

The Development of The Intec Nickel Process To Treat a Low-Grade Ni/Cu/Co/PGM Concentrate

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1.0 INTRODUCTION

Intec Ltd has patented a mixed halide hydrometallurgical process for the extraction of base metals and associated by-products from sulfidic mineral concentrates. The application of this technology has primarily focused on the Intec Copper Process (ICP), which has been developed to commercial readiness through the operation of a 350tpa demonstration plant [1]. The same mixed halide chemistry has been applied to the development of Intec Zinc Process (IZP) [2] that is the subject of a separate patent.

Most recently, the Intec process chemistry has been applied to complex ore bodies, where it permits maximum metal recovery into a low-grade bulk concentrate. All metals are leached into solution at high efficiency and subsequently converted to high purity metal and metallic by-products.

Nickel ore bodies are often mineralogically complex with economic levels of base metals (Cu and Co) and precious metals (Pt, Pd, Au, etc) often associated with the nickel. The capacity to treat low-grade bulk concentrates has the potential to significantly improve returns from complex nickel deposits. The need to find a low-cost processing route for such ores has driven the recent development of the Intec Nickel Process (INP). A laboratory-scale development program was completed in November 2002 based on a particularly low-grade nickel (3.1%)/copper (2.2%) concentrate produced from a complex nickel ore containing significant precious metals values.

The chemistry of the INP flowsheet is described in the context of the experimental results and preliminary capital and operating cost data are also presented.

2.0 INTEC NICKEL PROCESS FLOWSHEET

A major portion of nickel production is from nickel sulfide concentrate that is firstly smelted to nickel matte. The matte is subsequently converted to high purity nickel cathode via a number of hydrometallurgical routes, which includes well-established chloride-based hydrometallurgical processes such as Sumitomo's Matte Chlorine Leach Electrowin [3] and Falconbridge's Chlorine Leach Process [4]. The INP flowsheet shown in Figure 1 has a number of similar features to these processes and thus benefits from the existing commercial application of chloride hydrometallurgy to nickel production. In particular, conventional purification and electrowinning operations are readily applicable, however the INP differs from these processes in that the electrolyte contains both sodium chloride and sodium bromide. This mixed-halide electrolyte has the following advantages over straight chloride-based and sulfate-based electrolytes:

- Br^- is a stronger ligand than Cl^- , which in conjunction with a counter-current leach configuration allows the use of mild conditions (80-90°C and atmospheric pressure) to leach relatively refractory minerals such as pentlandite (NiFeS_2) and chalcopyrite (CuFeS_2). Due to these mild conditions, energy requirements benefit from the use of air to control iron rather than oxygen, eliminating the need for an oxygen plant, whilst materials of construction are predominantly low-cost FRP and plastics; and
- The relatively strong oxidant BrCl_2^- (referred to as Halex™) with an $E_h > 1000\text{mV}$ is generated during electrolysis. Halex facilitates the extraction of precious metals (PMs), which include gold and platinum group metals (PGMs).

The INP eliminates the need to smelt nickel concentrate to matte as high base metal and PM extractions can be achieved directly from the sulfide concentrate. Concentrates containing high levels of pyrrhotite are readily processed with the iron oxidized to form hematite with air.

Figure 1: Simplified flow diagram for the Intec Nickel Process

In overview, the INP consists of the main circuits of leaching, precious metal recovery, copper electrowinning, purification and nickel electrowinning. The leach circuit is of a two-stage counter-current configuration with concentrate primarily fed to Leach Stage 1, whilst oxidant from electrowinning is fed to Leach Stage 2. Precious metal recovery is via precipitation to an enriched intermediate product. Copper electrowinning employs high intensity diaphragm cells with continuous product removal as is practiced in the ICP, whilst nickel is electrowon as conventional cathode plate. A degree of flexibility exists in the purification of the pregnant liquor stream, as either precipitation reactions and/or solvent extraction can be used.

2.1 LEACHING

The main functions of each of the two leach stages are as follows:

- *Aeration Leach.* The bulk of the nickel and copper are leached in this stage, with the required oxygen input for maintaining both a high solution oxidation potential ($E_h > 580\text{mV}$ versus Ag/AgCl) and rejection of iron, provided by air addition at atmospheric pressure with the temperature maintained at 80°C .
- *Halex Leach.* Residual PMs are extracted in this stage by maintaining high solution oxidation potential ($E_h > 600\text{mV}$ versus Ag/AgCl), low pH (< 0.5) and high temperature (100°C).

2.2 PRECIOUS METAL RECOVERY

Precipitation of an intermediate PM by-product is achieved by lowering the solution oxidation potential in the presence of an active sulfide-bearing material. These conditions are generated by the addition of nickel concentrate rich in pyrrhotite and electrowon copper dendrites according to the following reactions:



PMS precipitate in either their elemental state or as sulfides. An alternative source of sulfide-bearing material can be a reagent such as NaHS (NASH).

2.3 COPPER ELECTROWINNING

After the reduction leach, the clear liquor stream is fed to the cathode chambers of the copper electrowinning cells where copper dendrites are deposited at the cathode. The copper tenor of the spent catholyte is maintained at 6g/l to minimise the residual copper passing to the copper precipitation circuit. This minimises concentrate, air and limestone inputs to downstream purification operations.

A small amount of the spent catholyte is fed to the anode chambers where Halex™ (BrCl₂⁻) oxidant is generated for return to the leach via the following reaction:



2.4 COPPER PRECIPITATION AND LIQUOR PURIFICATION

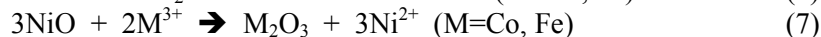
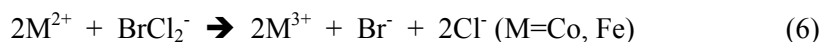
Residual copper, in the spent catholyte from copper electrowinning, is precipitated by a metathesis reaction where copper exchanges with iron from the pyrrhotite in the concentrate according to the following reaction:



The solids from the metathesis reaction are settled in a thickener with the underflow directed to the aeration leach. Due to the above reaction, the soluble iron in the thickener overflow is approximately 3g/l. This iron is subsequently rejected by air and limestone input, resulting in the following overall reaction:



The precipitated iron is settled in a thickener with the underflow sent to the Halex Leach. Removal of the trace impurities can be performed through either precipitation or solvent extraction, both of which are practiced in the chloride hydrometallurgical treatment of nickel matte. Precipitation is effected by the addition of Halex™ and alkali, with the following participating reactions:



A drawback of this method is the co-precipitation of nickel with the cobalt and iron. However, the concentrate used for the development program contained only low levels of cobalt and this in conjunction with 1g/l residual iron in the clear liquor stream from copper precipitation results in minimal residue mass and thus minimal nickel loss. Residual copper and lead in solution also precipitate through the addition of Halex™ and alkali. For copper precipitation, Cu⁺ is oxidised to Cu²⁺ by the Halex™, which in the presence of alkali is precipitated as Cu₂(OH)₃Cl, whilst lead removal is by precipitation as Pb₂(OH)₃Cl.

The solvent extraction option has not yet been thoroughly investigated and so is not currently included in the process flowsheet.

2.5 NICKEL ELECTROWINNING

Nickel electrowinning differs from copper in that a cathode plate is produced as per current commercial practice. The cathodic reaction is:



whilst the anodic reaction is the generation of Halex™ according to Equation 1.

3.0 RESULTS OF LABORATORY PROGRAM

3.1 LEACHING

Concentrate Characterisation

The particle size distribution of the concentrate was relatively coarse at a P_{80} of 105μ , which has the potential to affect metal extractions and hence all subsequent testwork was carried out on concentrate reground to $P_{80}=25\mu$. The metal composition of the concentrate (see Table 1) indicated it to be a low-grade nickel/copper concentrate, with economic levels of PMs.

Element	%	Element	ppm
Ni	3.23	Co	1230
Cu	2.03	Au	0.76
Fe	27.6	Pt	3.97
S	17.4	Pd	3.93

Table 1: Elemental Analysis of Concentrate

The mineralogical composition of the concentrate was not available, but chalcopyrite was assumed to be the predominant copper mineral and pentlandite the predominant nickel mineral. Based on these assumptions, the respective iron and sulfur not associated with these minerals inferred the presence of pyrrhotite (FeS) amounting to approximately 33% of the total concentrate composition.

The calcium and magnesium oxide content of the concentrate is important, as it must be compensated for by the addition of sulfuric acid to the leach. Rather than relying on the Ca/Mg elemental assay, a sample was titrated with H_2SO_4 to a pH of 2, indicating the need for 63kg per tonne of dry concentrate.

Aeration Leach

The majority (typically >95%) of the concentrate feed to the Intec Nickel Process is added to the aeration leach, where typically >95% of each of the base metals, nickel, copper and cobalt are extracted. The results from the laboratory leach test indicated slightly better extractions as shown in Table 2. Approximately, half of each of the gold, platinum and palladium was also leached in this stage.

Element	% Metal Extraction
Au	54
Pt	47
Pd	45
Ni	97.9
Cu	97.3
Co	97.1

Table 2: Aeration leach metal extractions

An important aim of the aeration leach tests was to quantify equipment design parameters, which are summarised in Table 3.

Design Parameter	Test 2 ($P_{80}=25\mu$)
Oxygen uptake efficiency	28%
Reactor volume ($m^3/kg-Fe^1$)	0.39
Filtration rate ($l/m^2/h$)	170

Table 3: Aeration Leach Design Parameters

Air is used to supply oxygen for the formation of hematite from the leachable iron minerals in the concentrate. Oxygen uptake efficiency is the rate-determining step for reaction, thus setting the overall leach retention time. As the oxygen input is largely related to the iron content of the concentrate, the retention time and consequently reactor volume is also a function of the iron content. Hence, the total reactor volume for the aeration leach has been expressed in terms of iron content in the concentrate feed. The filtration rate determined is the average to form a 20mm cake (equivalent to a 40mm cake for a plate and frame pressure filter).

Halex Leach

The overall metal extractions achieved in the final Halex leach stage are presented in Table 4. Additional base metal leaching in this stage further improved already high extraction to 98-99%. The primary function of the Halex Leach is to optimise precious metal extraction, with respective gold and palladium concentrations in the residue of <0.1 and <0.5ppm respectively.

Element	% Metal Extraction
Au	87
Pt	54
Pd	86
Ni	98
Cu	99
Co	98

**Table 4: Cumulative metal extractions
-Halex leach**

Overall platinum extraction remained unsatisfactory at 54%, in spite of significant improvement in the initial figure of 16% through optimisation in the areas of electrolyte composition, temperature, solution oxidation potential and retention time during the course of testing. The program was constrained in terms of time and further work will be carried out in the future to gain an understanding of the platinum mineralogy that should lead to improvement in extraction efficiency.

3.2 PRECIOUS METAL RECOVERY

Recycled copper product and fresh concentrate were added as reductants to the clear liquor from the Aeration Leach. The copper product reduces cupric to cuprous, decreasing solution oxidation potential, whilst the concentrate provides sulfides for PM precipitation. With decreasing solution oxidation potential, PMs were precipitated to reach a total of approximately 180ppm in the solids recovered after filtration (see Table 5).

¹ Reactor volume is defined per kg of non-refractory iron in the concentrate.

Final Eh (mV)	Gold (ppm)	Platinum (ppm)	Palladium (ppm)
336	58	59.5	57.4

Table 5: Precious metal content of Reduction Leach Product

Optimisation of the copper and concentrate inputs to the PM recovery circuit has not been carried out. Further work in this area and trial of precipitation by NASH can potentially improve the grade of the intermediate precious metal product.

3.3 COPPER ELECTROWINNING

The copper extracted during leaching is electrowon in a diaphragm cell as a copper dendrite product from the clear solution exiting the PM recovery step. During development, a two-stage electrowinning (EW) process was tested, based on the production of high purity copper from the first stage for sale and low grade copper from the second stage for use in the PM recovery step.

In the first stage, the spent catholyte is maintained at 10g/l with the spent catholyte passing to a stripping cell where the copper tenor is essentially zero. In both EW steps, about 10% of the spent catholyte is passed to the anode chambers to generate halex for return to the leach circuit.

The clear liquor was fed to the first stage EW at a tenor of 20-25g/l and at a rate to deliver a 10-15g/l strip, which is a compromise between maximising strip and minimising product contamination. A low current density of 250A/m² was employed to assist the copper product purity. Analysis of copper produced indicated nickel contamination at 97ppm and iron at 62ppm, exceeding LME Grade A purity for Group 5 elements of 10ppm combined. Consequently, the refining of copper produced either onsite or externally is required and so a single stage EW was substituted, operating at a spent catholyte tenor of 6g/l copper to minimize the residual copper passing to the copper precipitation step.

3.4 COPPER PRECIPITATION AND LIQUOR PURIFICATION

The spent catholyte from copper EW passes to copper precipitation where the remaining 6g/l is precipitated via a metathesis reaction at 100°C by the addition of nickel concentrate according to reaction (4). The copper tenor was reduced from 6.8g/l to 0.36g/l in five hours with analysis of the recovered solid product, which is returned to the leach, confirming copper enrichment with an increase from 2.17% to 20.9%.

The metathesis reaction liberates iron in a 1:2 mole ratio resulting in 3.3g/l of iron reporting to the liquor, essentially 100% in the ferrous oxidation state. Air is sparged to oxidize the ferrous iron to ferric with ferric iron precipitation by pH adjustment with limestone. By this method, iron was precipitated to 0.93g/l at 80°C over a 5.5 hours period. However, oxygen uptake efficiency at these low copper and iron tenors is also low and so the precipitation of the residual iron would require the addition of a small amount of halex-bearing leach liquor. This could be carried out in conjunction with the subsequent cobalt recovery step, but would result in contamination of the cobalt by-product. Both cupric and ferric ion precipitate at a pH below that of cobaltic ion and so their inclusion with the bulk of the iron precipitate is both feasible and desirable. This will probably be incorporated into the flowsheet in future.

Cobalt is recovered in the final purification step prior to nickel electrowinning. Residual copper, iron and lead are co-precipitated with the cobalt at 100°C unless these are removed first as described above. After 3 hours of reaction, 98.6% of the cobalt had been precipitated as shown in Table 6, leaving only Pb at a concentration sufficiently high to exceed the LME specification for 99.8% Ni purity in subsequent electrowinning.

Element	Co	Cu	Fe	Pb
Concentration (ppm) Before	320	230	20	10
Concentration (ppm) After	4.5	5.5	3.5	8.7

Table 6: Liquor assay before and after cobalt recovery

3.5 NICKEL ELECTROWINNING

Nickel is electrowon as a cathode plate in a conventional nickel cell. Purified pregnant liquor at 65g/l nickel was fed to the cell at a rate to maintain a 50g/l nickel background for a 15g/l strip. The strip is determined by the leach slurry density that was set at 25% w/w and kept constant during the development program. Further work is required to maximize the slurry density and consequently the nickel strip. Current density was set at 300A/m² and the temperature maintained at 55°C. Cell potential was 2.3V and the current efficiency was 95%, which translates to relatively low energy consumption of 2250 kWh per tonne nickel plated.

The purity of the electrowon nickel is presented in Table 7. The only contaminants to exceed LME specification were lead (15 ppm over the 50ppm specification) and zinc (565ppm over the 50ppm specification). Solvent extraction or ion exchange are reported as viable options [5] for zinc and lead removal, but this route has not yet been investigated.

Element	Ag	Bi	C	Co	Cu	Fe	Mn	Pb	S	Sb	Si	Zn
LME Spec. (ppm)	N/A	50	300	1500	200	200	50	50	100	50	10	50
Nickel cathode assay (ppm)	1.2	0.3	<100	45	143	90	<1	65	25	<1	<10	615

Table 7: Nickel cathode assay

4.0 PRELIMINARY COST ESTIMATION

A mass balance and process flow diagrams (PFDs) have been developed from the program data for an Intec Nickel plant treating a low-grade nickel concentrate to generate saleable metal and intermediate products. The Intec Nickel Process shares similar unit operations to those used in the Intec Copper Process, for which extensive cost data has been amassed. Such cost data, combined with the mass balance and PFDs, are used to estimate operating and capital costs for the production of 45 000 tonnes per annum of nickel. The costs can vary considerably with the mineralogy of the concentrate and thus can only serve as a general guide.

4.1 OPERATING COSTS

A detailed breakdown of the estimated operating cost is presented in Table 8. For annual production of 45 000 tonnes of nickel, the operating cost of US\$45.0 million equates to US\$45¢/lb-Ni before consideration of by-product credits.

The operating cost estimation has been divided into the five main categories of energy, labour, consumables, maintenance and miscellaneous. Energy has been calculated by comparing each unit operations with its equivalent in the Intec Copper Process. Labour uses North American rates, which has been the basis of previous calculations. Consumable costs were estimated using the same data as used in the Intec Copper Process. Over half of the consumables cost and a quarter of the total operating cost was due to sulfuric acid, for which a price of US\$120/tonne is assumed. Such a price assumes importation from overseas and thus the ability to source this reagent locally can potentially reduce overall costs to less than US\$40¢/lb-Ni.

Maintenance costs associated with electrowinning are calculated separately, due to their higher intensity when compared to the remainder of the plant. For the latter, a factor of 6% of direct capital expenditure has been assumed. Miscellaneous costs are those associated with gangue disposal at US\$1/t for return to the main tailings dam.

4.2 CAPITAL COSTS

The capital cost for an Intec Nickel plant with annual production capacity of 45 000 tonnes of nickel is summarised in Table 9. The plant cost is estimated at US\$300 million, inclusive of a 20% contingency, which equates to approximately US\$6500 per annual tonne of nickel capacity. This estimate is only accurate to $\pm 35\%$. The capital cost was calculated by estimating direct costs of equipment including installation. Factors were applied to the direct cost to estimate the indirect costs, which included owner's costs, consultant services, commissioning, construction management, capital spares and transportation.

The following are the major items in each of the direct cost categories:

- **General:** Piping, electrical, instrumentation and laboratory;
- **Building Civil Works:** Lighting, fire protection, ventilation, process buildings and site works;
- **Leach:** Agitated reactors and tanks, thickener, filters, PMS handling and conveyors;
- **Grinding/Material handling:** Grinding and concentrate handling;
- **Purification:** Copper precipitation, iron precipitation, cobalt recovery and magnesium bleed;
- **Product Recovery:** Nickel electrowinning, copper electrowinning, product washing, drying and packaging;
- **Utilities:** Electrolyte makeup, reagent inventory, leach scrubber, air compression, boiler and cooling towers.

ITEM	Cost	Quantity	US¢/lb	% of Opex	US\$(000/a)
Energy	3.5 US¢/kWh	8070 kWh/t-Ni	12.82	28.2%	12 908
Labour	35-75k US\$/annum	151 persons	5.50	12.1%	5 540
Consumables	N/A	N/A	17.81	39.3%	17 937
Maintenance	N/A	N/A	7.26	16.0%	7 313
Miscellaneous	N/A	N/A	1.99	4.4%	2 000
TOTAL ANNUAL OPERATING COSTS			45.37		45 698

Table 8: Estimated operating cost of treatment of concentrate

Area	Cost US\$
Direct costs	
General	28 570 000
Building/Civil Works	12 170 000
Leach	39 950 000
Grinding/Material Handling	12 850 000
Purification	9 330 000
Product Recovery	75 350 000
Utilities	25 020 000
Sub Total	202 240 000
Indirect costs	46 530 000
Project contingencies (20%)	49 760 000
Plant Cost	298 530 000

Table 9: Estimated Capital Cost of 45 000tpa Intec Nickel Plant

5.0 CONCLUSION

The INP development to date has demonstrated its potential to treat low grade and complex nickel concentrates at low capital and operating costs.

Further development is required to optimize several of the process unit operations prior to proceeding to a pilot operation. However, each of the unit operations has been successfully demonstrated at laboratory scale.

The cost structure of the INP is sufficiently low that many low-grade nickel deposits that are currently uneconomic may be transformed into viable projects.

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