.47	0.02	<0.01	0.02	0.06	0.12	0.04

The total divalent and trivalent cation equivalents accounted for on the resin increased linearly from approximately 1.0 eq/L at Contactor 10 to 2.0 eq/L at Contactor 1, with nickel and cobalt occupying 60% of the total equivalence on the Contactor 1 loaded resin, and aluminium accounting for an additional 25%. Similar to the way liquor concentrations through the adsorption contactors were visualised, the loading of a given element can be normalised against the maximum (peak) loading of that element in the adsorption train. This approach is applied in Figure 11, showing peak loadings of nickel, scandium, copper, as well as chromium and iron (not shown in Figure 11) peak occurring in Contractor 1, zinc occurring in Contactor 3, cobalt in Contactor 4, aluminium in Contactor 6, and manganese and magnesium both occurring in Contactor 10.

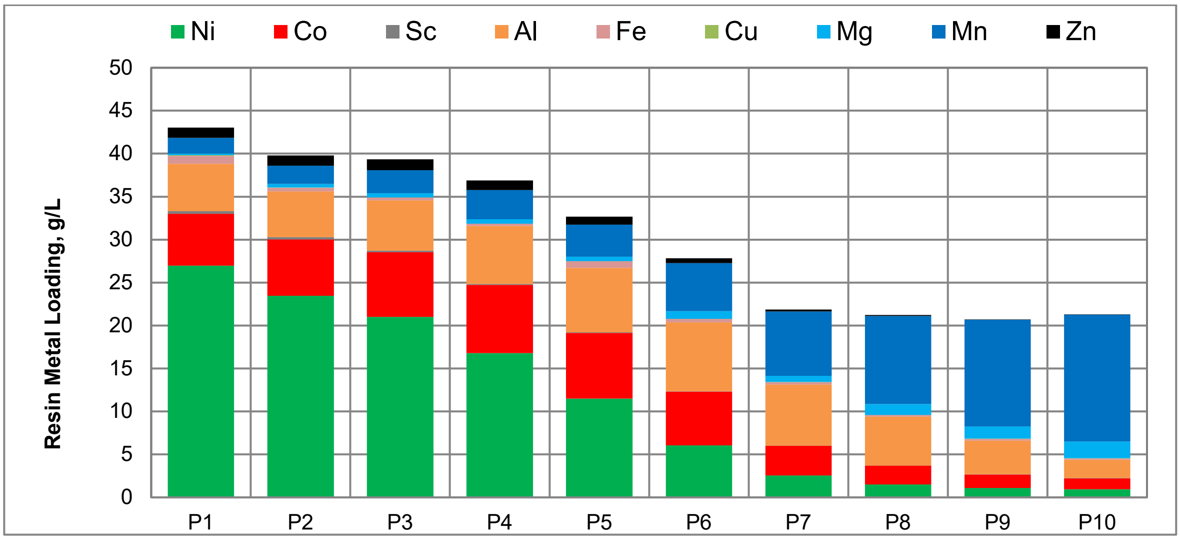

Figure 10: Resin Loading Profile (from PS0025)

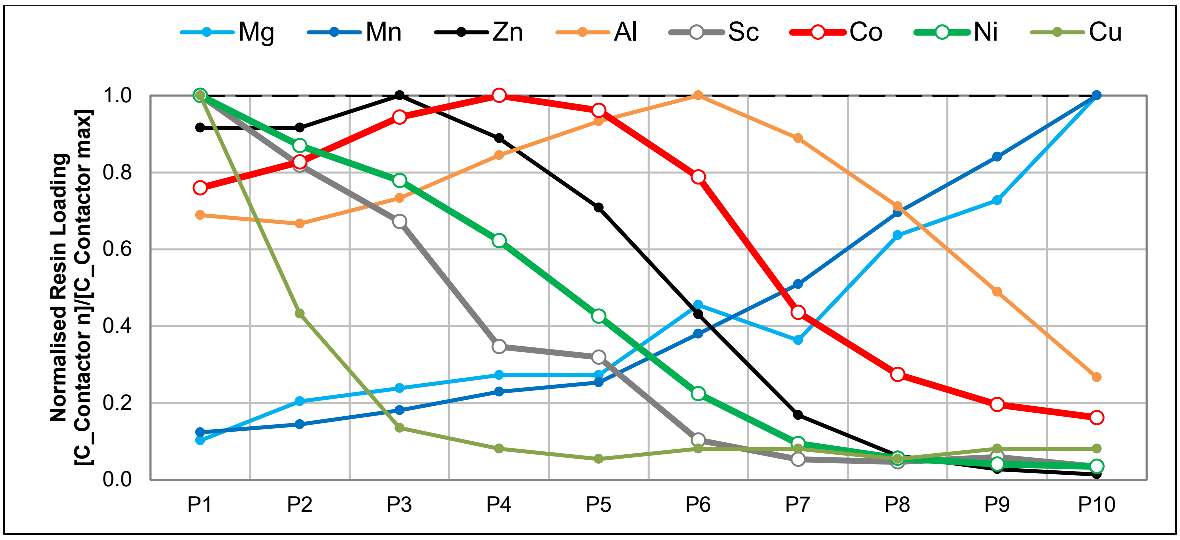

Figure 11: Normalised Resin Loading Profile (from PS0025)

Resin Selectivity

The resin selectivity in this circuit will depend upon factors such as pH and the relative concentrations of the various metals. Reviewing the profiles in Figure 8 and Figure 11, the demonstrated selectivity of the adsorption circuit was:

Cu, Fe(III), Sc > Ni > Zn > Co > Al > Mn > Mg > Na

Solid Residue Re-Leach

Reviewing the metal units advancing to the desorption circuit on the loaded resin, there is evidently more iron and scandium than can be accounted for by the dissolved metal concentration in the liquor entering adsorption Contactor 1. This is attributed to re-leaching some metals from the solid phase, with re-leach extents, as a percentage of the precipitation occurring in the PN circuit, calculated to be: scandium 60%, nickel 30%, cobalt 40%. As mentioned, iron re-leach is also occurring, but to a very small extent, relative to the bulk precipitation occurring in PN. Applying a silicon-tie to solid residue assays determined during a plant survey, this re-leach can be seen through the normalised residue metal concentrations in Figure 12. The behaviour was also observed by Nicol and Zainol (2003).

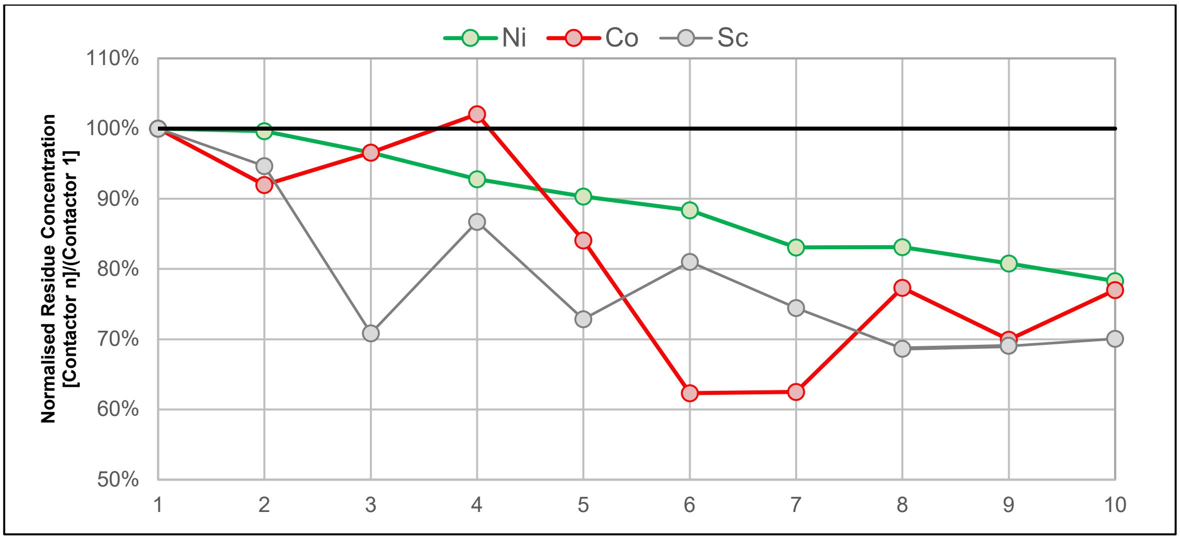

Figure 12: Normalised Residue Metal Concentrations (from PS0027)

Desorption Circuit Overview

The objective of the desorption circuit was the generation of an enriched, upgraded nickel sulfate and cobalt sulfate liquor. The results presented below focus on the two ion exchange processes occurring in the desorption circuit. Firstly, loaded resin is contacted with sulfuric acid, causing hydrogen ions to exchange for metal ions, liberating metal ions into solution. Secondly, barren resin is contacted with a dilute neutralising stream, with calcium, magnesium and manganese ions exchanging for hydrogen ions.

Resin Desorption

Desorption of the loaded IDA resin was performed in a SDK. Each hour, discrete batches of resin incrementally advanced through the SDK, with fresh parcels of loaded resin entering the upper left of the column, and fully desorbed resin exiting the upper right. Between these periodic resin movements, eluant (150 g/L H_2SO_4) was applied to the upper right of the column, flowing counter-currently to the resin. The main process streams are depicted in Figure 13a.

As the sulfuric acid eluant passed down the right leg (desorption side), hydrogen ions were exchanged for metal ions, with the advancing liquor becoming increasingly rich in metal ions. A portion of this liquor was extracted from the base of the vessel as eluate, typically having pH 1.5 – 1.6 (free acid of 4 – 6 g/L). The balance of the liquor was permitted to advance up the left-hand side (re-adsorption side), with residual acid and metal ions in the liquor providing a scrub function, exchanging for lower-affinity metal ions such as magnesium, manganese, calcium and aluminium on the resin.

The scrub solution exited the top left of the vessel with a pH typically 3.7 – 3.8 and, given its residual nickel and cobalt content (~4% of the SDK loaded resin feed Ni/Co concentration), was returned to the adsorption feed tank. This scrubbing action provides several benefits. Firstly, it rejects a portion of impurities, resulting in a partially purified eluate. Secondly, the re-adsorption of higher-affinity metal ions in the scrub zone allows eluate enrichment relative to loaded resin composition. Thirdly, the re-adsorption zone helps stabilise eluate quality in the face of loaded resin composition fluctuations.

These benefits are evident in the liquor composition and elemental deportments presented in Table 10 and Table 11.

Deportment of magnesium from PN discharge to eluate was ~0.01%, with manganese at ~0.8%. For the Clean TeQ Sunrise flowsheet, the ability to almost quantitatively reject manganese and magnesium is particularly beneficial given the high Co:Mg, Co:Mn, Ni:Mg and Ni:Mn ratios in the respective battery grade sulfate products.

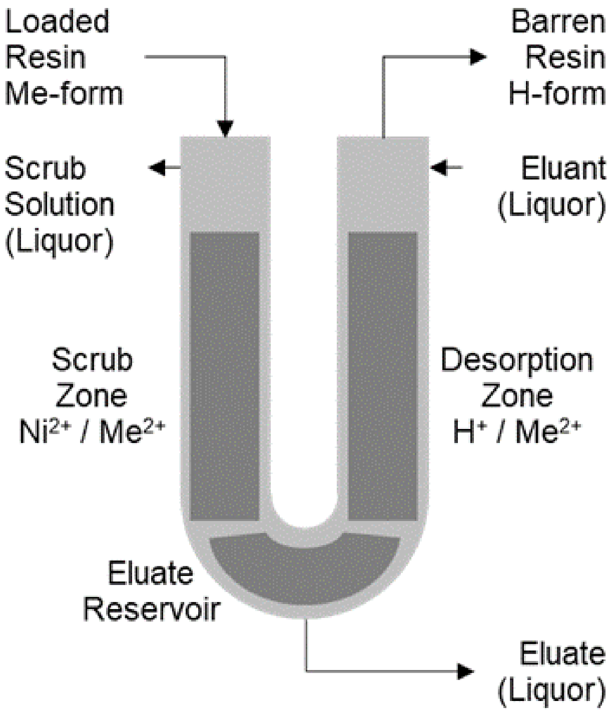

Figure 13a: Sorption Desorption Column (SDK, or U-Column) Primary Streams
Figure 13b: Pilot SDK (as fabricated) and Operating

Table 10: Typical Pilot Desorption SDK Liquor Tenors

Element	Ni	Co	Sc	Al	Cr	Cu	Fe	Mg	Mn	Zn
Eluate, mg/L	29776	6503	228	4124	73	76	343	5	448	1271
Scrub Solution, mg/L	524	399	1	563	2	0	0	218	1641	52

Table 11: Typical Pilot Desorption SDK Elemental Deportments

Element	Ni	Co	Sc	Al	Cr	Cu	Fe	Mg	Mn	Zn
Eluate, %	98	94	100	88	98	100	100	2	22	96
Scrub Solution, %	2	6	0	12	2	0	0	98	78	4

Resin Conditioning

Following desorption, entrained eluant (150 g/L H_2SO_4) was recovered from the barren resin in a resin washing step, before the resin entered a set of neutralisation contactors. As a process objective, resin neutralisation sought to convert the protonated IDA resin to a low-affinity cation form, thereby greatly reducing pH decreases through the adsorption train. If the resin was left in the protonated form, the pH in the adsorption contactors would drop as metal ions in the liquor exchanged for hydrogen ions on the resin, increasing the potential for dissolution of aluminium, chromium and iron precipitates in the cRIP residues.

During the pilot operation, this resin neutralisation conversion was achieved by contacting the resin with a 10% w/w limestone slurry, made from Sunrise project limestone in synthetic process water (i.e. addition of sulfate salts to achieve anticipated elevated levels of manganese and magnesium, and lesser amounts of calcium and sodium). Operating in a pH range of 5 – 6 at 25°C, the resulting neutralised resin composition is detailed in Table 12. The conversion extent from protonated to divalent metal form was approximately 50%, allowing the pH profile through adsorption to be maintained in the desired range with only minor amounts of limestone slurry addition for pH control. On an equivalence basis, calcium, magnesium and manganese occupied a comparable amount of the neutralised resin's loading capacity.

Table 12: Neutralised Resin Composition (g/L resin)

Element	**Ca**	**Mg**	**Mn**	**Na**
Loading, g/L	5.9	3.8	8.6	0.1
Loading, eq/L	0.29	0.31	0.31	<0.01

Resin Elemental Mapping

During the pilot campaigns, resin samples from the circuit were examined by semi-quantitative elemental distribution scanning (EDS) using a scanning electron microscope (SEM). The technique reveals the distribution of elements within the ion exchange resin bead, thereby providing an enhanced understanding of the mode of elemental adsorption and crowding off within the Clean TeQ Sunrise cRIP adsorption circuit.

The images below provide a relative proportion of elemental loading, not absolute loadings, with the brighter colouration indicating higher elemental concentrations within the resin. The images provided in Figure 14, show the behaviour of IDA resin in exchanging lower affinity cations Ca^{2+}, Mg^{2+} and Mn^{2+}, for the higher affinity Ni^{2+} as the resin advances up the adsorption circuit.

Further explanation for the EDS images follows;

- In the neutralisation contactor, barren H^+ form resin (post desorption and stripped of Ni^{2+}) is then converted to the neutralised form using $CaCO_3$ in process water, displacing the majority of H^+ and loading Ca^{2+}, Mg^{2+} and Mn^{2+} onto the resin bead.

- In adsorption contactor 8, the intensity (presence) of Ca^{2+}, Mg^{2+} and Mn^{2+} is beginning to be diminished as the lower impurity cations begin to be replaced with other higher affinity cations in the leach liquor, such as Al^{3+}. However, as nickel concentration is low in adsorption contactor 8 liquor, the relative presence of Ni^{2+} on the resin isn't detected. Co^{2+} is however present on the resin at this stage (not displayed).

- In adsorption contactor 6, the adsorption of Ni^{2+} is prevalent showing the significant increase in intensity at the expense of the displaced lower affinity impurity cations Ca^{2+}, Mg^{2+} and Mn^{2+}.

- In adsorption contactor 2, the higher affinity Ni^{2+} has increased intensity (i.e. loading) whereas the lower affinity cations are near fully displaced.

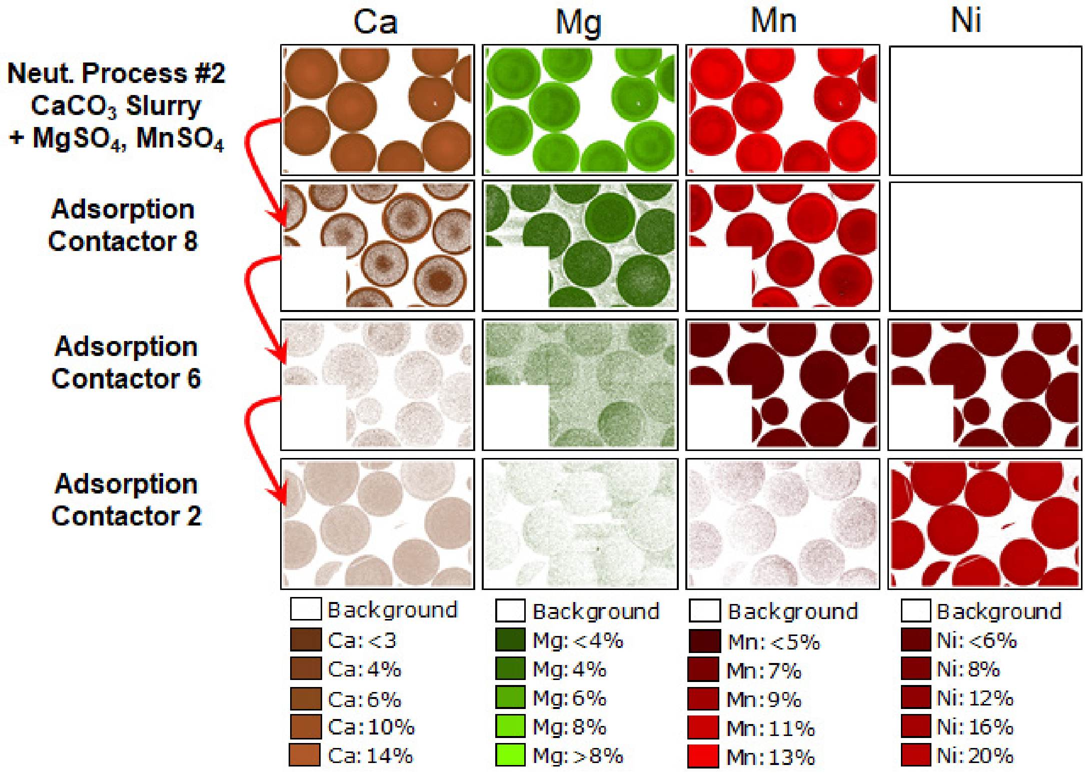

Figure 14: SEM-EDS Analysis of Resin Adsorption

Tailings Neutralisation

The bulk of the spent pulp exiting cRIP adsorption Contactor 10 was neutralised in a batch-wise fashion to pH 6.5 at 25°C using hydrated lime slurry, followed by dewatering in a filter-press and subsequent disposal. Commercially the neutralised tailings slurry will be deposited in the Tailings Storage Facility (TSF). A representative portion of this cRIP spent pulp was used for a range of neutralisation tests, flocculant screening, vendor thickening testwork, TSF characterisation and evaluation of rheological properties.

CONCLUSIONS

The successful completion of the recent 2018 Clean TeQ Sunrise pilot plant activities has validated the process design criteria for the current phase of Front-End Engineering Design. Quantification and demonstration of the benefits associated with the Clean TeQ cRIP technology for processing of nickel-cobalt laterites in comparison to the conventional nickel laterite hydrometallurgical PAL/CCD flowsheet was also made possible. These benefits include:

Enhanced Recovery

Extraction of nickel and cobalt across cRIP adsorption has been demonstrated to be above 99%, which is 3-4% higher than what is conventionally achieved in operational CCD circuits. Even a well-run 7-stage CCD circuit is frequently unable to achieve >96% soluble metal recovery leading to to > 50-100 mg/L Ni and >10 – 20 mg/L Co in the tailings.

It was shown that utilising cRIP technology, nominal residual spent pulp target metal tenors of <10 mg/L will be achieved in a stable circuit, with the ability to drive residual metal concentrations even lower.

Furthermore, the ability to target incremental metal extraction from the PN co-precipitated solid phase represents accretive nickel and cobalt recovery benefit (~0.7% Ni and 0.5% Co). Of significance to the Clean TeQ Sunrise flowsheet, however, is the co-current extraction of scandium from the cRIP feed residue (overall 40% Sc total recovery from ore is achieved).

Upgrade and Purification of Refinery Feed

Concentrations of major elements present in Clean TeQ Sunrise process streams are listed in Table 13.

Table 13: Demonstrated Upgrade Ratio of Refinery Feed Using cRIP Technology

Element	Ni	Co	Sc	Al	Cr	Cu	Fe	Mg	Mn	Zn
PN Discharge, g/L	4.00	0.97	0.02	0.93	0.03	0.01	0.00	7.14	7.13	0.18
Resin Loading, g/L	25.03	6.79	0.19	4.22	0.39	0.08	0.38	0.71	3.29	1.16
Eluate, g/L	29.60	6.54	0.24	4.15	0.07	0.08	0.33	0.005	0.47	1.27
Upgrade Ratio	7.4	6.7	13.4	4.4	3.0	7.6	46	0.001	0.075	7.2

The 2018 Clean TeQ Sunrise pilot data demonstrated that the eluate quality leads to a near six-fold increase in concentration. For an equivalent nickel / cobalt metal content, this will reduce the advancing volumetric flowrate to downstream processing relative to what could be achieved with alternative CCD technology. The net impact should be a substantial decrease in both installed capital and operating costs (reagent consumption) for the subsequent Fe/Al precipitation step.

Impurity Rejection

The configuration of the circuit – specifically the counter-current cRIP adsorption train and desorption using SDK – coupled with the selectivity of the resin enables the targeted extraction of nickel, cobalt and scandium over lower impurity species metals. The demonstrated selectivity achieves rejection of major impurity metals magnesium and manganese from the PAL discharge leach liquor.

The resultant impurity ratio relative to the nickel tenor can be seen in Table 14. The data also shows the ratio of scandium to nickel increases from cRIP feed to eluate, as expected due to higher affinity. This is also true for major competing impurity cations copper and iron, as Fe(III). Conversely, the ratio of lower affinity metals, including cobalt, decreases. The decrease in cobalt is minor and will reach equilibrium in stable operations. Magnesium and manganese are near fully rejected from the eluate.

The cRIP eluate quality generated through use of the SDK confirms the eliminated requirement for an intermediate precipitation step such as mixed hydroxide precipitation (MHP) or mixed sulfide precipitation (MSP).

The resultant elimination of this intermediate precipitation step enables Clean TeQ to advance a reduced impurity liquor directly to the nickel cobalt separation and refining processes.

Table 14: Impurity Deportment Post-PAL and Recovered to Eluate

Element	Co:Ni	Sc:Ni	Al:Ni	Cr:Ni	Cu:Ni	Fe:Ni	Mg:Ni	Mn:Ni	Zn:Ni
PAL Discharge Post Flash	0.228	0.012	0.634	0.016	0.008	0.459	1.580	1.524	0.047
PN Discharge	0.242	0.004	0.233	0.006	0.003	0.002	1.783	1.783	0.044
Eluate	0.218	0.008	0.139	0.002	0.003	0.012	0.000	0.015	0.043

The overall net benefit that cRIP technology introduces to the Clean TeQ Sunrise Project, and potentially the broader nickel laterite industry, is the ability to cost effectively maximise target metal recovery and deliver an upgraded and purified liquor well-suited to downstream refining directly to battery-grade sulfate products.

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