

RECOVERY OF COBALT FROM POLYMETALLIC CONCENTRATES - NICO DEPOSIT, NWT, CANADA - PILOT PLANT RESULTS

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ABSTRACT

This paper provides an overview of the NICO deposit process development work throughout the past decade, with emphasis on the latest results on the recovery of cobalt. The results presented in the paper were produced by an integrated pilot plant carried out in 2008. The NICO "IOCG" (iron oxide – copper - gold) type deposit is located in the Mazenod Lake district near the Snare hydroelectric complex, approximately 160 km northwest of Yellowknife, Northwest Territories, Canada. Cobalt is recovered by flotation into a concentrate wherefrom it is leached by pressure oxidation followed by solution purification (involving copper solvent extraction and iron removal), ion exchange, precipitation and electrowinning. The pressure oxidation occurs with selective rejection of arsenic and iron allowing environmentally acceptable tailings disposal as ferric arsenate. The process relies entirely on commercially proven unit operations and equipment and the integrated pilot exercise proved its technical feasibility. This paper summarizes the results of the purification of the cobalt solution and its electrowinning as cathode.

INTRODUCTION

The NICO cobalt-gold-bismuth-copper deposit is located in the Mazenod Lake district near the Snare hydroelectric complex, approximately 160 km northwest of Yellowknife, Northwest Territories. The mine development is planned based on a reserve of 21.8 million tonnes of ore averaging 0.13% Co, 0.16% Bi, and 1.08 g/t Au that will be mined using a combination of open pit and underground methods within a lower grade overall resource. These reserves, which support a minimum 15-year mine life at a production rate of 4,000 tonnes per day, include Underground Proven and Probable Mineral Reserves of 1,204,000 tonnes, grading 5.07 g/t gold, 0.14% cobalt and 0.19% bismuth and Open Pit Proven and Probable Mineral Reserves of 20,613,000 tonnes, grading 0.85 g/t gold, 0.13% cobalt and 0.16% bismuth.

The economic metals occur throughout the NICO deposit in concordant and discordant sulphiderich fractures and disseminations. Petrographic and electron microprobe analyses indicate that the cobalt is contained predominantly within a solid solution series between arsenopyrite and

cobaltite, with minor cobaltian loellingite. Bismuth occurs as native bismuth, bismuthinite, and bismuth tellurides, while copper is contained within chalcopyrite. Gold occurs as microscopic grains of native gold from less than 1 to greater than 100 micrometers in size, and as various gold-bismuth-antimony-tellurium alloys. Gold has been detected as inclusions within the sulphide minerals and as discrete grains on sulphide and silicate mineral grain boundaries.

PROCESS FLOWSHEET DEVELOPMENT HISTORY

Extensive bench scale flotation and hydrometallurgical testwork was carried out during the past decade on various samples as they become available during the deposit definition drilling campaign. The work focused on the recovery of cobalt, bismuth and gold contained in

typical NICO concentrates (1-5). Initial pressure oxidative (POX) leaching testwork was conducted in 1998 on the first NICO bulk cobalt-bismuth concentrate sample produced by bench and locked cycle flotation tests at SGS Lakefield. This concentrate assayed about 2.2 % Co, 13% Bi, 2.8% Cu, 11% As, 24% Fe, 22% S and 300 g/t Au (3). Cobalt leaching efficiency ranged from 93 to 96% when the pressure oxidation was conducted in the temperature interval from 140 to 180°C, and for a residence time of two hours. TCLP leachate tests carried out on various final pressure oxidation tailings assayed from 1 to 7 ppm As, classifying these tailings as a "Registerable Waste", as defined by Regulation 347.

Further work on solution purification and cobalt recovery proved that the sulphur dioxide-oxygen system was the most efficient for the precipitation of iron and arsenic from the cobalt POX pregnant leach solution. Under optimum conditions, about 99% of the arsenic and iron were precipitated without significant cobalt loss, producing final solutions assaying <1 mg/L As and <8 mg/L Fe. The efficiency of cobalt solvent extraction from the purified solution ranged from 91 to 98%, the co-extractions ranged from 88 to 100% for copper, from 28 to 33% for magnesium, from 6 to 7% for calcium and from 96 to 98% for zinc. Copper precipitation by using (SO₂/S) and (Na₂S) systems reduced the concentration of copper to less than 1 mg/L Cu in the purified cobalt electrolyte. Comparative base line precipitation test results indicated that the limits set for the specifications for most commercial cobalt carbonate products were met or exceeded.

Included in the previous bench-scale investigation was extensive bismuth recovery work that established a conceptual flowsheet based on ferric chloride leaching followed by counter-current iron powder cementation to produce a high-grade bismuth intermediate.

The initial testwork led to a conceptual flowsheet to recover cobalt, bismuth, gold and copper from the NICO concentrates. This was possible because the viability of bulk sulphide flotation was demonstrated which in turn allowed concentration of the pay metals (cobalt, bismuth and gold). Further refinements to the flotation flowsheet allowed for establishment of the conditions for selective recovery of cobalt and bismuth into their respective sulphidic concentrates under robust, reproducible and so commercially applicable conditions. At this point in time the hydrometallurgical investigations were initiated aiming to recover the pay metals from their concentrates. According to the initial hydrometallurgical flowsheet, copper was treated as a recoverable metal and its recovery was envisaged by copper solvent extraction-electrowinning (CuSXEW) directly from the pressure oxidation leach solution. Iron and arsenic

were removed from the de-copperized pressure oxidation leach solution by sulphur dioxide-oxygen precipitation. Cobalt was recovered from the de-copperized and purified pressure oxidation leach solution by solvent extraction (SX) followed by carbonation (i.e. precipitation as cobalt carbonate) followed by subsequent re-leach (RL) and electrowinning (EW). At the time, the copper option was considered with the assumption of the availability of copper concentrate produced from the neighbouring Sue Dianne deposit, also owned by Fortune Minerals, which contains a higher grade copper resource than NICO. Gold was recovered by carbon-in-leach (CIL).

Further process development efforts on the NICO project have been carried out over several phases during which the overall project objectives were at times, redefined. This process involved certain changes in the original flowsheet established in the late 90's as described above. For example, the recovery of bismuth as a separate product was not always the preferred option of the project owner and the decision was driven by the marketability of the potential product. This approach changed following the latest feasibility study (2007) indicating significantly improved project economics for recovering the bismuth in the form of an advanced intermediary or even pure metal. This new direction triggered a quite successful metallurgical effort towards selective flotation and subsequent hydrometallurgical work on the re-defined feed samples. The feasibility study mentioned above also concluded that the copper grade was too low to be worth pursuing economically until the nearby Sue Dianne project would come on line. Therefore, the newest (i.e. current) flowsheet version treats the copper as an impurity. At the same time, provisions have been made to retain some of the 2008 NICO pilot autoclave pressure oxidation leach solution for possible testwork on copper solvent extraction-electrowinning (CuSXEW) at a later time.

Extensive investigative testwork results indicated that pressure oxidation of the selective bismuth concentrate followed by brine leach (without ferric chloride)

recovered 95% of the bismuth and that cyanidation of the brine-leached residue recovered about 95% of the gold. This critical change was implemented in the 2008 hydrometallurgical flowsheet tested at pilot scale.

Several refinements were implemented with respect to the cobalt recovery flowsheet, primarily with respect to advanced purification aimed at removing the traces of copper, nickel, zinc, etc. Technical solutions tested included ion exchange and selective precipitation. These efforts allowed for the elimination of the copper solvent extraction step prior to its precipitation as carbonate. The multi-year process development testwork carried out in conjunction with the continuous deposit definition and mapping efforts has resulted in a flowsheet deemed sufficiently robust to be "pilota-ble". The objective of this paper is to highlight the final results of the 2007/2008 pilot campaign with emphasis on cobalt solution purification and subsequent recovery by electrowinning. The exercise was complex and it included a significant amount of preparatory and post-pilot laboratory testwork; details of which are beyond the scope of this paper, which focuses primarily on the key results.

2007/2008 HYDROMETALLURGICAL PILOT PLANT FLOWSHEET

The hydrometallurgical flowsheet subjected to pilot testing in 2008 is illustrated in Figure 1. The bismuth and cobalt concentrates are subjected to continuous pressure oxidation in two different autoclaves and under similar conditions (180°C, 1 hour residence time, ~ 13% wt. solids feed, as required to ensure autothermal conditions. The pressure-oxidized slurries from each stream (i.e. Bi and Co) are subjected to liquid-solid separation consisting of settling-thickening followed by filtration and displacement wash. The washed bismuth POX residue is directed to bismuth recovery, where it is brine-leached for two hours, 80°C, 200 g/L NaCl, ~27 g/L H₂SO₄, 5% wt. solids feed. The discharge is filtered, washed and directed to the gold recovery circuit. The combined pregnant and washed solution

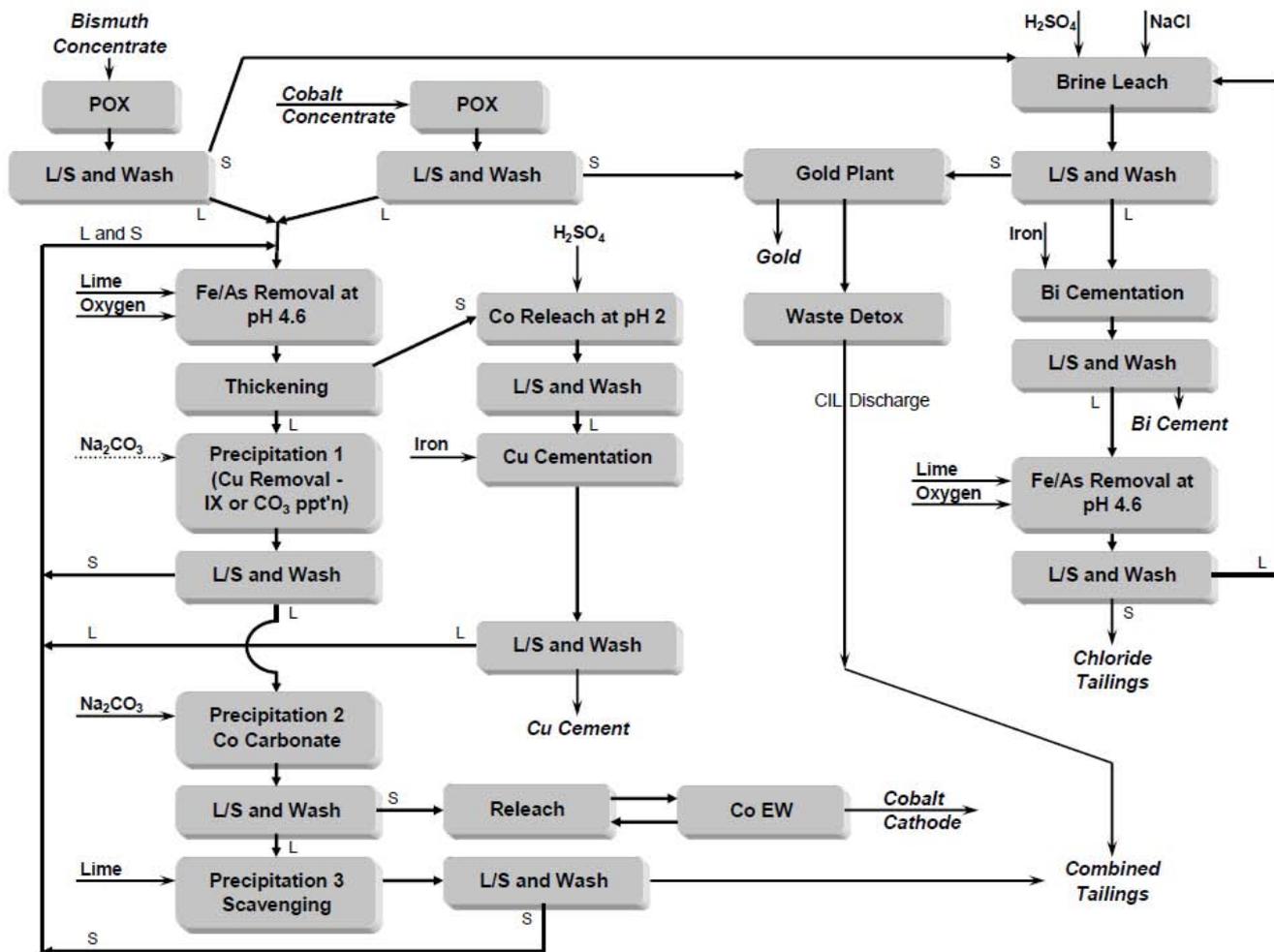


Figure 1 - Hydrometallurgical pilot plant flowsheet

is subjected to cementation with iron at 50°C, two hours residence time. The discharged cement is filtered, washed and dried. Iron and arsenic are removed from the cementation barren solution under conditions similar to those in the cobalt circuit. The underflow discharge is thickened. The underflow was stored in drums in lieu of the chloride tailings pond. The overflow is re-acidified with sulphuric acid and its chloride tenor is adjusted with NaCl, and then redirected to the brine leach. The bismuth recovery circuit will be the subject of a future publication.

The pregnant and wash discharge solutions resulting from the cobalt and bismuth autoclave-thickener-filter sequences are combined and directed to iron-arsenic removal (FeAsRe). The FeAsRe circuit operates at 50°C, 5 hours residence time and at a discharge pH of 4.6. The oxidising agent is oxygen. The resulting acidity is neutralized continuously using lime slurry. The

FeAsRe circuit discharge is thickened. The underflow is subjected to acid releach at pH ~ 2 with sulphuric acid at 50°C for 10 min reaction time. The acid-releached residue is washed and directed to the tailings pond. The acid releach pregnant solution and wash liquor are combined and sent to copper cementation at 50°C for one hour reaction. The product pulp is filtered and washed. The combined barren and wash is recycled to the FeAsRe circuit.

Two variants of the downstream processing of the FeAsRe thickener overflow were tested, differentiated by the method of removing the residual copper from the FeAsRe discharge overflow. Version "A" tested selective precipitation of copper, using 100 g/L sodium carbonate solution. The flow of sodium carbonate is actuated (on/off) by a pH controller responding to the pH in process tank. The rate at which pH modifier solution is added when called for by the controller is

also adjusted to limit overshoots. The primary precipitation target pH of 6.49 is achieved gradually in three stages (reaction tanks) within 50 min total residence time. Undesirable elements such as aluminium, zinc as well as some of the nickel are also removed during this stage. The overflow is directed to the Secondary precipitation circuit through a 1 micron filter.

Version "B" tested selective removal of copper by ion exchange. Therefore, Version "A" copper removal was carried out continuously while Version "B" copper removal was carried out continuously through an ion exchange circuit consisting of IX columns containing a resin selective for the removal of copper (Lanxess TP 207). The copper-free discharge solutions produced by both Versions "A" and "B" are directed to the secondary precipitation (CoCO₃) stage where cobalt is recovered as a high purity carbonate. The secondary precipitation is carried out

with 100 g/L Na₂CO₃ again added by a pump actuated by a pH controller, with independent control of the addition flowrate. The target pH of 7.4 is attained stepwise in four stages (reaction tanks) within 95 minutes total residence time.

The secondary precipitation overflow-discharge is directed to the tertiary precipitation with the objective of removing the (very low tenor) residual cobalt using lime slurry or sodium hydroxide solution (both options were tested in separate runs) regardless of the reagent used. The target pH of 9.2 is attained stepwise in three stages (reaction tanks) with about 70 minutes total residence time. The tertiary precipitate containing discharge slurry is thickened. The overflow is discharged into holding tanks in lieu of the tailings pond.

The primary and tertiary primary precipitate products are recirculated to the iron-arsenic removal circuit solution by direct blending with this circuit's feed stream.

The secondary precipitation product cobalt carbonate filter cake is re-leached in spent electrolyte, purified from the residual nickel and zinc using ion exchange and directed to electrowinning cells where the cobalt cathodes are produced.

2007/2008 PILOT PLANT UPSTREAM OPERATIONS RESULTS SUMMARY

The results summarized in this section were presented in detail in a recent paper (6). In summary, a comprehensive flotation test program and subsequent pilot exercise produced the selective bismuth and cobalt concentrates that were used as feeds for the hydrometallurgical pilot plant continuous pressure oxidation process. Typical assays are given in Table 1.

Five continuous pressure oxidation (POX) pilot runs, each of about one week's duration, were carried out, one on the bismuth concentrate and four on the cobalt concentrate. A schematic of the pilot plant flowsheet is depicted in Figure 2.

Table 1 - Typical continuous POX feed assays

| ASSAYS, % WT. | TYPICAL Co CONCENTRATE HIGH Au GRADE | TYPICAL BI CONCENTRATE HIGH Au GRADE |
|--------------------|--------------------------------------|--------------------------------------|
| Bi | 1.41 | 40.3 |
| Co | 3.64 | 0.85 |
| Fe | 26.6 | 14 |
| Cu | 0.95 | 3.8 |
| As | 18.3 | 3.6 |
| Ni | 0.09 | 0.34 |
| Al | 1.20 | 0.39 |
| Mg | 2.6 | 0.92 |
| Ca | 2.6 | 0.86 |
| S _{total} | 18.7 | 21.7 |
| Sulphide S | 18.6 | 21.5 |
| Sulphate | <0.04 | <0.04 |
| Elemental S | <0.05 | 0.16 |
| ASSAYUS, g/t | | |
| Au | 26.8 | 351 |
| Ag | 9 | 274 |
| Zn | 72 | 545 |
| Cd | <10 | <15 |
| Pb | 63 | 910 |
| Mn | 653 | 245 |
| Chloride | 445 | 78 |

Table 2 - Typical continuous POX discharge PLS assays

| ASSAYS, mg/L | Co CON POX DISCHARGE PLS | Bi CON POX DISCHARGE PLS |
|------------------|--------------------------|--------------------------|
| Bi | 81 | 1255 |
| Co | 3995 | 1235 |
| Cu | 955 | 5885 |
| Au | 0 | 0 |
| Ag | 0 | 0 |
| Ni | 110 | 38 |
| Zn | 7 | 74 |
| As | 1685 | 150 |
| Fe | 3110 | 4670 |
| Fe ²⁺ | 2800 | 2930 |
| Cd | <3 | <2 |
| Pb | <2 | <2 |
| Al | 625 | 200 |
| Mg | 1115 | 300 |
| Ca | 770 | 150 |
| Mn | 33 | 12 |
| Chloride | 108 | 25 |

secondary precipitation stage where cobalt was recovered as a high purity carbonate product using 100 g/L Na₂CO₃ solution. The secondary circuit overflow was directed to the tertiary precipitation ("Scavenging") circuit in order to remove the heavy metals to trace values.

In summary(6), continuous primary precipitation at 50°C, pH 6.49, 50 minutes residence time removed 99% of the copper along with 13% of nickel, 74% of zinc and 97% aluminium (Table 4, Table 5 and Figure 3). Cobalt co-precipitation was 7% (for recycle internally within the process) confirming

good selectivity. Continuous secondary precipitation at 50°C, pH 7.4, 95 minutes residence time recovered 97% of the remaining cobalt and nickel, along with 85%, 55% and 43% of the manganese, calcium and silica respectively. Continuous tertiary precipitation at 50°C, pH 9.2, 70 minutes residence time recovered cobalt and nickel quantitatively (for recycle internally within the process), along with 85% of the remaining calcium and 16% of the magnesium.

Table 3 - Typical FeAsRe feed and discharge solution assays

| ASSAYS, mg/L | FeAsRe FEED ¹ | FeAsRe DISCHARGE ² |
|--------------------------------|--------------------------|-------------------------------|
| Bi | 139 | 1 |
| Co | 4700 | 3905 |
| Cu | 1160 | 198 |
| Ni | 103 | 83 |
| Zn | 14 | 11 |
| As | 860 | 3.4 |
| Fe | 3790 | 1.8 |
| Fe ²⁺ | 1950 | <1 |
| Cd | <0.3 | <0.3 |
| Pb | <2 | <2 |
| Al | 545 | 13 |
| Mg | 1000 | 890 |
| Ca | 610 | 470 |
| Mn | 28 | 27 |
| 1 Co POX to Bi POX Blend ~ 9:1 | | 2 at pH 4.6 |

2008 PILOT PLANT DOWNSTREAM OPERATIONS RESULTS SUMMARY

Scheduling of the various downstream phases of the pilot plant is shown in Table 6. Generally, releach operations started up some time before electrowinning with the preparation of solutions needed to fill the circuit. The times shown are for releach operating periods concurrent with electrowinning operations excluding the startup period. The resin columns were prepared and pre-loaded with cobalt as needed before the pilot plants operated. Some resin columns were eluted during operation

Table 4 - Continuous precipitation (Version A) results – Efficiencies/Selectivities

| RUN FEED ID | PRECIPITATION EFFICIENCY, % wt. | | | | | | | | |
|-------------|---------------------------------|------|------|------|------|------|------|------|------|
| | Co | Cu | Ni | Zn | Al | Mg | Ca | Mn | Si |
| PRIMARY | 7 | 99.1 | 12.6 | 74.1 | 97.3 | 1.5 | 4.2 | 2.8 | 41.5 |
| SECONDARY | 96.5 | | 96.5 | | | 4 | 54.9 | 84.5 | 43 |
| TERTIARY | >98.5 | | | | | 15.8 | 85.1 | 97 | 90.3 |

Table 5 - Continuous selective precipitation (Version A) results – Typical Assays

| STREAM | SOLUTION ASSAYS, mg/L | | | | | | | | | | |
|-----------------------------------|-----------------------|--------|-------|------|--------|------|------|------|-----------------|------|------|
| | Co | Cu | Ni | Zn | Al | Mg | Ca | Mn | Cl ⁻ | Na | Si |
| PRIMARY FEED | 3100 | 71 | 67 | 8 | 9.4 | 1050 | 540 | 26 | 110 | 72 | 57 |
| SECONDARY FEED | 2850 | <0.5 | 62 | 2 | <0.2 | 1040 | 525 | 23 | 100 | 335 | 34 |
| TERTIARY FEED | 97 | <0.3 | 2 | <0.7 | <0.2 | 910 | 225 | 4 | 275 | - | 19 |
| TERTIARY DISCHARGE | <0.6 | <0.1 | <0.6 | <0.7 | <0.2 | 780 | 32 | <0.0 | 510 | - | <1 |
| | SOLIDS ASSAYS, % wt. | | | | | | | | | | |
| PRIMARY | 31 | 11 | 11 | 0.71 | 1.5 | 0.42 | 0.28 | 0.05 | 0.042 | 0.18 | 4.88 |
| SECONDARY | 46 | <0.01 | 0.92 | 0.03 | 0.0014 | 0.44 | 4.8 | 0.33 | 0.01 | 0.04 | 0.23 |
| TERTIARY WITH Ca(OH) ₂ | 7.8 | <0.01 | 0.235 | - | - | 9.1 | 13.5 | 0.13 | - | 1.08 | 0.74 |
| TERTIARY WITH NaOH | 3.8 | <0.005 | 0.095 | - | - | 4.1 | 30 | 0.07 | - | 0.54 | 0.89 |

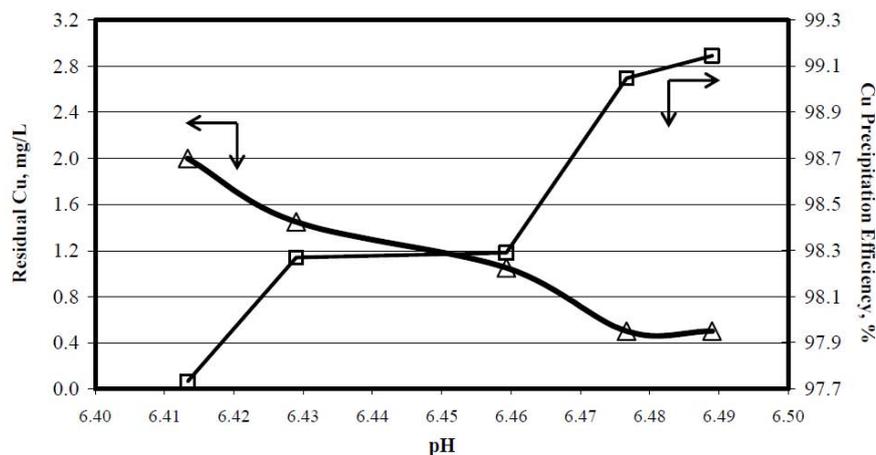


Figure 3 - Primary precipitation curves – continuous pilot

Table 6 - Piloting phases and operating times

| PILOTING PHASE | START | STOP | OPERATING TIME ¹ , H |
|-------------------------------|-------------------------|-------------------------|---------------------------------|
| TP 207 IX Copper Removal | June 02, 2001, 13:30 | June 06, 2008, 00:59 | 83.3 |
| PP9 CoCO ₃ Releach | June 17, 2008, 03:00 | June 20, 2008, 13:30 | ~80 |
| PP9 Co EW | June 16, 2008, 14:00 | June 20, 2008, 13:30 | 86.8 ² |
| PP11 Releach and EW | October 14, 2008, 21:10 | October 29, 2008, 20:00 | 232.5 ² |

¹excluding stoppages and intermediate shutdowns; ²EW elapsed time

Table 7 - Resins tested in bench-scale screening study

| RESIN | MANUFACTURER | TYPE/FUNCTIONALITY | TARGET METAL(S) |
|-----------|--------------|---|-----------------|
| TP 207 | Lanxess | macroporous chelating weak acid cation/iminodiacetate | Cu (Ni) |
| S 930 | Purolite | macroporous chelating/iminodiacetic | Zn |
| S 940 | Purolite | macroporous chelating/aminophosphonic | Zn |
| S950 | Purolite | macroporous chelating/aminophosphonic | Zn |
| XUS 43578 | Dow | macroporous chelating/ bis picolylamine | Ni |

while others were eluted after the pilot campaign they were used in had ended.

The initial larger scale operation of releach and Co EW had been designed to match the upstream pilot plant flows and this campaign is designated as PP9. All the process and remaining solutions that were available after the first (PP9) electrowinning pilot plant operation were collected and the contained cobalt was precipitated with sodium carbonate. This re-precipitated cobalt carbonate was

then used as feed to a much smaller scale releach and electrowinning circuit, designated PP11. The advantage of the smaller circuit was that it enabled the pilot plant to operate much longer and for many more cycles than were possible in the larger-scale pilot. In addition, problems that had been identified in PP9 were addressed in PP11 so that good cobalt deposits could be produced.

SOLUTION PURIFICATION PRELIMINARY SCREENING RESULTS

The principal impurity of concern in the production of cobalt carbonate was copper and as already described, its removal was investigated via two routes, as indicated in the previous section. The results of “Version B”, whereby the feed to the carbonate precipitation was processed through columns of Lanxess TP 207 ion exchange resin to extract copper, are discussed here. The effluent from the ion exchange columns was routed to the precipitation circuit where cobalt carbonate was precipitated with sodium carbonate. The resulting cobalt carbonate was advanced to redissolution, further solution purification aiming to remove nickel and zinc, followed by electrowinning. The performance of five different resins was evaluated at bench scale with respect to their extraction of copper, nickel, cobalt and zinc from three different solution matrices (Table 7).

From this preliminary work, it was concluded that Lanxess TP 207 was the best resin for extracting copper ahead of cobalt carbonate precipitation. Loading was less sensitive to pH than with the other resins. It also showed some promise for extracting nickel. For zinc extraction from the electrolyte, S 950 appeared to be the best choice, though complete extraction of zinc was not achieved. The first continuous pilot plant was nevertheless designed to use S 950 based on these results. In the subsequent PP11 campaign, it was replaced with Lanxess VP OC 1026, a divinylbenzene polystyrene matrix that is impregnated with di-2-ethylhexyl phosphoric acid (D2EHPA), a common solvent extraction reagent. XUS 43578 was able to extract nickel well from the lower cobalt content solutions, not as well from the electrolyte. Lower pH, within the pH 3 to 5 range, appeared to favour extraction. The conditions under which the resin was tested were not ideal and as the resin is used commercially in purification of cobalt electrolytes, it was decided to continue to piloting with it, and the results justified this decision, as is detailed further below.

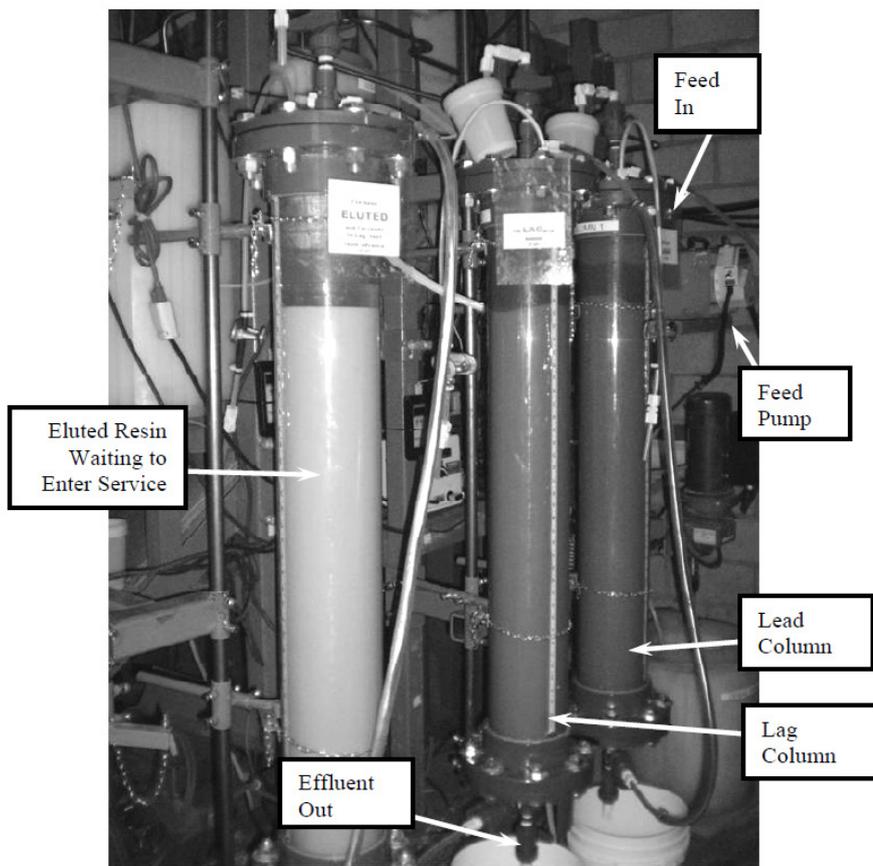


Figure 4 - TP 207 Columns in operation for Cu extraction

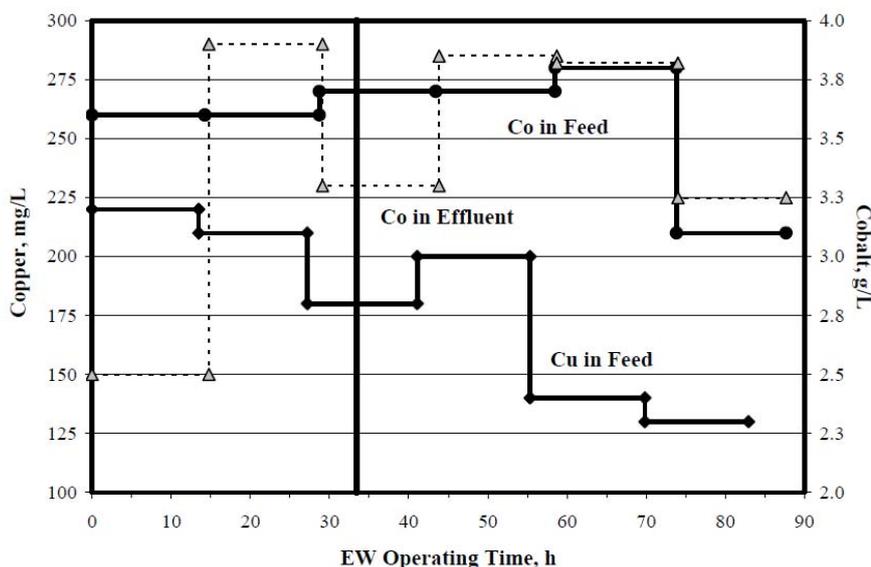


Figure 5 - Copper and cobalt in and cobalt out of TP 207 resin columns

PILOT SCALE VERSION B COPPER REMOVAL USING LANXESS TP 207

The ion exchange circuit (Figure 4) operated continuously for 83 hours, processing a total of 5840 L of feed solution. The time-weighted average feed flowrate was 1202 mL/min equating to a flow through the resin of 4.24 BV/h.

Figure 5 shows the copper tenor of the feed that was processed through the IX

resin circuit. The copper concentration did decline steadily over the course of the campaign, due to changes in upstream processing parameters. Effluent copper concentrations are not plotted because the Lanxess TP 207 resin columns removed copper efficiently to sub-mg/L levels from the iron/arsenic precipitation filtrate. Copper breakthrough to the effluent took around 100 bed volumes of feed.

The cobalt concentration in and out of the TP 207 circuit is also shown in the figure. When resin columns were advanced (as indicated by the heavy vertical line at ~33 h – and there was only one advance during the campaign), the resin initially picked up some cobalt, but this was subsequently displaced by the copper that was loaded. This can be seen from the graph where the feed cobalt concentration is higher than the effluent at the start and after the resin advance, but subsequently, the effluent concentration is higher than the feed as the loaded Co is pushed out by copper. This is another reason for aiming for full copper loading of the resin to minimize cobalt co-extraction.

The co-extraction of nickel with TP 207 was also investigated. While it was found that nickel would initially be extracted by the resin, that nickel would subsequently be displaced by copper. Therefore, either the copper extraction has to be quite inefficient with low loadings or nickel extraction has to be left for other process steps. Furthermore, it was found that when the poorly copper-loaded resin was eluted with acid, the nickel eluate, a discard stream, contained over 3 g Co for each g of nickel eluted and destined to be discarded and this was considered to be an unacceptable loss.

Elution of the TP 207 resin was readily accomplished with a two-step process using 95 g/L H₂SO₄ in each step. The first step took nickel and cobalt off the resin. The second step produced a high copper eluate with lower cobalt content. Since effective nickel cobalt separation from the eluate was considered as unlikely to be successful it was decided to use the XUS resin for nickel removal.

Cobalt carbonate was precipitated from the Lanxess TP 207 discharge solutions with 100 g/L Na₂CO₃ solution, under conditions described above (i.e. secondary precipitation). The analyses of the cobalt carbonate precipitates batches produced are presented in Table 8.

COBALT CARBONATE RELEACH AND PURIFICATION

The large-scale pilot plant designated PP9, was operated for about 87 h at a feed rate to EW of ~700 mL/min of rich

Table 8 - PP 9 cobalt carbonate batch assays

| | | Cu REMOVED BY SELECTIVE PRECIPITATION | | | | Cu REMOVED BY IX WITH TP 207 | | | |
|------------------------|-----|---------------------------------------|-------|-------|-------|------------------------------|------|------|------|
| CAKE NUMBER | | 1 | 2 | 3 | 4 | 1 | 2 | 3 | 4 |
| WET WEIGHT MOISTURE | kg | 23.20 | 14.84 | 21.25 | 11.74 | | | | |
| | wt% | 49.2 | 48.2 | 49.8 | 46.6 | 67.7 | 62.2 | 61.7 | 61.8 |
| ASSAYS, DRY BASIS | | | | | | | | | |
| Co | wt% | 46 | 44 | 45 | 42 | 46 | 42 | 43 | 43 |
| Ni | g/t | 9200 | 9100 | 9000 | 9200 | 620 | 2200 | 2200 | 2200 |
| Zn | g/t | 330 | 330 | 330 | 340 | 840 | 960 | 970 | 1100 |
| Mn | g/t | 3300 | 3300 | 3200 | 3300 | 3100 | 2900 | 3000 | 3000 |
| Cu | g/t | 72 | 72 | 75 | 79 | <40 | <40 | <40 | <40 |
| Ca | wt% | 4.8 | 4.6 | 4.7 | 4.5 | 5 | 3.7 | 4.6 | 4.6 |
| Mg | g/t | 4400 | 4100 | 4100 | 4200 | 8300 | 8500 | 8500 | 8700 |
| Na | g/t | 580 | 400 | 450 | 420 | 10000 | 6500 | 6400 | 6500 |
| K | g/t | <160 | <40 | <50 | <50 | 290 | 410 | 400 | 400 |
| Sr | g/t | 220 | 210 | 210 | 210 | 290 | 230 | 230 | 230 |
| Fe | g/t | 35 | 34 | 56 | 160 | 210 | 66 | 170 | 610 |
| Al | g/t | 14 | 17 | 31 | 46 | 1600 | 1600 | 1700 | 1600 |
| As | g/t | <30 | <30 | <30 | <30 | 190 | 150 | 150 | 150 |
| Cl | g/t | 82 | 90 | 91 | 98 | 56 | 56 | 79 | 56 |
| SO ₄ | wt% | 1.3 | 1.1 | 1.2 | 1.1 | 3.7 | 2.5 | 2.4 | 2.4 |
| CO ₃ | wt% | 25.6" | 25 | 22.7 | 22.9 | 24.2 | 25.3 | 27.3 | 28.6 |
| Ba | g/t | <0.2 | <0.2 | <0.2 | <0.2 | 19 | 19 | 20 | 19 |
| Tl | g/t | 130 | 130 | 130 | 130 | <60 | <60 | <60 | <60 |
| Sb | g/t | <60 | <60 | <60 | <60 | <60 | <60 | <60 | <60 |
| Sn | g/t | <30 | <35 | <90 | <50 | <50 | <50 | <50 | <50 |
| Bi | g/t | <20 | <20 | <20 | <20 | <20 | <20 | <20 | <20 |
| P, Se | g/t | <30 | <30 | <30 | <30 | <30 | <30 | <30 | <30 |
| Pb, U | g/t | <20 | <20 | <20 | <20 | <20 | <20 | <20 | <20 |
| Be | g/t | <1 | <1 | <1 | <1 | 0.7 | <1 | <1 | <1 |
| Y | g/t | 1.1 | <2 | <1 | <1.2 | <10 | <8 | <10 | <10 |
| Cr | g/t | <4 | <4 | <4 | <4 | <4 | <10 | <5 | <10 |
| Cd | g/t | <2 | <2 | <2 | <2 | <5 | <10 | <10 | <10 |
| Li, Mo | g/t | <5 | <5 | <5 | <5 | <5 | <5 | <5 | <5 |
| V | g/t | <3 | <2 | <2 | <3 | <5 | <5 | <5 | <5 |
| Ti | g/t | <2 | <2 | <2 | <2 | 3 | 2 | <2 | <2 |
| Ag | g/t | <2 | <2 | <2 | <2 | <2 | <2 | <2 | <2 |

electrolyte at 40 g/L Co, with an EW bite of 5 g/L Co. This continuous pilot circuit consisted of re-leaching cobalt carbonate followed by filtration, zinc removal with Purolite S 950 resin and nickel control with Dow XUS 43578 resin, followed by electrowinning of cobalt. For each type of resin, two IX columns were used in a series Lead/Lag configuration. The columns operated at ~8 and ~3 BV/h (Bed Volumes/h) for the S 950 and XUS resins respectively, and at 60-65°C. A

bed volume (BV) of resin was defined as the volume of wet settled resin in a resin column before any pretreatments other than washing with water. Spent electrolyte was recycled to the re-leach stage to dissolve more carbonate. The larger scale of the circuit resulted in a relatively short operating time before the available feed carbonate was exhausted.

The second continuous pilot plant campaign, designated PP11, was

operated at a much more modest scale with a feed rate to the EW cell of 86 mL/min. In this campaign, which ran for 232.5 h, re-leaching was done in batch mode, but the IX and EW sections of the circuit ran continuously. The feed to this pilot plant was cobalt carbonate precipitated from the solutions and electrolytes that remained at the end of the PP9 campaign, so the PP11 feed carbonate was lower both in cobalt as well as in some impurities, particularly

Table 9 - PP11 feed carbonate batch assays

| BATCH NUMBER | | 1 | 2 | 3 | 4 | 5 |
|------------------------|-----|-------|-------|-------|-------|-------|
| WET WEIGHT MOISTURE | kg | 19.34 | 16.14 | 18.63 | 19.84 | 9.21 |
| | wt% | 70.6 | 57.8 | 47.5 | 48.3 | 67.5 |
| ASSAYS, DRY BASIS | | | | | | |
| Co | wt% | 31.1 | 38 | 31.8 | 34.3 | 39.2 |
| Ni | g/t | 2100 | 2500 | 2300 | 2300 | 2900 |
| Zn | g/t | 9 | 8 | 71 | 73 | 86 |
| Mn | g/t | 770 | 920 | 750 | 760 | 980 |
| Ca | g/t | 2900 | 3300 | 3700 | 3800 | 4900 |
| Mg | g/t | 530 | 630 | 930 | 1000 | 1500 |
| Na | g/t | 290 | 180 | 1100 | 1200 | 290 |
| Fe | g/t | 280 | 180 | 110 | 180 | 540 |
| Ag | g/t | <8 | <8 | <8 | <8 | <8 |
| Cu | g/t | <10 | <10 | <10 | <10 | 17 |
| Al | g/t | 54 | <30 | <30 | <30 | 150 |
| Ba | g/t | 3.4 | 98 | 9.9 | 140 | 9.8 |
| Sr | g/t | 34 | 42 | 46 | 49 | 64 |
| K, Sn | g/t | <80 | <80 | <80 | <80 | <80 |
| Sb, Tl | g/t | <60 | <60 | <60 | <60 | <60 |
| As, P, Se | g/t | <30 | <30 | <30 | <30 | <30 |
| Bi, Pb, U | g/t | <20 | <20 | <20 | <20 | <20 |
| Cd, Cr, V | g/t | <10 | <10 | <10 | <10 | <10 |
| Li, Mo | g/t | <5 | <5 | <5 | <5 | <5 |
| Ti | g/t | 2 | <2 | <2 | <2 | 5 |
| Y | g/t | <0.6 | <0.6 | <0.6 | <0.6 | <0.6 |
| Be | g/t | <0.03 | <0.03 | <0.03 | <0.03 | <0.03 |

in zinc and copper (Table 9), than the carbonate used for PP9.

The schematic of the flowsheet used in PP11 is shown in Figure 6. Second campaign (PP11) results were considered representative of the pilot exercise and they are briefly summarized below. The releach operating conditions in PP11 were in principle similar to PP9. Carbonate was releached in a batch stirred tank of inert materials with provisions for controlled heating and acid addition.

Spent electrolyte from the electrowinning cell was collected in batches of 20 L in polyethylene pails. When sufficient solution was available, it was transferred to the insulated polypropylene releach tank (~80 L tank) where carbonate was added and the releach process was carried out. After releaching the pulp was filtered to recover any unleached carbonate

for recycle and to produce a clear filtrate to advance to the ion exchange circuit. Filtration was carried out on a polypropylene vacuum pan filter using Whatman No. 3 filter paper. The filtrate was collected in a second plastic tank (~70 L capacity) that was used to feed the IX circuit. All wetted parts were of inert materials. Pumping was done with peristaltic units. The releach tank was operated at 50°C with a target rich electrolyte concentration of ~40 g/L Co, taking into consideration the moisture and cobalt content of the batch or batches of carbonate that were being used. While adding carbonate, the pH was monitored and it was not allowed to exceed 5.0 with a target of 4.5 for the releach product. Manganese concentration was adjusted to a target of 1.5 g/L Mn using manganese sulphate salt. The initial batch of electrolyte was made up to contain 20 g/L boric acid to serve as pH buffer. As well, 20 mg/L Guartec (Cognis) guar gum was added

initially as a deposit smoothing (levelling) agent. Subsequently, 5 mg/L Guartec and 1 g/L boric acid were added to each releach batch as makeup. The guar gum is decomposed and consumed to some extent during processing and some is likely to be adsorbed on the ion exchange resins. The boric acid is not consumed, but adding excess or exceeding the solubility is not a problem for EW so it was decided to keep adding this reagent to each batch. The main source of dilution was the moisture in cobalt carbonate cake that was added as evaporation from the hot EW cell was compensated for with DI water and the pH adjusting NaOH solution added to the EW reheater reservoir. The undissolved wet residue that was collected on the filter was usually added to the next releach batch prepared.

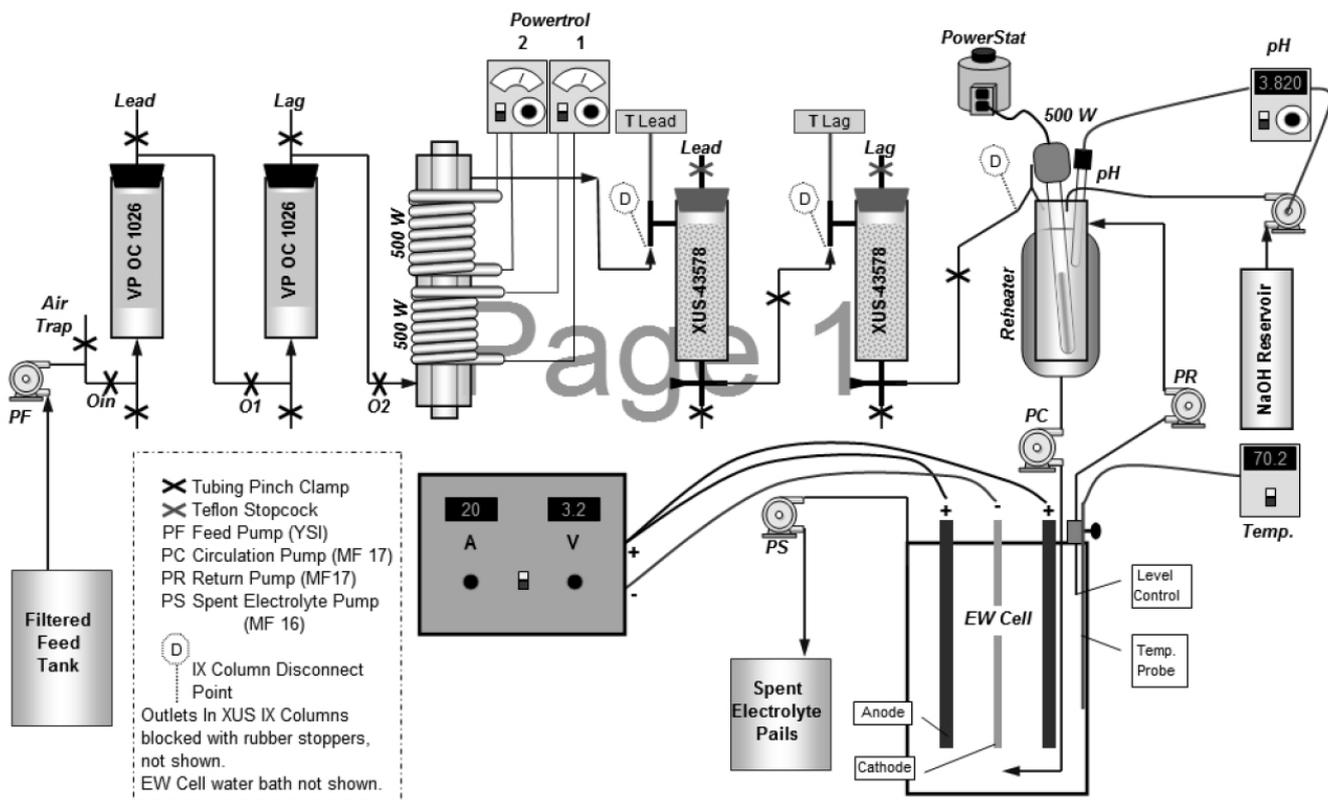


Figure 6 - PP9 cobalt releach and electrowinning schematic

ZINC REMOVAL

Zinc removal was achieved using Lanxess VP OC 1026 resin. The resin particles are irregularly shaped and relatively fine (0.3-1.6 mm). The resin is suitable for application in pH ranges below 6 as above that value the impregnate is lost at unacceptably high rates. Below a pH of 1, the resin is not very effective as this is outside the range at which D2EHPA extracts common cations from solution. The extraction mechanism of the resin is analogous to that in solvent extraction with this reagent. Protons are exchanged for the metal ion being extracted so the pH of the effluent is lower than the pH of the feed. The ionic species extracted and the degree of extraction depend on pH. To strip the resin, the acidity is increased and the extraction reaction is reversed. The selectivity of the resin depends on the pH and what else is in solution as D2EHPA is a relatively omnivorous extractant. In the pH ranges of interest in the Co circuit, D2EHPA will extract zinc and some manganese as well as some cobalt.

Releach filtrate solution was pumped at a set flowrate to the two ion exchange columns filled with Lanxess VP OC 1026

resin. Because the resin floats, solution was pumped through these resin beds in upflow mode. The IX feed pump was the only pump used in the entire ion exchange train. The feed lines to the ion exchange columns were fitted with traps to catch any air bubbles that might be introduced and to prevent these from entering the columns. The zinc removal step operated at ambient temperature in upflow mode at a flowrate of 7-7.4 BV/h. Zinc was determined in feed and spent electrolyte samples. In almost all cases the concentration was below reporting limits which were either 2 mg/L or 0.7 mg/L depending on the analytical method and the sample matrix, indicating quasi-quantitative removal.

NICKEL REMOVAL

Dow bis-picolyamine resin XUS 43578 was used to control the nickel concentration in the electrolyte at tolerable levels. This resin extracts both nickel and cobalt and the relative amounts depend on pH. If present, copper is also extracted and tends to poison the resin, so its removal from the feed to the XUS resin was carefully monitored.

Nickel extraction kinetics are slow and operating at elevated temperature favours them so the effluent solution from the VP OC 1026 Lag column was heated to about a target temperature of 60-65°C using an in-line heater. This minimized solution holdup. The temperature entering the Lead XUS column was monitored, and power to the heaters adjusted (manually) accordingly.

The resin columns were glass cylinders, 6.55 cm in diameter with a height of 25.0 cm to the solution inlet. The two columns were operated in series in Lead Lag configuration. Both operated in flooded downflow mode with solution introduced at the top. Normally, the columns and the entire resin train including the in-line heater were operated with all openings sealed so that solution was forced through the two beds by the pressure provided by the feed pump feeding the VP OC 1026 columns.

The two resin columns were filled, each with 800 mL of wet settled resin. The resin was preloaded with cobalt before being put into service, in order to minimise the pH change that would result from nickel extraction. Pre-loading

allows the resin to exchange Co ions for Ni, rather than H⁺ ions. The columns were operated at a target flowrate in the range 6.1-6.5 BV/h. Resin elution was carried out offline and extra resin columns were prepared to be brought into the circuit as needed.

A primary objective was to keep the Ni concentration in the electrolyte below 200 mg/L and this was achieved as can be seen in Figure 7. The vertical bars in the figure indicate times when the XUS resin columns were advanced. It was possible to control cobalt losses by loading the nickel well and by using a two-step elution where the first step removed the bulk of the cobalt in the resin bed so that the cobalt lost with the discarded nickel eluate could be kept to about 0.1 g Co/g Ni. This performance was sufficient to prevent excessive nickel deposition on the cathode during the subsequent EW step.

RECIRCULATION AND ELECTROWINNING

The solution discharged from the Lag XUS column entered the reheating reservoir. This was a double-walled glass vessel fitted with a glass immersion heater that was powered by a Variac variable voltage transformer, manually controlled. Electrolyte was pumped from the bottom of the reservoir into the cell at a fixed rate of 500 mL/min. The solution was pumped by a peristaltic pump entering the EW cell through a distributor pipe under the cathode. At the top of the cell, a suction line was fixed in place. The depth of the suction line was set to control the cathode immersion at the desired value. A peristaltic pump was used to draw solution from the waterline in the EW cell and recirculate it to the reheating reservoir. This pump maintained a constant level in the cell. Another pump was set up to pump cell electrolyte from behind one of the anodes out of the cell into the spent electrolyte pail. The flowrate of this pump was set to match the flowrate of effluent being discharged from the IX column train. The flowrate of this pump also allowed the operators to gradually adjust the Co tenor in the cell. If the tenor was too low, the flowrate could be slowed and with evaporation, the concentration of Co in the cell would gradually increase if the plating rate was constant. If the

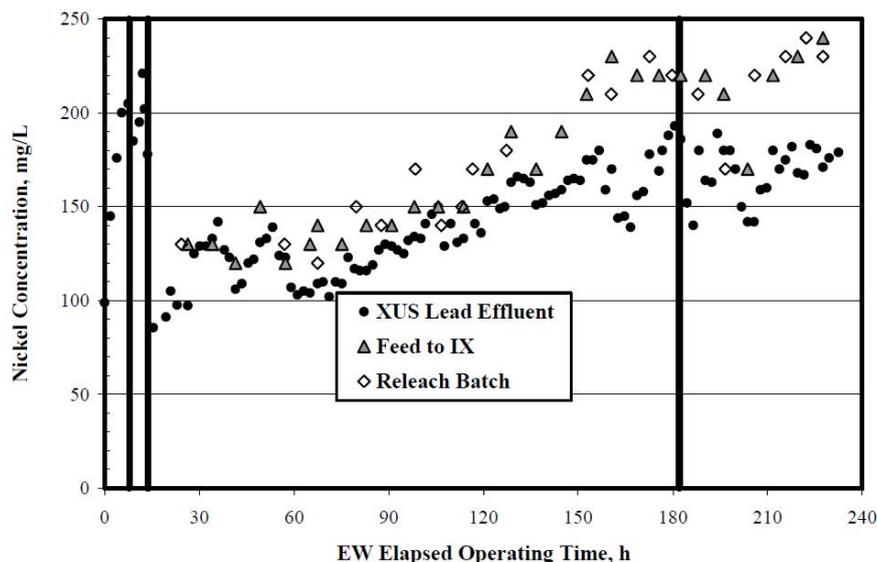


Figure 7 - Nickel in the electrolyte (PP11)

flowrate out was set higher than the flowrate in, adding water in the reheater diluted the electrolyte bringing the concentration back down again. As the cell was operating at an elevated temperature, either 65°C or 70°C, there was significant evaporation taking place. The evaporation could be monitored by the level of solution in the reheating reservoir because the level in the cell itself was fixed. Initially DI water was used to periodically adjust the volume in this reservoir back to a set level making up for evaporation. Early on in the campaign, it was found that pH control in the cell was advantageous. To do this, the reheating reservoir was fitted with a pH electrode connected to a pH controller. The controller actuated a peristaltic pump that pumped NaOH solution out of a calibrated reservoir when the pH in the reheater was too low. This allowed the maintenance of a stable pH in EW and it provided another way to compensate for evaporation. Ultimately it was found that with a 10t% wt. NaOH solution, almost no additional water was required to balance evaporation.

The cell itself was constructed of polypropylene plate with a unit area of 360 mL/cm of depth. It was fitted with two anodes of Pb/Ca/Sn alloy and one stainless steel (316 alloy) cathode between them. The anode-cathode spacing was 5 cm. The anodes were explosion bonded to copper header bars and the cathode was bolted to a copper

header bar. The cathode was 20 cm wide and the immersion depth was initially 22 cm, but later was reduced to 20 cm. The entire cell was mounted inside a heater bath so the immersion heater in the reheater unit was only used to trim the temperature of the cell, while the heater bath provided most of the heat to get the cell to the operating temperature. The cell was open on top and a loose-fitting vent hood was installed to collect any vapours or acidic mists that might be evolved. The electrolyte in the cell was circulated at a replacement rate of approximately 20 min. To minimize operating shutdowns when cathodes were harvested, two identical cathode blanks were prepared so that one was always ready to go into service. The cathode was prepared by removing any prior deposits, washing thoroughly with detergent and water, rinsing with DI water degreasing with acetone and air drying. To promote deposit adherence, the clean cathode surface was roughened with 120 grit emery cloth. This was subsequently changed to a coarser 80 grit cloth and finally to 50 grit for the last four plating cycles. Initially, the cathode was fitted with edge strips to facilitate stripping, but as these were found to be unnecessary and further to promote cathode adhesion, they were removed very early in the campaign.

Power to the cell was furnished by a constant current rectifier unit. This unit also indicated the applied voltage to maintain the current. A drop in voltage

was one of the signs the operators watched for to determine if a cathode was self-stripping off the blank.

The cell was operated with a targeted 5 g/L Co bite with the rich electrolyte coming in at ~40 g/L Co and the cell electrolyte or leaving spent electrolyte having ~35 g/L Co.

The key parameters monitored or controlled in the electrowinning were applied current, current density, cell voltage and cell temperature, shown in Figure 8 where the dashed vertical lines correspond to the times when a cathode was harvested. The thicker vertical lines indicate the cathode strips that took place before a weekend shutdown.

During the campaign, cathode immersion was changed once from 22 cm to 20 cm. The change was made at the time that the current was reduced to 16 A at 24 h of operation. This change was made because it was observed that the spent electrolyte concentration was dropping which meant that plating was faster (or more efficient) than had been assumed in the design calculations. It would have been possible to maintain the immersion by increasing the feed flowrate to the cell, but it was desirable to keep the specific flows through IX constant (BV/h) so instead, the current was reduced and the cathode area was also reduced to maintain the current density constant.

The target cathodic current density was 250 A/m² and this was maintained for most of the campaign as Figure 8 shows. Cell operating temperature was also increased from 65°C to 70°C at ~26 h, so shortly after the current density was changed, and this complicates the interpretation of the effect of lowering the current density. Drops in operating temperature do appear to be correlated with shorter plating cycles as can be seen at ~70 h and again in the period from 85 to 100 h when the operating temperature was left too low. The frequency of cathode strips as shown by how close the dashed vertical lines are, increased in periods of low operating temperature. It can be seen that after the first weekend shutdown, the cell was restarted too cold and this appears to have contributed to a need to strip the

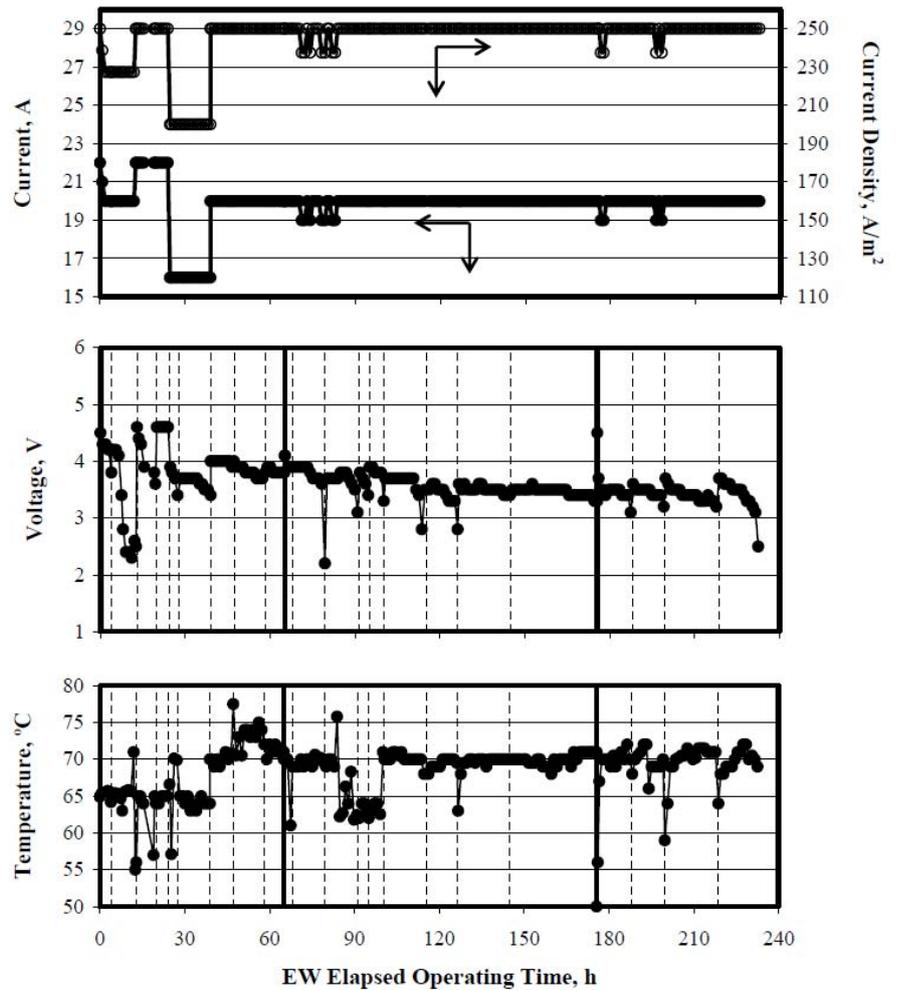


Figure 8 - Electrowinning cell operating parameters (PP11)

cathode shortly after starting. Following the second weekend shutdown (~175 h), the cell temperature was brought up to the target before EW was commenced and the length of the plating cycle was not adversely affected, though the longest plating cycle that had ended with that weekend shutdown was not repeated. Plating cycles typically ended

with the cathodes cracking and self-stripping. To try to stop this from taking place, as mentioned, the edge strips were removed after the first cycle. Increasingly coarser emery cloth was used to roughen the stripped cathode blank surface and ultimately a 50 grit cloth was employed. The change from 120 to 80 grit may have been useful, but

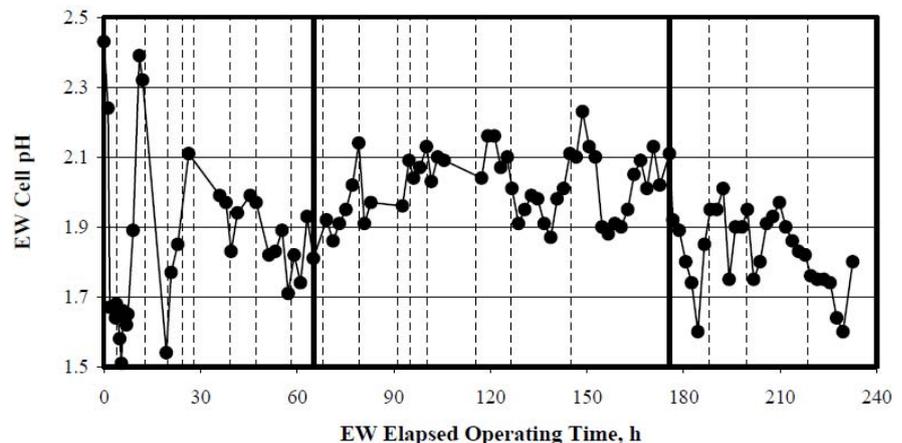


Figure 9 - Effect of Cell pH on Length of Plating Cycle

the data are inconclusive about the effect of using the coarser 50 grit cloth.

Cell pH is plotted in Figure 9 along with the dashed vertical lines showing the times at which cathodes were stripped (and the heavier solid lines designating a cathode harvest before a weekend shutdown). It is evident that self-stripping tendencies decreased as the campaign progressed. The shortest plating cycle was 2.9 h, the longest 30.8 h. The mean plating cycle time was 10.6 h and half the cycles were longer than 10.9 h. These data showed that longer plating cycles should be possible and that it is likely that electrolyte quality is not the issue, but rather the physical operation of the cell.

A factor that may influence the plating cycle time is pH. As the figure shows, the cell pH did not vary much over the course of the campaign. However, there is a slight trend towards a greater spacing between the bars, longer plating cycles, when the pH was higher, at least over 2.0.

One parameter that was not investigated in this campaign was the solution circulation rate in the EW cell, which was rather slow at about 20 minutes for replacement of one cell volume. It is suspected that this circulation rate was too slow and in fact, better results in terms of self-stripping and the length of plating cycles were achieved in the larger scale pilot campaign (PP9) where the cell operated at a higher circulation rate (4-6 min), despite the fact that the electrolyte contained higher levels of impurities, and pH control was not as rigorous in that campaign. Higher circulation rates are often employed to give better agitation in the cell. Some operations even sparge air or an inert gas under the cathodes to provide agitation and to drive any hydrogen bubbles that nucleate on the cathode surface out of solution.

The need to remove a cathode was seen by the operators principally in the drop in cell voltage which reduced the anode-cathode gap, decreasing cell resistance and so operating voltage. It can be seen in the voltage plot in Figure 8 that stripping almost always took place after the voltage dipped. On a number of



Figure 10 - PP11 cobalt cathode

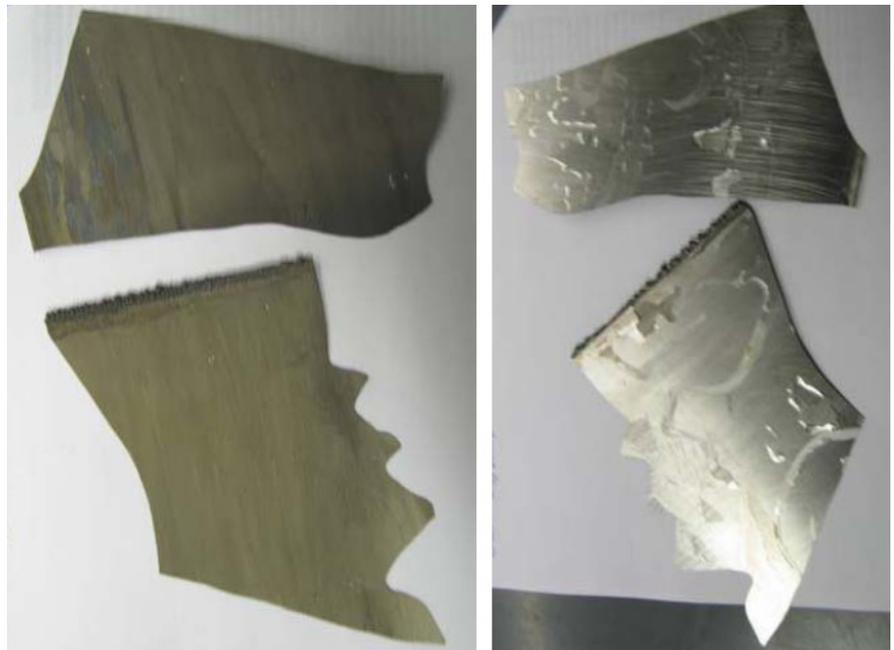


Figure 11 - PP11 cobalt cathode, front and back views

occasions, the cracking of the cathode could actually be heard by the operators. In some cases, this resulted in the need to strip the deposit before it fell off as the voltage dropped fairly quickly after the cracking was heard. In other cases, the cracking was not fatal and the cathode stayed in place and plating continued.

Despite the problems with cathode self-stripping, the cathodic deposits were always smooth, even across their surfaces and dense. There was only a very small amount of dendritic growth at the edges of the cathode sheets, as

normally occurs, especially when edge strips are not employed. In a few cases there was some indication of gas pits on the cathode surface.

Photographs of PP11 cathodes are shown in Figure 10 and Figure 11. Cathodes were readily stripped from the stainless steel blanks and they generally came off in fairly large pieces because the metal was very brittle and hard. A few gas pits can be seen in the surface of the left piece of cobalt in Figure 10.

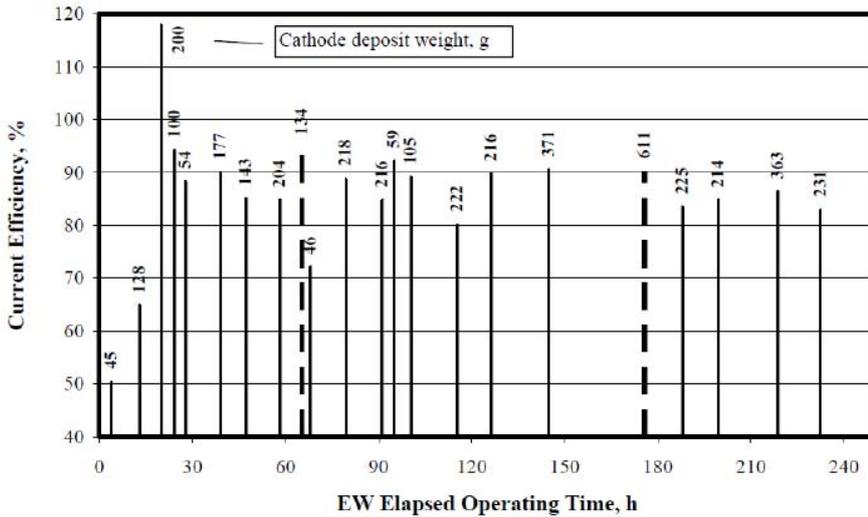


Figure 12 - Current efficiency (PP11)

CATHODE PRODUCTION AND CURRENT EFFICIENCY

A total of 4376 g of cobalt cathode was harvested in 22 plating cycles. Figure 12 is a plot of the current efficiency along the campaign timeline. The vertical bars are placed at the time that a cathode was stripped and the height of the bar indicates the current efficiency calculated for the cathode deposit in the preceding time period up to the cathode harvest. The two thicker dashed bars show the strips carried out before weekend shutdowns. The weight of cobalt harvested at each strip is indicated by the number at the top of the bar.

Steady state current efficiency was generally in the 85-90% range with only a few exceptions. The time-weighted average current efficiency over the whole campaign was 86.4%, and 87/1% excluding the initial few cycles in the startup period. This is a reasonably good result for plating cobalt, and near the upper end of the range typically reported in commercial operations. Good pH control in EW was most

likely the reason for the good efficiency, and it may be advisable to operate EW at a slightly higher pH, targeting perhaps 2.5 instead of 2.0, to further increase plating current efficiency. This may also help to reduce self-stripping as discussed above.

CATHODE PURITY

A number of cathode samples were submitted for chemical analysis and the results are shown in Table 10. The first three lines in the table show the target values for the three grades of cobalt that are marketed by Umicore, a major producer. The table also indicates with the heavier division lines, the cathodes produced in each of the three weeks of operation. Week 1 ended at cathode 10 which was in fact material recovered from the cell bottom. Week 2 ended at cathode 21 which was the heaviest cathode produced. Two cathode blanks were used in the piloting identified as A and B and these are indicated in the cathode identification column.

In general, the cathodes were of Umicore A quality except for a few elements. Lead tended to be high in all the cathodes but decreased progressively as the campaign progressed, staying below 50 g/t in the final operating week. Lead originates

Table 10 - PP11 cathode assays

| ELEMENT UNITS | CO ³ % | Ni g/t | Cu g/t | Fe g/t | Zn g/t | Mn g/t | Ca g/t | Mg g/t | Cd g/t | Pb g/t | S g/t | Cr g/t | Al g/t | Si g/t |
|-----------------------------|-------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|-------|--------|--------|--------|
| Umicore A | >99.8 | <1000 | <30 | <50 | <50 | <10 | <50 | <20 | <10 | <20 | <10 | <10 | <10 | <10 |
| Umicore B | >99.6 | <1000 | <50 | <100 | <100 | <50 | <100 | <50 | <50 | <50 | <400 | <50 | <50 | <40 |
| Umicore C | >98.5 | <5500 | <200 | <2000 | <500 | <1000 | <250 | <250 | <100 | <100 | <500 | <100 | <600 | <500 |
| Pilot Plant Cathodes | | | | | | | | | | | | | | |
| 1A | 99.94 | 150 | <8 | 4 | <7 | 19 | <9 | <2 | 29 | 360 | | <4 | <6 | |
| 2B | 99.91 | 270 | <8 | 18 | <7 | 41 | <9 | <2 | 34 | 540 | | <4 | <6 | |
| 3A | 99.92 | 310 | <8 | 16 | 14 | 26 | <9 | <2 | 24 | 390 | | <4 | <6 | |
| 4B | 99.92 | 220 | <8 | 86 | <7 | 8.8 | <9 | <2 | 27 | 400 | | 18 | <6 | |
| 5A | 99.93 | 250 | <8 | 18 | <7 | 20 | <9 | <2 | 30 | 410 | | <4 | <6 | |
| 6B | 99.79 | 1700 | <8 | 55 | <7 | 13 | <9 | <2 | 35 | 290 | | 15 | <6 | |
| 6B W ¹ | | 1100 | <5 | 8 | <7 | 4.1 | <9 | <0.7 | 18 | 150 | 2800 | <5 | <9 | |
| 7aA | 99.45 | 4600 | <8 | 6 | 160 | 12 | <9 | <2 | 16 | 700 | | <4 | <6 | |
| 7bA | 99.52 | 4100 | <8 | 6 | 190 | 33 | <9 | <2 | 16 | 460 | | <4 | <6 | |
| 8B | 99.92 | 430 | <8 | 24 | 95 | 14 | <9 | <2 | 26 | 230 | | 6 | <6 | |
| 8B W | | 380 | <5 | 12 | 97 | 7 | <9 | <0.7 | 15 | 130 | <100 | <5 | <9 | |
| 9A | 99.9 | 370 | <8 | | | | <9 | <2 | 26 | | | | <6 | |

¹ cathode number (cycle) and blank used (A or B); W indicates sample washed in 10% HNO₃

² recovered from cell bottom

³ calculated by difference

| | | | | | | | | | | | | | | |
|-----------------|-------|------|-----|-----|-----|------|----|------|----|------|------|----|----|----|
| 9a W | | 400 | <5 | 26 | <7 | 3.1 | <9 | <0.7 | 14 | 190 | <100 | <5 | <9 | |
| 10 ² | 99.75 | 1200 | <40 | 17 | 15 | <0.4 | <9 | <2 | 32 | 1200 | | <4 | <6 | |
| 11A | 99.91 | 360 | <8 | 220 | 36 | 43 | 10 | <2 | 13 | 120 | | 52 | <6 | |
| 12B | 99.93 | 370 | <8 | 79 | <7 | 19 | <9 | <2 | 14 | 140 | | 21 | <6 | |
| 13A W | 99.95 | 340 | <10 | <50 | 11 | 3.2 | <9 | <5 | <8 | 99 | | 9 | <9 | <8 |
| 14B W | 99.95 | 400 | <10 | <50 | <10 | 4.6 | <9 | <5 | <8 | 80 | | 7 | <9 | <8 |
| 17B W | 99.95 | 400 | <10 | <50 | <10 | 3.9 | <9 | <5 | <8 | 60 | | <5 | <9 | <8 |
| 19B W | 99.95 | 460 | <10 | <50 | <10 | 3.6 | <9 | <5 | <8 | 61 | | <5 | <9 | <8 |
| 20A W | 99.95 | 450 | <10 | <50 | <10 | 3.2 | <9 | <5 | <8 | 62 | | <5 | <9 | <8 |
| 21B W | 99.94 | 550 | <10 | <50 | <10 | 11 | <9 | <5 | <8 | 43 | | <5 | <9 | <8 |
| 22A W | 99.95 | 480 | <10 | <50 | 10 | 2 | <9 | <5 | <8 | 49 | | <5 | <9 | <8 |
| 23B W | 99.94 | 480 | <10 | 73 | 11 | 3.1 | <9 | <5 | <8 | 42 | | 13 | <9 | <8 |
| 24A W | 99.94 | 540 | <10 | <50 | 37 | 2 | <9 | <5 | <8 | 45 | | <5 | <9 | <8 |
| 25B W | 99.94 | 540 | 28 | <50 | 14 | 2.2 | <9 | <5 | <8 | 40 | | 6 | <9 | <8 |

from the anodes. Addition of manganese helps coat the anode with manganese dioxide which prevents lead sulphation and dissolution but some lead will always dissolve, particularly with fresh anodes. As the anodes age, lead dissolution becomes less of a problem and this was the case here. Cobalt cathodes must be degassed in a high temperature operation. Normally, any contaminating lead is removed in this process, so the Umicore A specification should be met after degassing, even with metal containing 50 g/t Pb as it comes out of electrowinning. In some re-leach batches, sodium metabisulphite was added because it was thought that some of the re-leach residue contained cobaltic salts and the reducing agent would help redissolve these. The reducing agent addition was subsequently stopped as amount of re-leach residue was minimal, but metabisulphite may have interfered with the formation of the protective layer of MnO₂ on the surface of the anodes, by making this layer more soluble. The other element that appeared to be a problem was cadmium. It is known that this will follow cobalt and readily contaminate the cathode. In all the earlier and unwashed cathodes, cadmium levels corresponded to Umicore B quality, even in the re-washed samples. In all the cathodes from 13 onwards, Cd was below detection limits and met Umicore A quality. The source of the Cd is not really known, and there may be a small amount in the cobalt carbonate.

The other elements of concern were nickel and zinc. Zinc was well controlled. It was high in cathode 7 but that cathode was produced when zinc was released (eluted) off the VP OC 1026 resin because the re-leach solution pH was too low. Zinc levels were very low in cathodes 13 through 23 but there was a slight rise in the last two cathodes. This may be a result of operating with only one column of OC resin in this period because of temporary blockage problems in the Lead OC resin column.

Nickel was only outside the Umicore A limits in cathodes 6 and 7 which appeared to be contaminated. Cathode 10 is also high and this was in fact material recovered from the bottom of the cell, rather than being a cathode deposit stripped off the blank.

Manganese appears to be elevated (Umicore B) in most of the cathodes in the first group and easily meets the requirements of Umicore A in the second and third weeks. Acid washing the cathode samples appeared to have a significant effect on Mn levels as all of the re-washed samples from the first week readily met Umicore A specifications. Therefore, the higher values are ascribed to analysis of unwashed cathode samples and it is likely that all the cathodes met the Mn specification for Umicore A.

Finally, sulphur was only determined in a few samples and it was high in cathode 6 which was also the most heavily contaminated with other elements.

The source of sulphur in this deposit is likely to be from the incorporation of sulphate salts precipitated on the surface of the deposited metal. Sulphur may also be present from incorporation of some electrolyte within the metal as the surfaces were acid-washed. Subsequently it was verified that the sulphur contents were below 100 g/t but above 50 g/t, so in the Umicore B range. Elemental sulphur will volatilize during degassing and so should be significantly reduced to meet Umicore A specifications in the final products. Precipitated sulphates will not be removed in degassing, and good solution chemistry and plating conditions, along with formation of smooth deposits will minimize the incorporation of sulphate salts into the plated cobalt.

In general it can be expected that the cathodes, once they are vacuum degassed at temperature, will readily meet the Umicore A specification.

CONCLUSIONS

The proposed flowsheet has been successfully tested in a series of integrated pilot plant campaigns. High extractions of cobalt (~95%) were achieved. Arsenic was immobilized in the pressure leach residue as scorodite to produce tailings that can be safely discharged in a controlled impoundment. Some cobalt is co-precipitated in the Fe/As removal step, but close to 80% of this is recovered and recycled back into the circuit in the re-leach step.

The current flowsheet concept envisages removal of copper as a cement product rather than production of cathode copper as there is not enough copper in the ore to economically justify the latter option. Solvent extraction and electrowinning may play a role in the future when a higher Cu grade neighbouring deposit is processed. For now, residual copper must be removed from the feed to cobalt carbonate precipitation and two methods were investigated. It was possible to get over 99% good copper separation using selective precipitation under carefully controlled conditions. Processing the solution through Lanxess TP 207 resin was able to reduce the copper concentration to even lower levels, below the analytical reporting limits. The choice of copper removal technique will depend on the relative economics.

Cobalt carbonate was precipitated at high efficiency with sodium carbonate from feed solutions produced by both of the copper removal methods. It was possible to re-leach this carbonate in spent electrolyte to produce cathode cobalt. In the initial campaign various operating problems were encountered and these were overcome with a subsequent campaign of longer duration at a smaller scale. Zinc and cobalt concentrations in the electrolyte were controlled using Lanxess VP OC 1026 resin and Dow XUS 43578 resin respectively. The Co EW cell operated at 70°C with a current efficiency that exceeded 86%. Cell pH was controlled at 2 by the addition of NaOH. The remaining operating problem was that the cathodes tended to self-strip and there are strong indications that this can be solved by increasing the agitation of the solution in the EW cell.

Over four kilograms of cobalt cathode of excellent purity were produced. The deposit morphology was also good with dense and smooth plated metal. The pilot plant demonstrated the overall metallurgical viability of the flowsheet.

ABOUT FORTUNE MINERALS

Fortune Minerals (TSX-FT) is a diversified natural resource company with several mineral deposits and a number of exploration projects, all located in Canada. They include the Mount Klappan

anthracite coal deposits in British Columbia, and the NICO cobalt-gold-bismuth deposit, the Sue-Dianne copper-silver deposit and other base and precious metals exploration projects in the Northwest Territories.

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